

The use of flax and hemp resource for particleboard

by

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Abstract

The focus of this study was to investigate flax shive and hemp hurd as alternate residue for particleboard production, investigate the lowest percentage of the pricier polymeric diphenyl methane diisocyanate (pMDI) resin that can be used to effectively bond the residues and evaluate an acrylic-based resin for particleboard manufacture.

The flax shive and hemp hurd had lower bulk densities and higher aspect ratios compared with wood. Their higher aspect ratios offered greater overlap in bonding leading to consistently higher bending properties that exceeded American National Standards Institute (ANSI) requirements for low density (LD2) particleboard and in some cases, medium density (M2) particleboard. Because of their particle geometry, the flax shive and hemp hurd particleboards also showed minimal linear expansion with changes in moisture content between 50% and 90% relative humidity (at $20 \pm 3^{\circ}\text{C}$) and were within ANSI requirements. The high absorption capacity of the residues resulted in higher thickness swell and water absorption properties in contrast to wood.

Improvements in bending strength above 40% and stiffness properties above 25% was achieved for wood, hemp hurd and flax shive particleboards by incorporating 15 weight % flax and hemp fiber in continuous mat form at the points of maximum tensile and compressive stresses in particleboard.

Test results confirmed the possibility of using 2.5% pMDI resin load, a percentage lower than the commercially viable 3%–6% addition levels that are commonly used with wood residues. The results further demonstrated that based on 2.5% pMDI resin load and as much as 20% mass lignin substitution boards with satisfactory mechanical properties that exceed LD2 grade requirements could be manufactured from hemp hurd and flax shive. Dynamic scanning calorimetry results and the current cost of the acrylic-based resin suggests that it is not suited for particleboard manufacture from flax shive and hemp hurd.

Overall, based on mechanical performance flax shive and hemp hurd residues can be considered as alternate biomass for particleboards of greater performance to wood for use in shelving and furniture applications. But the high cost of the residues compared to wood does not currently make it economical for particleboard manufacture.

Preface

The research work conducted in this dissertation was identified by Solace Araba Sam-Brew. The research study including the review of the particleboard industry and manufacturing sequence, characterization of the properties of the flax shive and hemp hurd residues, thermal analysis of the different resin and residue combinations, manufacture and testing of the particleboard types, and statistical analysis of results were conducted by Solace Araba Sam-Brew.

The idea to use a lignin-based resin formulation was proposed by Dr. Frank Ko and the lignin sourced by Martin Feng.

The dissertation including the peer-reviewed manuscript was prepared by Solace Araba Sam-Brew and co-authored by Dr. Gregory D. Smith. Versions of Chapters 3 and 4 will be submitted for publishing. A version of Chapter 6 has been published as follows:

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Table of Contents

Abstract	ii
Preface.....	iii
Table of Contents	iv
List of Tables	ix
List of Figures	xii
List of Abbreviations	xxiii
Acknowledgements.....	xxv
Dedication	xxvii
1. Introduction.....	1
1.1 Overview of wood based composites	1
1.2 Particleboard — the manufacturing process	2
1.2.1 Definition	2
1.2.2 Raw material — wood residue.....	4
1.2.3 Residue processing, classification and drying	5
1.2.4 Adhesive — types and quantities used	6
1.2.5 Blending	7
1.2.6 Mat forming	10
1.2.7 Prep-pressing.....	12

1.2.8	Hot pressing	13
1.2.9	Edge trimming, cooling, sanding and cutting to size	15
1.2.10	Quality control	18
1.3	Particleboard industry	19
1.3.1	Major industry problem	22
1.3.2	What remains to be done.....	28
1.3.3	Proposed means of tackling the issues identified.....	30
1.4	Research objectives.....	37
1.5	Research structure	38
2.	Materials and methods	40
2.1	Raw materials used in the study.....	40
2.1.1	Flax shive and hemp hurd residues	40
2.1.2	Wood residues.....	43
2.1.3	Flax and hemp fiber	44
2.1.4	Resins	45
2.2	Description of experimental methods employed	46
2.2.1	Residue and fiber characterization.....	46
2.2.2	Differential scanning calorimetry (DSC).....	47
2.2.3	Particleboard manufacture	50

2.2.4	Short-term property testing	53
2.2.5	Data analysis	61
3.	Flax shive and hemp hurd residues as alternate raw material for particleboard production.	62
3.1	Background	62
3.2	Experimental design.....	64
3.3	Results and discussion	65
3.3.1	Particle geometry and bulk densities	65
3.3.2	Mat core temperature	68
3.3.3	Moisture content, average board density and vertical density profile	70
3.3.4	DSC analysis	77
3.3.5	Mechanical properties	81
3.3.6	Physical properties	86
3.3.7	Comparison of flax shive and hemp hurd particleboard properties with particleboard from other non-wood residues	92
3.3.8	Cost competitiveness of wood versus flax shive/hemp hurd	94
3.4	Conclusions.....	96
4.	Formaldehyde free acrylic-based resin—a binder for non-wood residue particleboards	98
4.1	Background	98
4.2	Experimental design.....	99
4.3	Results and discussion	100

4.3.1	Moisture content, average board density and vertical density profile	100
4.3.2	DSC analysis	103
4.3.3	Mechanical properties	112
4.3.4	Physical properties	116
4.4	Conclusions.....	121
5.	Lignin-based resin for the manufacture of wood and non-wood particleboards	123
5.1	Background.....	123
5.2	Experimental design.....	125
5.3	Results and discussion	127
5.3.1	Moisture content, average board density and vertical density profile	127
5.3.2	DSC analysis	129
5.3.3	Mechanical properties	143
5.3.4	Physical properties	150
5.4	Conclusions.....	157
6.	Flax and hemp fiber-reinforced particleboard	159
6.1	Background.....	159
6.2	Experimental design.....	162
6.3	Results and discussion	164
6.3.1	Moisture content, average board density and vertical density profile	164

6.3.2	Mechanical properties	167
6.3.3	Physical properties	174
6.4	Conclusions.....	178
7.	Research summary and conclusions	181
7.1	Summary.....	181
7.2	Significance of the study to the particleboard industry	184
7.3	Limitations of the study and future research directions.....	186
	References.....	189
	Appendices.....	207
	Appendix A: wood-based panels production and consumption statistics	207
	Appendix B: statistical analysis - chapter 3.....	208
	Appendix C: statistical analysis - chapter 4.....	227
	Appendix D: statistical analysis - chapter 5	240
	Appendix E: statistical analysis - chapter 6.....	263

List of Tables

Table 1.1: ANSI A208.1-2009 particleboard grade requirements. H= high density ($> 800 \text{ kg/m}^3$), M= medium density ($640 - 800 \text{ kg/m}^3$), LD= low density ($< 640 \text{ kg/m}^3$), PBU=underlayment, D=manufactured home decking. ns*- not specified.	19
Table 1.2: Canadian particleboard companies, locations and product specifications.....	21
Table 1.3: Canadian particleboard production and trade from 2012 to 2015.	21
Table 1.4: Chemical components of flax shive and hemp hurd residues.....	35
Table 2.1: Physical and chemical properties of resins used in the research.	46
Table 3.1: Design of experiment for low density wood, flax shive and hemp hurd particleboards.	65
Table 3.2: Mean aspect ratio of wood, flax shive and hemp hurd residues. n=50 for each mean. Values in parenthesis are standard deviations.....	65
Table 3.3: Bulk density of wood, hemp hurd and flax shive residues. n=3 for each mean. Values in parenthesis are standard deviations.....	67
Table 3.4: Average board density and moisture content of low density particleboards. Each board density mean was computed from 12 samples. Mean moisture content was calculated from 12 samples for the 2.5% boards and 11 for the 5% boards. Values in parenthesis are standard deviations.....	70
Table 3.5: Face and core layer density variations for 2.5% and 5% resin load boards.	74
Table 3.6: Face and core layer density variations for particleboards manufactured with residues conditioned to 11% moisture content.....	75
Table 3.7: Thermal properties of curing reaction between pMDI resin and residues.....	79

Table 3.8: Static bending properties of pMDI-bonded particleboards. Data is based on ANSI A208.1-1999 panel averages. n=12 for each mean.	84
Table 3.9: Thickness swell and water absorption properties of pMDI-bonded particleboards. Each mean was computed from 12 samples for the 2.5% boards and 11 for the 5% boards.	89
Table 3.10: Comparison of the strength properties of particleboards manufactured from isocyanate resin and non-wood residues.	93
Table 3.11: Estimated production costs for a 4 foot by 8 foot particleboard panel.	95
Table 4.1: Design of experiment for 3-layered particleboards using Acrodur® 950L resin.	100
Table 4.2: Average board density and moisture content of Acrodur® 950L bonded particleboards. n=20 for average board density means and n=16 for moisture content means. Values in parenthesis are standard deviations.	101
Table 4.3: Face and core layer density variations for Acrodur® 950L particleboards.	103
Table 4.4: Thermal properties of curing reaction between Acrodur® 950L resin and residues..	106
Table 4.5: Thermal properties of curing reaction between urea formaldehyde (UF) resin and residues.	107
Table 4.6: Bending strength and stiffness properties of Acrodur® 950L particleboards. Panel means are presented according to ANSI A208.1-1999. Values in parenthesis are standard deviations.	115
Table 4.7: Properties comparison of particleboards manufactured from non-wood residues. ...	120
Table 5.1: Design of experiment for particleboards manufactured with pMDI-lignin resin.	127
Table 5.2: Average board density and moisture content of pMDI-lignin particleboards. n=12 for each mean. Values in parenthesis are standard deviations.	128

Table 5.3: Thermal properties of the curing reaction between pMDI and Kraft lignin.....	131
Table 5.4: Internal bond properties of particleboards made with 2.5% pMDI-lignin resin load. Panel means based on ANSI A208.1-1999. For each mean n=32. Values in parenthesis are standard deviations. * indicates control samples.	143
Table 5.5: Mechanical strength properties of pMDI-lignin particleboards. Panel means presented according to ANSI A208.1-1999. For each mean n=32 for IB and n=12 for MOR/MOE. Values in parenthesis are standard deviations. * indicates control panels.	147
Table 6.1: Mechanical properties of some fiber materials.....	161
Table 6.2: Design of experiment for 3-layered fiber-reinforced particleboard.	163
Table 6.3: Average board density and moisture content of fiber reinforced particleboards. n=11 for each mean. Values in parenthesis are standard deviations.....	165
Table 6.4: Mechanical strength properties of 3-layered hemp and flax fiber-reinforced particleboard. Data presented is based on ANSI A208.1-2009 lower 5th percentile panel averages.	168

List of Figures

Figure 1.1: Three-layered particleboard configuration.	3
Figure 1.2: Particleboard production process.....	3
Figure 1.3: Green residue drier. Source: Sam-Brew 2016.	4
Figure 1.4: Overhead classifying bins for screening residue into face and core material. Source: Sam-Brew 2016.....	5
Figure 1.5: Storage silos for classified wood furnish. Source: Sam-Brew 2016.	6
Figure 1.6: Furnish being transported into blender. Source: Sam-Brew 2016.....	8
Figure 1.7: Batch blender with several hoses/nozzles for spraying resin and additives. Source: Sam-Brew 2016.....	9
Figure 1.8: Blended furnish with a) moisture meter and b) magnet positioned above conveyor belt. Source: Sam-Brew 2016.....	9
Figure 1.9: Forming bin with resinated face furnish. Source: Sam-Brew 2016.	10
Figure 1.10: Mat formed on a) continuous caul-less line and b) caul plate. Source: Sam-Brew 2016.....	11
Figure 1.11: Trimming of mat edge on caul-less forming line. Source: Sam-Brew 2016.	11
Figure 1.12: Magnet located above formed mat to remove ferro-magnetic objects. Source: Sam- Brew 2016.	12
Figure 1.13: Prep-pressing of formed mat using a system of rollers. Source: Sam-Brew 2016.	13
Figure 1.14: Daylight press with multiple openings for hot pressing mats. Source: Sam-Brew 2016.....	14

Figure 1.15: Sensors for blister detection and weight measurements fixed to conveyor system. Source: Sam-Brew 2016.....	16
Figure 1.16: Cooling wicket for manufactured particleboard. Source: Sam-Brew 2016.....	17
Figure 1.17: Sanding of particleboard. Source: Sam-Brew 2016.	18
Figure 1.18: Total wood-based panel production in Canada 2015. Source: United Nations Economic Commission for Europe (UNECE) Committee on Forests and the Forest Industry. 2016.....	20
Figure 1.19: Non-wood natural fiber classification. Source: Anandjiwala and Blouw 2007. .	23
Figure 1.20: Total production of flax in western Canada. Source: Flax Council Canada 2016.	31
Figure 1.21: Total hemp seeded area in Canada. Source: Laate 2012.	32
Figure 1.22: Schematic of the cross-section of a flax stalk showing the arrangement of bast fibers and woody core tissue. Modelled after Van Den Oever et al. 2000, Zimmermann et al. 2004 and Munder et al. 2005.	34
Figure 1.23: SEM image of the cross-section of flax shive and hemp hurd woody tissue. Source: Sam-Brew 2016.....	35
Figure 2.1: Flax shive and hemp hurd residues. Source: Sam-Brew 2016.	41
Figure 2.2: Mechanical shaker with 4 sieves for residue fractioning. Source: Sam-Brew 2016.	41
Figure 2.3: Flax shive and hemp hurd residues collected on the 0.5 mm, 1 mm and 2 mm sieves after screening. Ruler on bottom is demarked in cm. Source: Sam-Brew 2016. ..	42
Figure 2.4: Hemp fiber aggregates as seen in hemp hurd furnish collected on 0.5 mm sieve and blended with resin. Source: Sam-Brew 2016.	43

Figure 2.5: Industrial wood residue. Ruler on bottom is demarked in cm. Source: Sam-Brew 2016.	44
Figure 2.6: Flax and hemp fiber bundles. Source: Sam-Brew 2016.	44
Figure 2.7: Aligning of hemp fiber using carding board and hand carder: (a) custom made carding board and hand carder, (b) carding of hemp fiber, and (c) aligned hemp fiber mat. Source: Sam-Brew 2015.	45
Figure 2.8: Bulk density measurement of hemp hurd residue. Source: Sam-Brew 2016.	46
Figure 2.9: Ball mill for grinding residues. Source: Sam-Brew 2016.	48
Figure 2.10: DSC curve illustrating reaction heat, onset and peak temperature data extraction.	49
Figure 2.11: Three-layered hemp hurd particleboard with fine face and coarse core furnish. Source: Sam-Brew 2016.	50
Figure 2.12: Drais paddle-type particleboard blender. Source: Sam-Brew 2016.	51
Figure 2.13: Mat forming and prepressing process during particleboard manufacture. Source: Sam-Brew 2016.	52
Figure 2.14: Hot pressing operation. Source: Sam-Brew 2016.	53
Figure 2.15: Sample cutting pattern used to obtained test samples for physical and mechanical property tests. Source: Sam-Brew 2016.	54
Figure 2.16: Test samples stacked in a constant humidity chamber. Source: Sam-Brew 2016.	54
Figure 2.17: X-ray machine for vertical density gradient measurements. Source: Sam-Brew 2016.	55
Figure 2.18: Internal bond test with a universal testing machine. Source: Sam-Brew 2016. ..	56

Figure 2.19: Three-point bending test conducted on hemp hurd particleboard. Source: Sam-Brew 2016.	57
Figure 2.20: Linear expansion measurements for wood particleboard measured a) parallel and b) perpendicular to the forming direction of the board. Arrow indicates the forming direction of the original board. Source: Sam-Brew 2016.....	59
Figure 2.21: Submerged particleboards in thickness swell and water absorption measurements. Source: Sam-Brew 2016.....	60
Figure 3.1: Aspect ratio of face and core wood, flax shive and hemp hurd residues. n=50 for each residue type.	66
Figure 3.2: Visual comparison of core wood, flax shive and hemp hurd residues. Ruler on bottom is demarked in cm. Source: Sam-Brew 2016.....	66
Figure 3.3: Differences in mat core temperature during hot pressing of the 5% pMDI boards.	68
Figure 3.4: Face and core density profiles of low density particleboards, expressed as peak face (F1, F2) and core densities (C). n=6 for each mean. Error bars represent least significant difference (LSD) between means.	72
Figure 3.5: Differences in compressive force plotted as a function of time during hot pressing.	74
Figure 3.6: Face and core density profiles of particleboards manufactured with conditioned residues, expressed as peak face (F1, F2) and core densities (C). n=6 for each mean. Error bars represent least significant difference (LSD) between means.	75
Figure 3.7: SEM image of vessels in flax shive and hemp hurd residues. Source: Sam-Brew 2016.....	76

Figure 3.8: DSC curves of the curing reaction between (a) oven dry wood, hemp hurd, flax shive residues and pMDI resin and (b) similar residues combined with pMDI resin and distilled water.	78
Figure 3.9: Internal bond strength of low density pMDI particleboards. Horizontal line indicates minimum value stipulated by ANSI A208.1-1999 for M2 boards. n=32 for each mean. Error bar represents least significant difference (LSD) between means. ...	83
Figure 3.10: Bending strength and stiffness properties of particleboards manufactured with 2.5% AND 5% pMDI resin load. Horizontal line indicates minimum value stipulated by ANSI A208.1-1999 for LD2 and M2 grade particleboards. n=12 for each mean. Error bar represents least significant difference (LSD) between means.	85
Figure 4.1: Density profile of particleboards; peak face (F1, F2) and core densities (C). n=6 for each mean. Error bars represent least significant difference (LSD) between means.	102
Figure 4.2: Dynamic scan of Acrodur [®] 950L resin.....	104
Figure 4.3: Dynamic scan of urea formaldehyde resin (Casco-Resin [™]).	105
Figure 4.4: DSC curves of curing reaction between wood, hemp hurd, flax shive residues and Acrodur [®] 950L resin.	106
Figure 4.5: DSC curves of curing reaction between wood, hemp hurd, flax shive residues and urea formaldehyde (UF) resin.	107
Figure 4.6: Onset temperature comparison between Acrodur [®] 950L and UF resin based on residue type. Error bars represent 95% confidence intervals.	108
Figure 4.7: Peak temperature comparison between Acrodur [®] 950L and UF resins combined with different residue types. Error bars represent 95% confidence intervals.....	110

Figure 4.8: Reaction heat comparison between Acrodur® 950L and UF resin based on residue type. Error bars represent 95% confidence intervals.....	111
Figure 4.9: Internal bond strength of low density particleboards bonded with Acrodur® 950L resin. Horizontal line indicates minimum value stipulated by ANSI A208.1-1999 for M2 boards. n=32 for each mean. Error bars represent 95% confidence intervals.	112
Figure 4.10: Bending strength and stiffness properties of particleboards manufactured with Acrodur® 950L resin. Horizontal line indicates minimum value stipulated by ANSI A208.1-1999 for LD2 and M2 grade particleboards. n=20 for each mean. Error bars represent 95% confidence intervals.....	114
Figure 4.11: Comparison of bending strength and stiffness properties between 2.5% pMDI and 7% Acrodur® 950L particleboards. Horizontal line indicates minimum value stipulated by ANSI A208.1-1999 for LD2 grade particleboards. Error bars represent 95% confidence intervals.	116
Figure 4.12: Linear expansion with changes in moisture content between 50% and 90% relative humidity. Horizontal line indicates maximum value stipulated by ANSI A208.1-2009. n=14 for each mean. Error bars represent 95% confidence intervals.....	117
Figure 4.13: Thickness swell and water absorption properties of Acrodur® 950L particleboards; 2h (2 hours) and 24h (24 hours). n=16 for each mean. Error bars represent 95% confidence intervals.....	118
Figure 5.1: Mechanical stirring of Kraft lignin and pMDI resin. Source: Sam-Brew 2016. .	126
Figure 5.2: Face and core density profiles of pMDI-lignin particleboards; peak face (F1, F2) and core densities (C), 5% lignin (5L), 20% lignin (20L). n=6 for each mean. Error bars represent least significant difference (LSD) between means.	129
Figure 5.3: DSC graph of pMDI and Kraft lignin at different mass substitutions.	130

Figure 5.4: DSC graph of a mixture of distilled water, pMDI and Kraft lignin at different mass substitutions.....	131
Figure 5.5: DSC graphs of different residues combined with pMDI and 5 weight % Kraft lignin substitution.	132
Figure 5.6: Onset temperature, peak temperature and reaction heat comparison between different residues combined with 5 weight % lignin in pMDI resin. n=3 for each residue mean and n=2 for pMDI-lignin resin. Error bars represent 95% confidence intervals.	133
Figure 5.7: DSC graphs of different residues combined with pMDI and 10 weight % Kraft lignin substitution.	135
Figure 5.8: Onset temperature, peak temperature and reaction heat comparison between different residues combined with 10 weight % lignin in pMDI resin. n=3 for each residue mean and n=2 for pMDI-lignin resin. Error bars represent 95% confidence intervals.	136
Figure 5.9: DSC graphs of different residues combined with pMDI and 20 weight % Kraft lignin substitution.	137
Figure 5.10: Onset temperature, peak temperature and reaction heat comparison between different residues combined with 20 weight % lignin in pMDI resin. n=3 for each residue mean and n=2 for pMDI-lignin resin. Error bars represent 95% confidence intervals.	138
Figure 5.11: Thermal properties of mixtures of wood residue and pMDI resin based on mass lignin substitution levels. n=3 for each mean. Error bars represent 95% confidence intervals.	141
Figure 5.12: Thermal properties of mixtures of hemp hurd residue and pMDI resin based on mass lignin substitution levels. n=3 for each mean. Error bars represent 95% confidence intervals.....	142

Figure 5.13: Thermal properties of mixtures of flax shive residue and pMDI resin based on mass lignin substitution levels. n=3 for each mean. Error bars represent 95% confidence intervals.....	142
Figure 5.14: Internal bond strength of particleboards manufactured from pMDI-lignin resin; 5% lignin (5L), 20% lignin (20L), w (wood), hh (hemp hurd), fs (flax shive). Horizontal line indicates minimum value stipulated by ANSI A208.1-1999 for M2 grade particleboards n=32 for each mean. Error bars represent 95% confidence intervals.	144
Figure 5.15: Comparison of internal bond strength of particleboards manufactured with pMDI and pMDI-lignin resin; 5% lignin (5L), 20% lignin (20L), w (wood), hh (hemp hurd), fs (flax shive). Horizontal line indicates minimum value stipulated by ANSI A208.1-1999 for M2 grade particleboard. n=32 for each mean. Error bars represent 95% confidence intervals.	145
Figure 5.16: Bending strength properties of pMDI-lignin particleboards; 5% lignin (5L), 20% lignin (20L), w (wood), hh (hemp hurd), fs (flax shive). Horizontal line indicates minimum value stipulated by ANSI A208.1-1999 for LD2 grade particleboard. n=12 for each mean. Error bars represent 95% confidence intervals.....	147
Figure 5.17: Bending stiffness properties of pMDI-lignin particleboards; 5% lignin (5L), 20% lignin (20L), w (wood), hh (hemp hurd), fs (flax shive). Horizontal line indicates minimum value stipulated by ANSI A208.1-1999 for LD2 and M2 grade particleboards. n=12 for each mean. Error bars represent 95% confidence intervals.	148
Figure 5.18: Comparison of the bending strength and stiffness properties of pMDI and pMDI-lignin particleboards; 5% lignin (5L), 20% lignin (20L), w (wood), hh (hemp hurd), fs (flax shive). Horizontal line indicates minimum value stipulated by ANSI A208.1-1999 for LD2 and M2 grade particleboard. n=12 for each mean. Error bars represent 95% confidence intervals.....	149

Figure 5.19: Linear expansion of samples subjected to increasing relative humidity from 50% to 90%; 5% lignin (5L), 20% lignin (20L), w (wood), hh (hemp hurd), fs (flax shive). Horizontal line indicates maximum value stipulated by ANSI A208.1-2009. n=16 for each mean. Error bars represent 95% confidence intervals.	151
Figure 5.20: Comparison of the linear expansion values of pMDI and pMDI-lignin particleboards; 5% lignin (5L), 20% lignin (20L), w (wood), hh (hemp hurd), fs (flax shive). Horizontal line indicates maximum value stipulated by ANSI A208.1-2009. n=16 for each mean. Error bars represent 95% confidence intervals.	152
Figure 5.21: Thickness swell and water absorption properties of particleboards manufactured with pMDI-lignin resin; 5% lignin (5L), 20% lignin (20L), w (wood), hh (hemp hurd), fs (flax shive). n=12 for each mean. Error bars represent 95% confidence intervals.	154
Figure 5.22: Comparison of the thickness swell and water absorption characteristics of pMDI and pMDI-lignin based particleboards; 5% lignin (5L), 20% lignin (20L), w (wood), hh (hemp hurd), fs (flax shive). n=12 for each mean. Error bars represent 95% confidence intervals.	156
Figure 6.1: Particleboard reinforced with continuous layers of flax or hemp fiber. Source: Sam-Brew 2016.	162
Figure 6.2: Three-layered particleboard mat comprising of flax shive residues and flax fiber. Source: Sam-Brew 2015.	164
Figure 6.3: Comparison of vertical density profile through fiber-reinforced particleboard. Vertical lines indicate fiber-reinforcement zones).	166
Figure 6.4: Face and core density profile of fiber-reinforced particleboards expressed as peak face (F1, F2) and core (C) densities. n=6 for each particleboard mean. Error bars represent least significant difference between means.	167

- Figure 6.5: Internal bond strength of fiber-reinforced particleboards: wood-hemp (w-h), wood-flax (w-f), hemp hurd-hemp (hh-h) and flax shive-flax (fs-f). Horizontal line indicates minimum value stipulated by ANSI A208.1-1999 for M2 boards. n=32 for each mean. Error bars represent least significant difference between means. 169
- Figure 6.6: Comparison of 100% wood, hemp hurd and flax shive particleboards with flax and hemp fiber-reinforced particleboards: wood (w), wood-hemp (w-h), wood-flax (w-f), hemp hurd (hh), hemp hurd-hemp (hh-h), flax shive (fs) and flax shive-flax (fs-f). n=32 for each mean. Error bars represent 95 % confidence intervals. 170
- Figure 6.7: Photograph of typical delamination during internal bond testing in the lower flax fiber layer for particleboard composed of wood residues and flax fibers. Source: Sam-Brew 2015. 170
- Figure 6.8: Fracture surface of (a) flax fiber layer in a wood-flax particleboard and (b)hemp fiber layer in a wood-hemp particleboard. Source: Sam-Brew 2016. 171
- Figure 6.9: Bending strength properties of fiber-reinforced particleboard: wood-hemp (w-h), wood-flax (w-f), hemp hurd-hemp (hh-h) and flax shive-flax (fs-f). Horizontal line indicates minimum value stipulated by ANSI A208.1-1999 for M2 boards. n=14 for each mean. Error bars represent least significant difference between means. 172
- Figure 6.10: Comparison of the bending strength and stiffness properties of 100% wood, hemp hurd and flax shive particleboards with flax and hemp fiber-reinforced particleboards: wood (w), wood-hemp (w-h), wood-flax (w-f), hemp hurd (hh), hemp hurd-hemp (hh-h), flax shive (fs) and flax shive-flax (fs-f). n=14 for each mean. Error bars represent 95% confidence interval. 173
- Figure 6.11: Short and long-term thickness swell and water absorption of flax and hemp fiber-reinforced particleboards: wood-hemp (w-h), wood-flax (w-f), hemp hurd-hemp (hh-h) and flax shive-flax (fs-f). n=11 for each mean. Error bars represent least significant difference between means. 175

Figure 6.12: Comparison of the 2 and 24 hours thickness swell and water absorption characteristics of 100% wood, hemp hurd and flax shive particleboards with fiber-reinforced particleboards: wood (w), wood-hemp (w-h), wood-flax (w-f), hemp hurd (hh), hemp hurd-hemp (hh-h), flax shive (fs) and flax shive-flax (fs-f). n=11 for each mean. Error bars represent 95% confidence interval. 176

Figure 6.13: Microscopic images of the cross-section of uncompressed and compressed flax and hemp fiber mats. Source: Sam-Brew 2016..... 178

List of Abbreviations

AAFC	Agriculture and Agri-Food Canada
AARD	Alberta Agriculture and Rural Development
ANCOVA	Analysis of Covariance
ANOVA	Analysis of Variance
ANSI	American National Standards Institute
APA	American Plywood Association
ASTM	American Society for Testing and Materials
ATCM	Airborne Toxic Control Measure
BIMAT	Biomass Inventory Mapping and Analysis Tool
CARB	California Air Resource Board
CHTA	Canadian Hemp Trade Alliance
DSC	Differential scanning calorimetry
EWPA	Engineered Wood Products Association of Australasia
IB	Internal bond
LD2	Low density grade particleboard
LE	Linear expansion
LSD	Least Significant Difference
LSL	Laminated Strand Lumber
LVDT	Linear voltage differential transducer
LVL	Laminated Veneer Lumber
M2	Medium density grade particleboard
MC	Moisture content
MDF	Medium Density Fiberboard
MDI-	Methylene diphenyl diisocyanate
MF	Melamine formaldehyde
MOE	Modulus of Elasticity

MOR	Modulus of Rupture
MUF	Melamine urea formaldehyde
NAF	No-added formaldehyde resins
OSB	Oriented Strand Board
PF	Phenol Formaldehyde
pMDI	Polymeric diphenyl methane diisocyanate
QC	Quality control
SEM	Scanning electron microscope
TS	Thickness Swell
UF	Urea Formaldehyde
ULEF	Ultra-low emitting formaldehyde resins
VDP	Vertical Density Profile
WA	Water absorption

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1. Introduction

The main aim of this chapter is to provide contextual information on the wood products industry and processing, specifically particleboard production, to give readers an understanding of the research work presented in subsequent chapters. The chapter provides some information on the raw material problems being faced by the particleboard industry and what has been done so far to address some of these problems. Other issues that still need to be tackled are presented and detailed descriptions of how this research attempts to deal with these issues are also included in the chapter.

1.1 Overview of wood based composites

For centuries wood has been one of the most used renewable material. Its usefulness dates back as far as the prehistoric era where it was used for simple household furniture, fuel, weapons and shelter, and in more complex structures as frames for biplanes during the pre-war period (Jakab 1999). Over the years through technological advancements there has been an increase in the use of wood for more applications in the construction, transportation, agriculture, mining, printing and publishing industries (Kennedy 2004). Because of continual demand and extensive use the price of lumber continues to increase on the world market and it has become necessary to research and develop alternative ways of using low quality logs, lesser known species, all parts of the tree (branches, stumps) and wood by-products (sawdust, chips, shavings, slabs) efficiently, encouraging the manufacture of wood-based composite products.

Wood composites, also known as engineered wood, are a combination of wood (veneer, flakes, strands, chips, fibres, by-products or recycled materials) and an adhesive bonded together (Youngquist 2007). Wood is an anisotropic material and its properties also vary within and between species. This concept of composites helps eliminate some of the variability through the manufacture of panels that have more uniform properties (Irle et al. 2013). Wood composites are also beneficial in that their properties can be engineered making it possible to produce panels in different size ranges unlike solid lumber which is restricted by log size.

The oldest wood composite, Plywood, is reported to have its origins in Egypt around 3500 years ago, and the concept modernised in the early 19th century with the invention of the rotary lathe and water resistant resins in the 20th century (Kollman et al. 1968; EWPAA 2012; APA 2013). This composite consists of odd number of layers of wood veneer glued together with the wood grain/fibre direction perpendicular to each other to produce a dimensionally stable panel. With a shortage of lumber by the 1940's the lumber industry sought to make use of the large quantities of wood waste produced by the mills and factories. This led to the manufacture of Particleboard from layers of wood particles or fibres bonded with adhesives and consolidated under heat and pressure (Kollman et al. 1968; Cheetham 2009). Then in the late 1970's another wood composite known as Oriented Strand Board (OSB) evolving from Waferboards was introduced to the industry where strands of wood were oriented to produce panels with great bending strength (OSBGuide 2013). The term wood composite also encompasses the lamination of lumber and veneer to produce structural products such as Glulam, Laminated Strand Lumber (LSL), Laminated Veneer Lumber (LVL) and Parallel Strand Lumber.

The production processes for the different wood-based composites have evolved over the years. In recent years, the focus has been on how to increase panel strength properties and reduce the production cost (i.e. material or direct production) to meet society's need for products that are durable, affordable and environmentally friendly. This is being achieved through the use of advance technologies such as novel blending techniques, development of new resin types and combining wood with other materials (such as plastic, gypsum etc.) to produce composite products engineered to specific design conditions.

1.2 Particleboard — the manufacturing process

1.2.1 Definition

Particleboard is a non-structural wood composite made from lignocellulosic particles bonded with an adhesive under heat and pressure typically 165-200 °C and 2-4 MPa respectively depending on the adhesive, raw material, board density and thickness. It is a randomly oriented composite which takes advantage of the wood particle characteristics for the final board strength properties. It

generally consists of approximately 90 weight % wood and less than 10 weight % adhesive. Unlike conventional fiber-reinforced composites such as glass fiber polypropylene, particleboard makes use of a lower weight percentage of resin which is applied in droplet form on the particle surface. The boards are typically 3-layered (face-core-face as illustrated in Figure 1.1), formed with a face to core ratio of 40:60 with the face comprising of fine particles and the core consisting of the coarse particles; this sandwich design provides a smooth board surface for lamination.

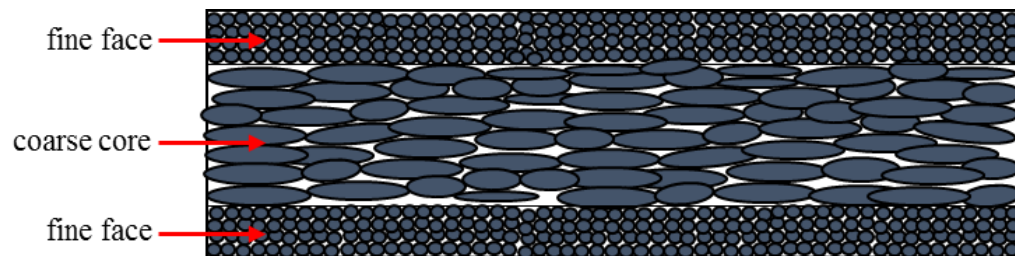


Figure 1.1: Three-layered particleboard configuration.

Particleboard production involves several steps which are summarised in the flow diagram shown in Figure 1.2 and explained in detail in subsequent subsections.

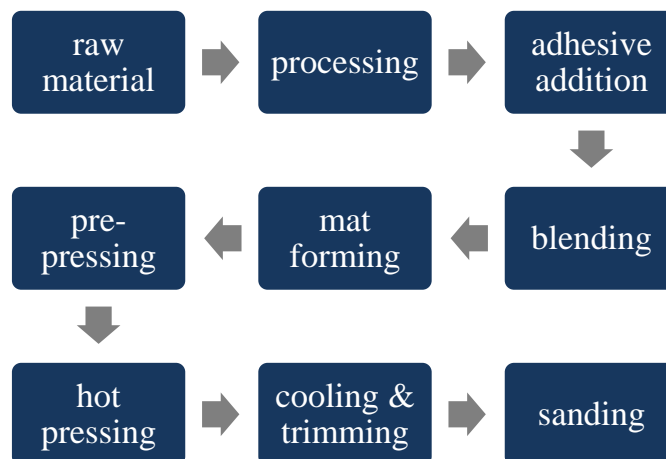


Figure 1.2: Particleboard production process.

1.2.2 Raw material — wood residue

Wood residues in the form of sawdust, chips, slabs, shavings, and plywood trims from sawmills, joinery manufacturers and plywood mills are commonly used for particleboard production, the shavings being the most expensive and sawdust the cheapest. Residues from low density wood species which are less dense and easier to process are preferred for particleboard manufacture as the material needs to be compacted above its natural density to ensure good contact between the particles. This is because the level of compaction determines the physical and mechanical strength properties of the particleboard. As such in North America, particleboard is mostly manufactured with softwood species usually a mixture of Spruce, Pines and Firs. Most of the wood residues for particleboard production are obtained dry but in some cases wet which are referred to as green residues. To use the green residues, they are put through a green drier to reduce their moisture content (MC) before further processing (Figure 1.3). Large industrial size magnets are used to pick up ferro-magnetic materials from the wood residue pile before processing and more magnets are positioned at every stage of processing until the pressing of the particleboard. To safeguard against fires, most mills have spark detection and dust filtering systems located throughout the entire mill.



Figure 1.3: Green residue drier. Source: Sam-Brew 2016.

1.2.3 Residue processing, classification and drying

The wood residues (usually a mix of large, medium and fine residues) go through a secondary breakdown process using hammer mills, impact mills, wing beaters or knife ring flakers to further reduce the particle size. The refined residue is then transferred to bins with mechanical screens/sieves where they are classified into core (≥ 1 mm) and face (≤ 1 mm) material (Figure 1.4); face and core particle sizes vary from mill to mill based on individual recipes. Oversize residues are returned to the secondary breakdown machines and sieved again.



Figure 1.4: Overhead classifying bins for screening residue into face and core material. Source: Sam-Brew 2016.

The face and core residues are conveyed separately into designated dryers where their MC is reduced to approximately 4-6% for the face and 3-4% for the core. Mostly more moisture is preferred in the face than the core to create a moisture gradient between them and enhance the steam shock effect which will be explained in subsequent sections. The residue commonly called furnish is transported on conveyor belts to storage silos (Figure 1.5). Moisture meters/sensors positioned at specific locations above the conveyor belt determines the moisture content of furnish.



Figure 1.5: Storage silos for classified wood furnish. Source: Sam-Brew 2016.

1.2.4 Adhesive — types and quantities used

The most common and inexpensive adhesive used for particleboard manufacture is urea formaldehyde (UF); other resins such as melamine urea formaldehyde (MUF) and phenol formaldehyde (PF) resins are also used. These are thermosetting resins which are supplied typically as water-based solutions with 50% to 60% solids. UF resin, aside from being cheap, is fast curing (implying shorter press times) and provides a colorless glueline. It is made from a combination of urea and formaldehyde and has high pre-cure tackiness required to hold particles in place as they are transferred from point to point in the production of particleboard. UF resins however have some disadvantages such as their low resistance to moisture hence its use for interior products, and continuous formaldehyde emissions while in use because of the hydrolysis of the

weakly bonded formaldehyde. These disadvantages of UF resin can be reduced by fortifying it with melamine which has a low solubility in water but is more expensive compared with urea. PF resins formed by a reaction of phenol with formaldehyde on the other hand have more durable bonds which are resistant to moisture, thus low formaldehyde release after board production and as such their use for exterior products. The disadvantages of PF resins include its dark color, glue line and slow curing nature (compared to UF) requiring longer press times at higher temperatures.

The quantity of adhesive used for board manufacture is referred to as the mass resin load and is calculated based on the percentage of the resin solids content to the oven dry weight of the wood residue, and usually ranges from 6% to 12% for UF and PF resins (Irle et al. 2013). Different quantities of resin may be added to the face and the core furnish. In most cases, more resin is used in the face as the finer particles tend to consume more resin solids because of their greater surface area. But in general, the type and amount of resin incorporated in the face or core depends on the type of residue, the type of particleboard being manufactured and pressing parameters being employed. Additives such as hardeners/catalyst to enhance the rate of reaction of the resin, scavengers to reduce the formaldehyde emissions, wax to help boards repel moisture and fire retardants to delay combustion can be mixed with the resin for particleboard production. Depending on the mill, resin can be applied on a continuous basis where individual nozzle heads spray the resin, water and or additives into a blender using a computerized system, or in some cases these adhesive formulations are pre-mixed in known quantities in a chamber and applied directly into a blender.

1.2.5 Blending

Based on proven recipes, furnish from the silos is weighed into blenders (Figure 1.6) and resin is uniformly applied to the furnish via air atomizing spraying nozzles with the core and the face blended separately. Blenders are either rotary or have paddles installed in them to ensure thorough mixing of its constituents (Figure 1.7). Blending is done for approximately 2 minutes after the required amount of resin, water and or additives have been sprayed on the furnish to ensure a uniform application of these to every particle. The resinated furnish is conveyed on belts to forming

bins. Moisture meters measure the MC of furnish as it leaves the blender while magnets above the conveyor belt remove any ferro-magnetic objects that might still be in the blended material (Figure 1.8).



Figure 1.6: Furnish being transported into blender. Source: Sam-Brew 2016.



Figure 1.7: Batch blender with several hoses/nozzles for spraying resin and additives. Source: Sam-Brew 2016.

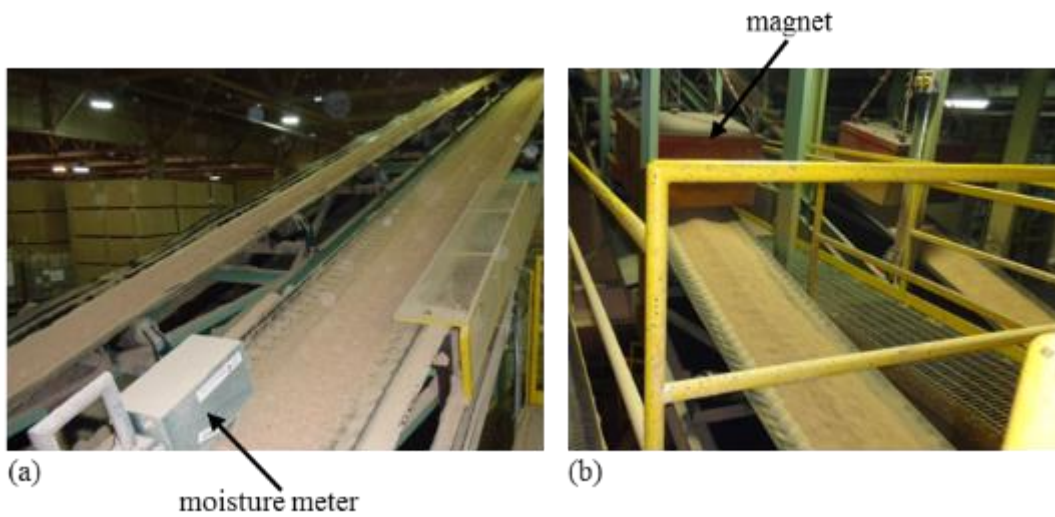


Figure 1.8: Blended furnish with a) moisture meter and b) magnet positioned above conveyor belt. Source: Sam-Brew 2016.

1.2.6 Mat forming

The resinated furnish must be uniformly laid along the length of the mat and across its width (to specific thicknesses and densities) as variations in these greatly affect particleboard density which in turn affects other board properties. Most commercial particleboards are produced in a 3-layer mat comprising of fine face material followed by a coarse core material and finally fine face material. Therefore 3 or 4 forming bins (2 face and 1 or 2 core) are positioned apart from each other in the order in which the mat is to be formed (Figure 1.9). Forming bins differ based on the principle that is employed to randomly orient and lay down specified quantities of furnish, common ones being air and textured rollers. With air formers air flow is used to uniformly blow furnish to form mats, while the textured rollers rotating at uniform speed and set distance apart ensure material which falls off it are evenly laid into mats.



Figure 1.9: Forming bin with resinated face furnish. Source: Sam-Brew 2016.

The mats are formed in a continuous process on conveyor belts (caul-less lines) or in a slightly less continuous process on caul plates (tempered steel plates) (Figure 1.10). Thickness allowances are made during mat forming to permit sanding of the particleboards after hot pressing. Trimmers with extractors are situated on the edge of the mat towards the end of the forming line to trim the

mats to size and scrape off excess material which is recirculated into the forming bins (Figure 1.11). Moisture readings are taken again for the formed mat as it is transferred to the next processing stage. Keeping track of the MC of the furnish throughout the initial stages will help operators to modify the pressing parameters that will be used in the hot pressing operation. More magnets located above the formed mats are used in a final attempt to remove any ferro-magnetic objects that might have been missed in the previous stages (Figure 1.12).

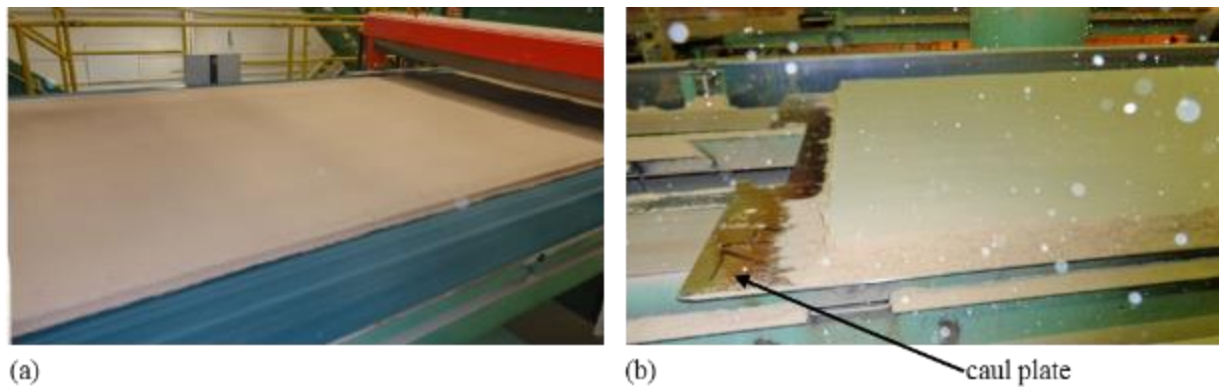


Figure 1.10: Mat formed on a) continuous caul-less line and b) caul plate. Source: Sam-Brew 2016.

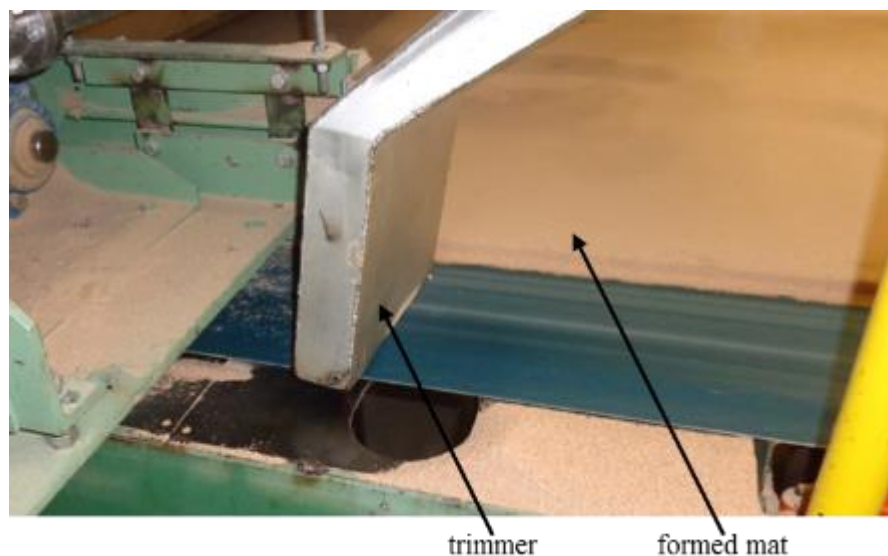


Figure 1.11: Trimming of mat edge on caul-less forming line. Source: Sam-Brew 2016.



Figure 1.12: Magnet located above formed mat to remove ferro-magnetic objects. Source: Sam-Brew 2016.

1.2.7 Prep-pressing

Before the formed mats are transferred into the hot press, they go through a pre-pressing stage designed to reduce the thickness of the mat by removing as much air as possible (Figure 1.13). This step is advantageous as a reduced mat size suggests the press does not need to open fully in order to have the mat deposited into it, and in turn corresponds to faster closing and production rates and lower chances of precure on the bottom face of the mat in contact with the heated press platen. Material that spills out on the edges of the mat are collected by an extractor and recirculated into the forming bins. The mats are cut to size depending on the press dimensions and transferred on conveyor belts to the press; the conveyors are equipped with scales to measure the weight of the mat.



Figure 1.13: Prep-pressing of formed mat using a system of rollers. Source: Sam-Brew 2016.

1.2.8 Hot pressing

Hot pressing of the particleboard can be done in a batch press or continuous press which is hydraulically operated by steam or oil. The batch presses are either single daylight with one opening to make a single particleboard or multi-daylight presses with multiple openings to make as much as 14 particleboards per press cycle (Figure 1.14). Most of these batch presses can make particleboards anywhere from 4-6 foot wide and 24 foot long. The continuous presses on the other hand as the name suggests produces one long particleboard and is a continuous process from mat forming, pre-pressing to hot pressing unlike the batch presses which requires mats to be transferred from conveyor belts or caul plates to loading racks known as pre-loaders prior to loading into the press. The pre-loaders ensure the mats are deposited in the press simultaneously.



Figure 1.14: Daylight press with multiple openings for hot pressing mats. Source: Sam-Brew 2016.

The hot-pressing cycle consists of a complex mix of parameters ranging from temperature, pressure, moisture content, mat thickness, density of the particleboard to be produced, the type of resin and residue being used. Particleboards are commonly manufactured with density and thickness ranges between 640–800 kg/m³ and 3 mm–25.4 mm (1/8–1 inch) respectively. The press temperatures employed are normally dependent on the curing behavior or characteristics of the resin being used; UF resin is usually cured between 150 °C to 165 °C and PF between 170 °C to 180 °C. Typical industrial press cycle times range from 3 minutes maximum for thinner boards to 5 minutes for thicker boards. During hot pressing, the press is closed fast to avoid pre-cure of the bottom face in contact with the platen and ensure quick contact with the top platen. Once contact is made with the mat, the press is adjusted to close slowly to generate steam in the face layers in contact with the heated platen and force the steam into the core to heat and cure the resin. It is to ensure a faster rate of heat transfer to the core that as mentioned in the residue processing section the face furnish MC is usually higher than that of the core furnish to create a moisture gradient between the face and core and produce a steam shock effect. The heating of the mat causes plasticization of the residue allowing it to be compacted to the desired thickness and activation of the resin for curing. When the mat reaches the designated thickness, either using metal stops or a computerized system, the press is maintained at that position to allow the resin to cure and permit

internal relaxation of the compressive stresses that were generated as a result of compaction; this is referred to as the cooking period and is based on the resin cure chemistry. After the cooking period, a decompression stage is initiated for a few seconds to degas the high vapor pressure within the particleboard. This is essential to prevent delamination (blows or blisters) once the press opens and the internal gas pressure within the particleboard is more than its bond strength.

1.2.9 Edge trimming, cooling, sanding and cutting to size

After the hot-pressing operation, panels in the continuous press system are transferred to the trimming stations. With the multi-daylight presses the panels are off loaded unto loading racks (similar to the pre-loaders) which then unloads the boards onto a conveyor system that transports the boards to the next manufacturing step. The conveyor belt to the trimming station is equipped with sonar or ultra sound transmitters (Figure 1.15) to help detect blisters/blows within the particleboards. This will enable the press operators to adjust the production parameters (such as MC, resin content) and or press cycle for subsequent loads to prevent further delamination. The conveyor belt is also fitted with weight scales and thickness gauges which measures each particleboard to ensure they meet the target weight and thickness requirement; boards that do not are automatically rejected and pushed off the belt into a reject bin. Acceptable particleboards are transferred to the trimming station where the length and width of the boards are trimmed to removed edge effects and square the boards. Approximately 2 to 3 inches (50–76 mm) is cut from each edge. The waste generated at this stage and the rejected boards are sent back to the front end of the production line where they are crushed and added to the raw material storage bins.

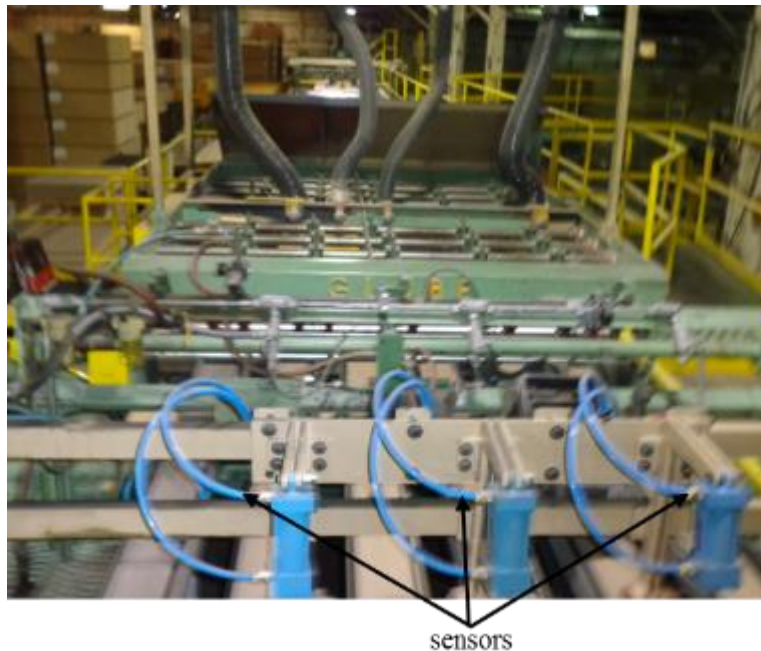


Figure 1.15: Sensors for blister detection and weight measurements fixed to conveyor system. Source: Sam-Brew 2016.

The trimmed particleboards are conveyed to wicket or star coolers where they are stacked to allow the boards to cool (Figure 1.16). This is specific to the type of resin used for board manufacture. UF resins have low heat tolerances therefore the cured resins tend to breakdown when exposed to excessive heat; hence particleboards made with UF resins are cooled soon after they exit the press. PF resins have higher temperature tolerance and as mentioned earlier require longer press times to cure the resin. Thus, particleboards manufactured with PF resin are hot stacked shortly after leaving the press to ensure further curing of the resin. Similarly, caul plates used in multi-daylight presses are also cooled down before mats are formed on them to avoid pre-cure of the resinated furnish. After cooling the particleboards are off-loaded into stacks on a series of automated roller conveyor systems which serves as intermediary storage before the stack is transported to the sanding station.



Figure 1.16: Cooling wicket for manufactured particleboard. Source: Sam-Brew 2016.

As mentioned previously, tolerances in thickness allow for sanding of approximately 0.5 mm off each face for thinner boards and 1 mm for thicker boards. Sanding of the particleboards is done to remove pre-cured material off the board surface and to bring the boards to the desired thicknesses. Board sanding is done in two stages - a primary rough sanding with coarse grit sandpaper to remove the pre-cured surface, grooves and any surface defects resulting from the caul plates or conveyor belts such as fissures, and a secondary sanding with fine grit sandpaper to smoothen the particleboard surface to allow for lamination (Figure 1.17). Thickness gauges situated on the conveyors as the particleboard leaves the sanding belt measure board thicknesses to ensure they are within the specified range. The sanded particleboards are then conveyed to another station where a series of saws cut the boards to standard sizes of 4 foot by 8 foot, or other specialty product size requirements as requested by the customer, mostly secondary manufacturers. Boards are then stacked, strapped/packaged and labelled. The stacks are manually arranged using forklifts in a warehouse awaiting shipping to customers.



Figure 1.17: Sanding of particleboard. Source: Sam-Brew 2016.

1.2.10 Quality control

In all particleboard mills quality control (QC) measures have been put in place to ensure boards of excellent quality that meet standard requirements are being produced. The QC department selects particleboards at random and at specified intervals for each product type to conduct physical and mechanical property tests according to recognized particleboard standards such as the American Society for Testing and Materials (ASTM) and American National Standards Institute (ANSI). These tests include density measurements, vertical density profiles, internal bond, modulus of elasticity and rupture, thickness swell, water absorption, linear expansion, screw withdrawal and formaldehyde emissions. The standards specify particleboard grades (high, medium and low density panels) and stipulates relevant physical and mechanical property requirements that particleboards in the various grades must conform to (Table 1.1). The information provided from these tests enable operators to optimize the production parameters where the particleboard properties fall below stipulated limits.

Table 1.1: ANSI A208.1-2009 particleboard grade requirements. H= high density ($> 800 \text{ kg/m}^3$), M= medium density ($640 - 800 \text{ kg/m}^3$), LD= low density ($< 640 \text{ kg/m}^3$), PBU=underlayment, D=manufactured home decking. ns- not specified.*

Grade	Modulus of rupture MOR (MPa)	Modulus of Elasticity MOE (MPa)	Internal bond IB (MPa)	Linear expansion max. avg. (%)
H-1	14.9	2160	0.81	ns*
H-2	18.5	2160	0.81	ns*
H-3	21.1	2475	0.90	ns*
M-0	7.6	1380	0.31	ns*
M-1	10.0	1550	0.36	0.40
M-S	11.0	1700	0.36	0.40
M-2	13.0	2000	0.40	0.40
M-3	16.50	2750	0.55	0.35
LD-1	2.8	500	0.10	0.40
LD-2	2.8	500	0.14	0.40
PBU	11.0	1725	0.40	0.35
D-2	16.5	2750	0.55	0.30
D-3	19.5	3100	0.55	0.30

Source: American National Standards for Particleboard

1.3 Particleboard industry

The two main drivers for the particleboard industry are the furniture industry (especially flat-pack furniture) where it is used in the manufacture of cabinets, desks, shelves etc. and the construction sector mainly for interior wall paneling, flooring and stair tread applications. Particleboard consumption is therefore strongly tied to the housing industry. In 2014 the total production capacity of particleboard mills in North American was estimated at $8,191,000 \text{ m}^3$, and with a 7.2% growth in the housing and renovation statistics particleboard consumption also increased by 8.5% (Eastin et al. 2015; Wood Based Panels International 2015). In 2016 and beyond, production capacities are predicted to increase to approximately $8,981,000 \text{ m}^3$ per annum with majority of the increase expected in the United States with Aracuo North American's new line to be built in

Grayling, Michigan and in Canada, an increase in the production capacity of Uniboard's existing line in Val d'Or, Quebec.

In 2015, a total of 19,433,000 m³ wood-based panel products including particleboard, MDF (including hardboard), OSB, plywood, and veneer sheets were produced in Canada with particleboard making up 45% of the total volume (Figure 1.18 and Appendix A).

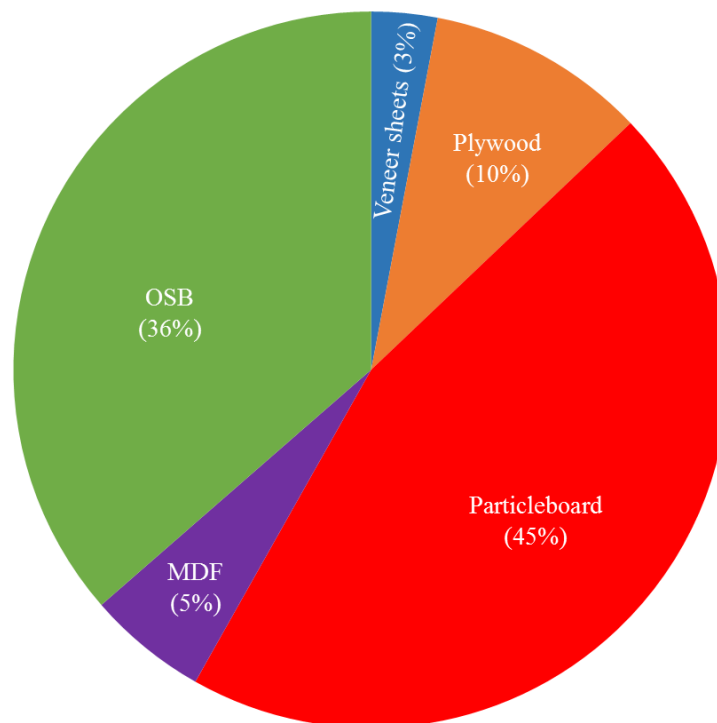


Figure 1.18: Total wood-based panel production in Canada 2015. Source: United Nations Economic Commission for Europe (UNECE) Committee on Forests and the Forest Industry. 2016.

Currently, there are 5 particleboard companies in Canada (Table 1.2) with an estimated production of 8,796,000 m³ in 2015, an increase of 2.4% from that reported in 2014 (UNECE Committee on Forests and the Forest Industry 2016). Table 1.3 provides information on the production quantities, import and export values of Canadian particleboard from 2012 to 2015.

Table 1.2: Canadian particleboard companies, locations and product specifications.

Company	Location	Grades	Thickness range (mm)	Density range (kg/m³)
Arauco North America	St Stephens, New Brunswick	M0, M1, MS, M2, M3I	3-28.6	673-753
Northern Engineered Wood Products	Smithers, British Columbia	—	11.1-25.4	641-689
Panolam Industries International Inc.	Huntsville, Ontario	M3I	6.35-38.1	673-721
Tafisa Canada Inc.	Lac-Megantic, Quebec	M1, MS, M2, M3I	6.35 38.1	609-705
Uniboard	Sayabec, Quebec	M0, M1, MS, M2, M3I	6.35-44.45	449-689
	Val-d'Or, Quebec	M0, M1, MS, M2, M3I, LD1, LD2	9.5-28.58	577-689

Source: surface & panel buyers guide 2016

Table 1.3: Canadian particleboard production and trade from 2012 to 2015.

Year	Production (m³)	Import Quantity (m³)	Import Value (1000 \$US)	Export Quantity (m³)	Export Value (1000 \$US)
2012	7,446,000	452,000	86,796	4,192,000	1,085,040
2013	7,968,269	604,983	95,800	4,770,831	1,466,886
2014	8,587,000	1,035,829	102,993	5,278,301	1,296,520
2015	8,796,000	1,227,000	—	5,698,628	1,265,568

Source: FAO, United Nations Statistics Division 2016

It is evident from Table 1.3 that particleboard production has been steadily increasing from 2012 to 2015. Data provided by the Policy, Economics and Industry Branch of the Canadian Forest Service, Natural Resources Canada indicates that production volumes are projected to increase by

8.5% to 9,540,000 m³ at the end of 2016, with further increments to 10,107,00 m³ by 2017 (UNECE Committee on Forests and the Forest Industry 2016) (Appendix A). The apparent consumption of particleboard however decreased by 0.5% from 4,345,000 m³ in 2014 to 4,324,000 m³ in 2015 (UNECE Committee on Forests and the Forest Industry 2016). The quantities of particleboard exported from Canada continues to grow with the United States being its major market (Eastin et al. 2015); 99.8% of the Canada's particleboard export in 2014 was reported to have gone to the United States.

The demand for and consumption of particleboard for products such as door cores, kitchen cabinets and furniture can be expected to grow with the steady rise in North American housing statistics.

1.3.1 Major industry problem

A major problem panel manufacturers face is increasing material costs for both resin and wood residue. The resin cost over the years has been the result of rising oil prices globally. The growing wood costs owing mainly to the closure of several sawmills which led to limited residue supply; other contributing factors include the higher cost of hauling the residues over long distances and competition for the residue with pellet manufacturers. In 2013 for instance, particleboard mills in eastern Canada encountered problems with non-uniform supply of wood residue. Residue was being sourced from several locations (300-mile radius) resulting in a wide variability in the wood residue obtained. This in turn required more resin consumption to compensate for the furnish variability and maintain board properties (M. Feng, personal communication, November 29, 2013). These factors resulted in an increase in the production cost.

To help address the issues associated with the wood residue supply and haulage, other abundant suitable residues which can support or absolutely replace wood in particleboard production have been considered.

1.3.1.1 What has been done so far

The idea of exploiting other non-wood natural resources has long been and continues to be a subject for research and development. Several studies have been conducted over the years to

ascertain the viability of using a wide variety of agricultural crop and plant residues (Youngquist et al. 1994); particularly so in countries such as Egypt, Greece, Turkey and India where the residues are abundant.

There are several classifications for non-wood natural resources based on their botanical and agricultural grouping, their technological processing or the part of the plant from which they are obtained such as the bast, leaves, fruits and or seeds (Figure 1.19) (Anandjiwala and Blouw 2007; Batra 2007).

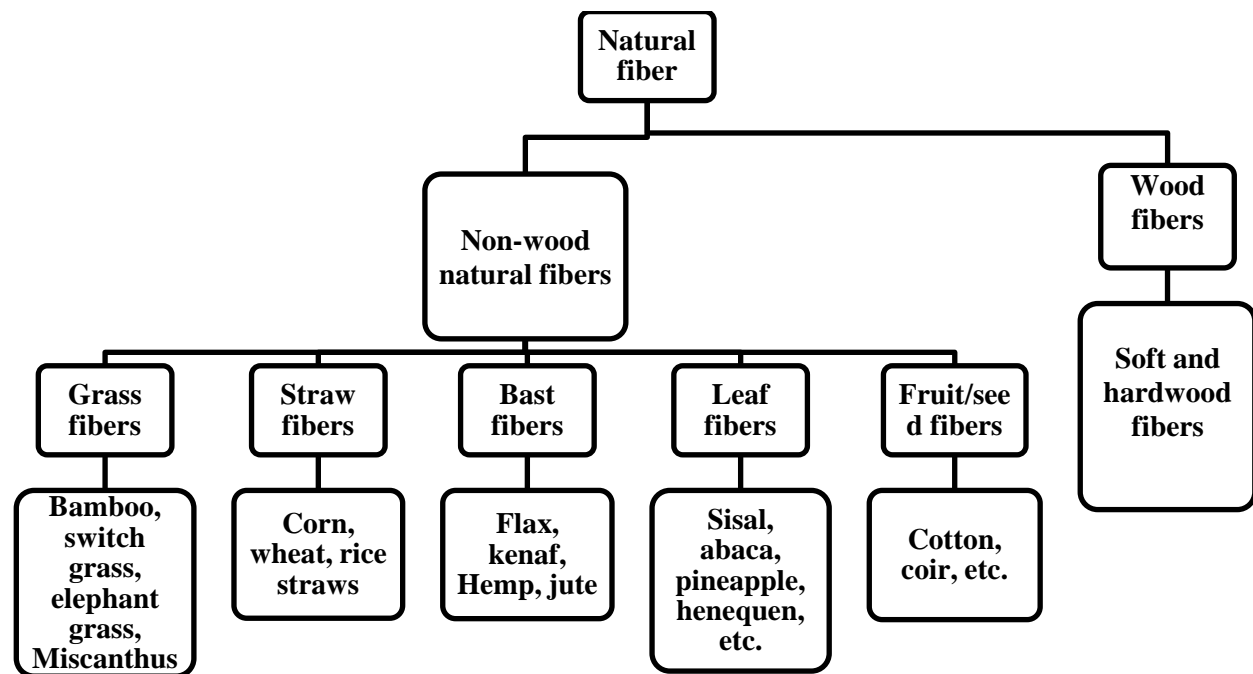


Figure 1.19: Non-wood natural fiber classification. Source: Anandjiwala and Blouw 2007.

Most of these plants and or agricultural crops are grown for food or fibers and their residues used as fodder or beddings for livestock, others plowed back into the soil, some as raw materials for composite panels, pulp and paper, textiles and a few as biomass for ethanol production. These residues are renewable, biodegradable and have the advantage of a low density.

A number of non-wood residues for example rice husk (Gerardi et al. 1998), cotton stalks (Guler and Ozen 2004), peanut shells (Batalla et al. 2005), jost tall wheatgrass (Zheng et al. 2007), pepper

stalks (Guntekin et al. 2008), waste grass clippings (Nemli et al. 2009), date palm fronds (Hegazy and Aref 2010), wheat straw (Azizi et al. 2011), tobacco stalk (Acda and Cabangon 2013), sycamore leaves (Pirayesh et al. 2015) have been investigated as potential wood substitutes for the production of particleboard.

1.3.1.1.1 Particleboard from non-wood residues only

Some of the studies in this area have focused on using only non-wood residues for particleboard products. For instance, in 1997, Markessini et al. (1997) as part of a European research project aimed at the development of environmentally friendly composites for furniture and construction, worked on the production of particleboard from wheat straw. The panels produced were reported to conform to European standards for particleboard materials. Han et al. (1998) went further to manufacture particleboard from reed and wheat straw residues specifically studying the effect of board density and particle size on panel properties. In 2002, Wang and Sun (2002) evaluated the possibility of producing low density boards from wheat straw and corn pith focusing on the straw particle size and press time. The main aim of their study was to characterize the tensile and compressive strength properties of the low density boards. The results indicated an increase in tensile and compressive strength with an increase in board density and particle size. Nemli et al. (2003) studied the anatomical and chemical properties of kiwi stalks and investigated the suitability of the stalks for particleboard manufacture. They concluded based on the trials that kiwi pruning had the potential to be used for particleboard production but further research was required in terms of their dimensional properties.

In 2004, Papadopoulos et al. (2004) examined the feasibility of manufacturing one layer particleboard from bamboo chips and concluded that bamboo could effectively replace wood in the manufacture of interior grade particleboard. Research conducted by Alma et al. (2005) on the production of a 3-layered particleboard (coarse-fine-coarse) from chips of cotton carpels produced panels which met the minimum requirements for general grade particleboard. Kalacioglu and Nemli (2006) investigated the feasibility of using kenaf stalks for a 3-layered particleboard and concluded the ratio of face-to-core material, the press temperature and pressure significantly affected the board properties; the panels produced satisfied the minimum requirements for internal

bond and modulus of rupture (MOR) as stipulated in the European Standards. In 2007 studies carried out by Cöpur et al. (2007) indicated that hazelnut husk could also be considered as an alternative lignocellulosic residue for particleboard manufacture; the addition of water repellents was however recommended to reduce the thickness swelling and water absorption rates.

With the aim of developing light-weight panels with lower environmental impacts, Balducci et al. (2008) considered the production of one layer particleboards from hemp hurd, sunflower stalks, topinambur, maize and miscanthus. From their results the single-layer boards met the internal bond strength requirements outlined in the European standards but not those for modulus of elasticity (MOE) and rupture (MOR). In 2009, Pariah et al. (2009) evaluated the possibility of using kenaf bast fiber and core materials in a multi-layered particleboard configuration. The results of the study indicated that homogenous boards manufactured from kenaf core were suitable substitutes for wood in particleboard production. Xu et al. (2009) also evaluated the effect of wax on the dimensional stability and mechanical strength properties of particleboards fabricated from bagasse (processed sugar cane residue) and concluded the addition of wax had no recognizable negative effect on bending strength properties.

Particleboard manufactured from corn cob was also assessed, and after a series of trials corn cob was suggested as a sustainable alternative to traditional sound insulation materials such as glass or rock wool (Faustino et al. 2012). Mixtures of rice straw and coir fibers in different mass ratios have also been studied to produce low cost particleboard panels with higher strength properties (Zhang and Hu 2014). Using a combination of Cattail (*Typha latifolia* or *Typha angustifolia*) a herbaceous plant that grows in marshy areas and wheat straw, Bajwa et al. 2015 also investigated the feasibility of manufacturing low density particleboard.

1.3.1.1.2 Particleboard from admixtures of wood and non-wood residues

There have equally been many studies geared towards the viability of combining non-wood residues with wood in the production of particleboard. In these studies, different proportions of both wood and non-wood residues are mixed together to create a layered composite and/or a relatively homogenous board. Troger et al. (1998) attempted to reinforce particleboard panels by careful combination and layering of wood (spruce, beech, and poplar), miscanthus, flax fiber and

straw. The wood and miscanthus chips were mostly incorporated into the face and core layers while the flax fiber and straw were investigated as reinforcements in the face layers. Their results indicated that admixtures of wood and miscanthus were suitable for the manufacture of particleboards; boards with flax fiber and straw reinforcements yielded consistently higher bending strength properties compared with those made solely from wood chips.

In 2001, Philippou and Karastergiou (2001) fabricated 3-layered particleboards using different percentages of Poplar flakes and several non-wood residues (reed, kenaf, miscanthus and cotton stem particles) in the core (50:50%) and face (50:50 or 75:25%) layers. The work was aimed at the production of low density environmentally friendly building materials. They concluded that the selected non-wood residues could satisfactorily supplement wood in particleboard production. Admixtures of vine pruning and industrial wood particles in the core of a 3-layer particleboard were also evaluated by Ntalos and Grigoriou (2002); based on their results vine pruning had a negative effect on the panel's strength and dimensional properties. However, the mechanical properties of boards made of 100% vine pruning exceeded the stipulated minimum for European standards. In another instance, single-layer particleboards consisting of various mass ratios of wood chips and flax shive (9:1, 4:1 and 7:3) mixtures were manufactured to determine the feasibility of flax shive as an alternative lignocellulosic residue for panel production (Papadopoulos and Hague 2003). Supplementing wood with up to 30% flax shive resulted in particleboards which satisfied the relevant European standards for interior grade boards.

Three-layered general purpose particleboards were fabricated by Bektas et al. (2005) from several combinations of sunflower stalks and Poplar particles (25:75%, 50:50% and 75:25% respectively) and tested for their physical and mechanical strength properties. The studies revealed that increasing the percentage of sunflower stalk chips in the mixture resulted in panels with low strength properties; notwithstanding only their mechanical properties (MOE, MOR, internal bond and screw holding strength) complied with the minimum requirements stipulated by the Turkish Standards for general grade particleboard. In a similar study Guler et al. (2006) manufactured 3-layered particleboards from admixtures of sunflower stalks and Calabrian pine and established that panels made with 50:50% mass ratios produced boards with the highest mechanical strength properties for interior grade particleboard.

In another study by Guler et al. (2008), 3-layered particleboards were produced from a mixture of peanut hull and European Black Pine (*Pinus nigra* Arnold) wood chips with different mass fractions. Laboratory tests revealed a general trend where an increase in peanut hull invariably resulted in a decrease in physical and mechanical strength properties. By means of single-layer boards Nemli et al. (2009) evaluated the feasibility of grass clippings (*Lolium perenne* L.) and Eucalyptus particles (*Eucalyptus camaldulensis* Dehn.) for furniture grade particleboards. Various combinations of grass-to-Eucalyptus were manufactured and tested in accordance with the European standards. Panels with no more than 13% grass clipping result in boards with acceptable panel properties.

Combinations of various concentrations (10, 20, 30, 50 and 100 %) of hemp hurd, bagasse or canola residues with wood chips were also considered by Nikvash et al. (2010) for the core layer in the production of a 3-layer particleboard panel. The face layers were made purely of wood chips. Except for panels produced from canola residues, the bending strength and dimensional properties of boards containing up to 50% hemp hurd or bagasse were in accordance with the European Standards for general purpose particleboard. The researchers recommended further studies to examine the feasibility of canola residues as alternative residue for particleboard manufacture. In 2011, Ghalehno et al. (2011) studied the effect on panel bending properties of a 3-layered particleboard when bagasse particles were incorporated in the face layers and wood chips in the core at mass ratios of 3:7 and 4:6 respectively. Particleboards with 40% bagasse particles were identified to have optimum bending strength properties in accordance with European standards.

Reh (2013) tested the physical and mechanical strength properties of a 3-layered particleboard whose core comprised of a mixture of wood particles, hemp hurd and cereal straw. Several mass ratios (0 to 30%) of hemp hurd or cereal straw was mixed with wood particles. The results indicated that a maximum of 20-30% hemp hurd and 10-15% cereal straw was ideal to produce general purpose particleboard. Particleboards containing different mass ratios of rubber wood and kenaf were evaluated by Paridah et al (2014) and panels comprising of the 50:50% mass ratio yielded the highest bending strength properties. Using a combination of sorghum stalks as the face material and industrial hardwood chips for the core Khazaeian et al. (2015) produced 3-layered particleboards by varying the particle sizes, material mass ratios (10% and 50%), UF resin content, press time and temperature. The particleboards containing a greater mass ratio of sorghum stalks

exhibited higher mechanical strength properties which exceeded the European norm for general purpose particleboard.

1.3.1.1.3 Resin for particleboard manufactured from non-wood residues

Various studies have proven the inability of UF resin to satisfactorily bond with agricultural crops and plant residues (Grigoriou 2000; Alma et al. 2005; Ye et al. 2007). This is because most of these plants and agricultural crop residues have waxy outer surfaces which hinder a combination of the residues with conventional resins, usually resulting in a poor interfacial interaction and composites which exhibit poor mechanical properties. Some studies using PF resin reported adequate bond strengths (Batalla et al. 2005; Cöpur et al. 2007; Tabarsa et al. 2011).

Ultimately, polymeric diphenyl methane diisocyanate (pMDI) resin which can penetrate the waxy outer layer of most of the agricultural residues has been recommended and established to form effective bonds with such residues (Tröger et al. 1998, Wang and Sun 2002; Mo et al. 2003; Pan et al. 2006; Balducci et al. 2008; Bajwa et al. 2015). Dow Bioproducts Ltd. previously known as Isobord based in Elie, Manitoba was one of the first companies in Canada to successfully produce particleboard on an industrial scale from wheat straw bonded with pMDI.

1.3.2 What remains to be done

The literature that has been reviewed shows the profound interest and enormous strides that have been made globally in the search of alternate raw materials for particleboard production. Majority of the studies has been centered in regions that have vast resources of agricultural crop and plant residues such as Greece, Turkey, Malaysia, India, Iran, Australia, Thailand, USA, Germany, Philippines, China, Egypt, Italy, Hungary and Brazil. In Canada, there is a need to research further into nationally available agricultural or plant residues that can help address wood fiber shortage issues that arise.

Aside the residue availability issues there are other problems that still need to be dealt with. These are discussed below:

1.3.2.1 Strengthening particleboard panels

Most of the particleboard research conducted has focused on random blends of discontinuous milled furnish. Particleboard happens to be a composite with a great deal of dimensional stability issues. It experiences the most deformation with time in response to applied loads, a behavior known as creep, and produces great changes in dimension with increase in moisture and temperature; both situations lead to the shortening of the products life in service. Particleboard will therefore benefit from some form of reinforcement within the board. So far only Tröger et al. 1998 have considered the use of non-wood natural fibers in a continuous mat form for particleboard fortification. Sahoo et al. 2012 have also proposed the use of jute felt to stabilize plywood core. This layered configuration is on the other hand more common for bio-composites where it has been reported to improve board strength properties (Burgueño et al. 2005; Sapuan et al. 2006; Liu and Hughes 2008; Abdul Khalil et al. 2011; Behera et al. 2012; Porras and Maranon 2012).

1.3.2.2 Identifying resins capable of bonding non-wood residues

With regards to resin, there has been growing concerns about formaldehyde emissions from the formaldehyde-based resins used in the manufacture of wood composites and their effect on the human respiratory system. Regulations have therefore been implemented, specifically the Airborne Toxic Control Measure (ATCM) legislature by the California Air Resources Board (CARB), to help control the amount of formaldehyde emitted from hardwood plywood, particleboard, hardboard and medium density fiberboard. This legislation covers formaldehyde-based resins, ultra-low emitting formaldehyde resins (ULEF) and no-added formaldehyde resins (NAF). Consequently, most resin manufacturers have been pursuing and making developments in innovative resin formulations with either ULEF or NAF with the aim of satisfying the current market needs. The drawback of most of these resins include their low degree of moisture resistance and inability to successfully bond non-wood residues to meet panel quality standards. There is the need to investigate alternative resins with improved adhesion and limited formaldehyde emissions to effectively bond non-wood residues for particleboard production.

1.3.2.3 Reduction of pMDI quantities for board manufacture

Polymeric diphenyl methane diisocyanate (pMDI) resin mentioned earlier is a non-formaldehyde emitting resin which has gained popular use with agricultural crop and plant residues in the manufacture of composites especially because of its ability to penetrate the waxy outer layer of these residues. Presently this resin which costs \$1.8-\$2.2/kg is expensive compared to UF (\$0.9-\$1.1/kg based on liquid UF) and PF (\$1.45) resins. Even though lower dosages of pMDI (3-6% resin mass load) are used for board manufacture compared to the UF resin (8-12% resin mass load) its cost has been a major hindrance to its wider industrial acceptance and use in most particleboard plants, except for specialty products such as moisture resistant boards reserved for niche markets. A case in point being Dow Bioproducts Ltd/Isobord which unfortunately closed in November 2005 with one of its challenges being the resin cost and consumption. Typically reducing the quantities of resin required for panel production is not preferred since it leads to boards that do not meet the relevant standards. But moving forward it is essential to ascertain if a reduction (no matter how minor) in the present quantities of pMDI used in particleboard production can be achieved without compromising board integrity.

1.3.3 Proposed means of tackling the issues identified

From the previous section, it is evident the problems and needs of the particleboard industry that were identified can be categorized under two main points – residue and resin problems. This section proposes means of dealing with these issues.

1.3.3.1 Alternate non-wood residues available in Canada

To address the issue of alternate residue for particleboard production in Canada, flax (*Linum usitatissimum*) and industrial hemp (*Cannabis sativa* L) crops have been identified among the agricultural crops available (aside barley, wheat, corn, canola and oats) as possible raw material for particleboard manufacture.

1.3.3.1.1 Flax crop

Flax is mainly grown in the western Canadian prairies (Alberta, Saskatchewan, and Manitoba). The total production of flax crop in western Canada in 2015 (Figure 1.20) was estimated at 816,200 tonnes (Flax Council Canada 2016) a sizable increase over that produced in 2013, giving a positive outlook for the flax crop in the coming years. Furthermore, a web-based tool known as the Biomass Inventory Mapping and Analysis Tool (BIMAT - www.agr.gc.ca/atlas/bimat) has been developed by Agriculture and Agri-Food Canada (AAFC) to provide approximations of the amount of flaxseed produced and the quantities of flax straw biomass available within a specified distance. For instance, using BIMAT a total of 85,463 tonnes of oven dried flax straw residue per year was identified within a 161 km (100 mile) radius of Saskatoon. Similarly, 126,517 tonnes per year oven dried flax straw residue was identified within the Winnipeg area. The average cost per tonne to transport the flax straw residues ranges from \$28-\$34.

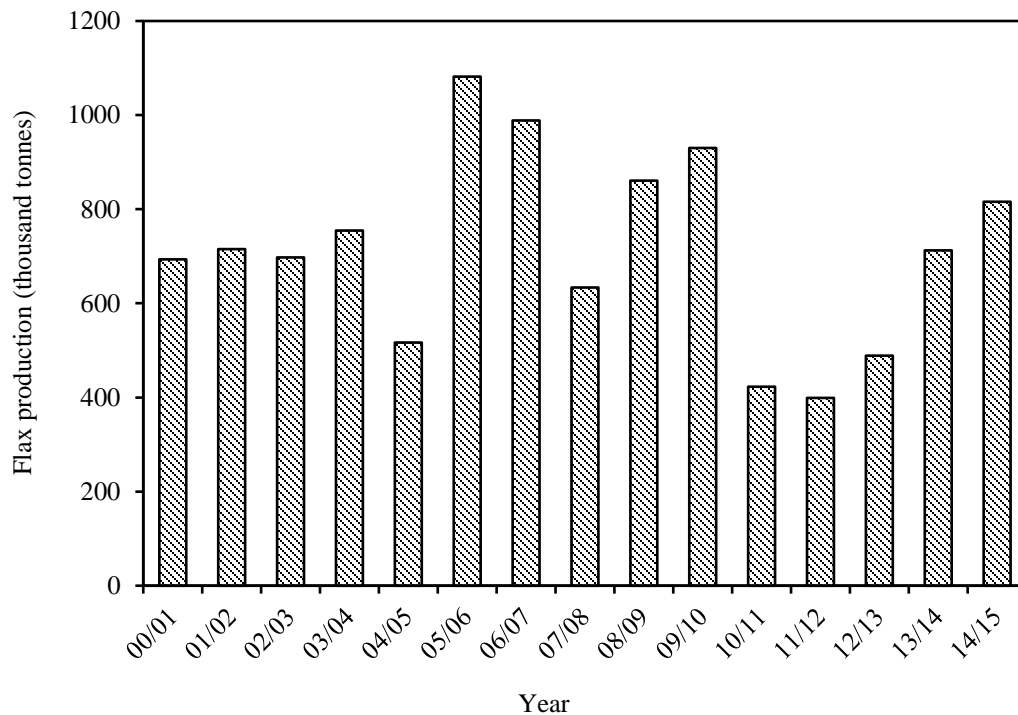


Figure 1.20: Total production of flax in western Canada. Source: Flax Council Canada 2016.

1.3.3.1.2 Hemp crop

Following the legalization of industrial hemp cultivation in Canada in 1998, several companies, universities and provincial governments have taken a renewed interest in its production and processing. In 2013 there were over 100 farmers mostly in western Canada and a few in the central region (Alberta, Saskatchewan, and Manitoba, Ontario and Quebec) who cultivated hemp (AAFC 2013). As of 2007 a total of 4,684 hectares of land was licensed for hemp cultivation and a total of 876 tonnes of hemp fiber was exported. A hectare of hemp is projected at producing an average of 6 tonnes of straw from which approximately 1.5 tonnes of fiber can be extracted (AAFC 2013). The total hemp seeded area in Canada between 1998 and 2011 is shown in Figure 1.21. The Canadian Hemp Trade Alliance (CHTA) reports that 33,000 hectares of land was licensed for hemp cultivation across Canada in 2015 (CHTA 2016). This suggests an estimated annual hemp straw of 190,000 tonnes available nationally.

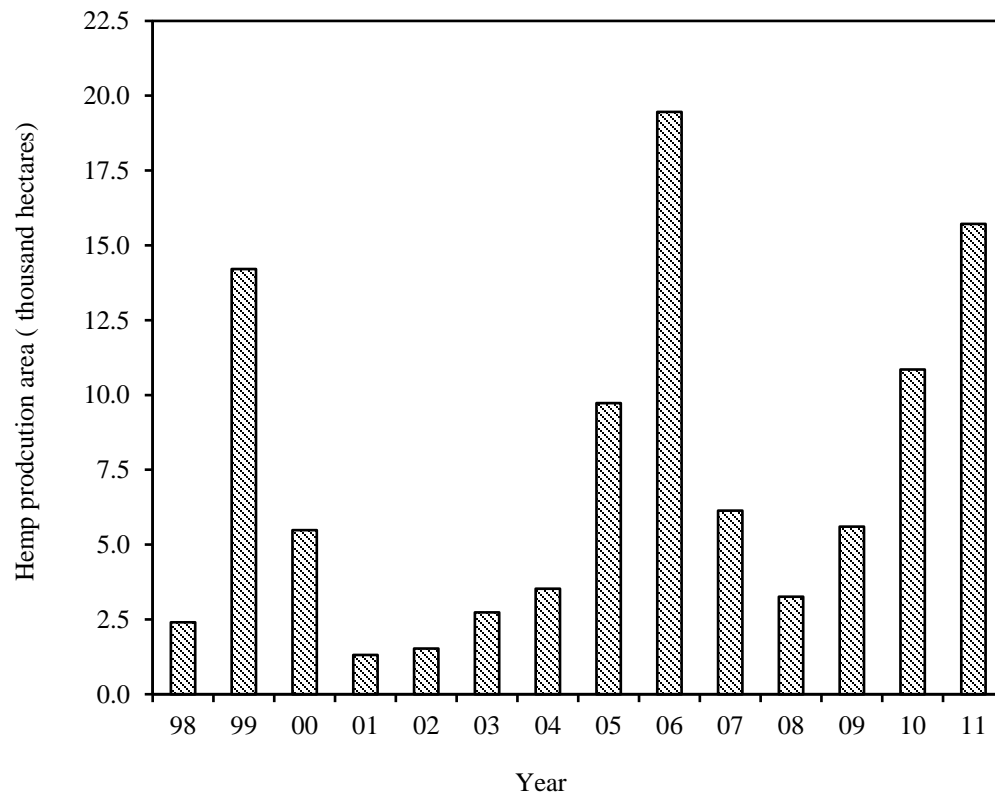


Figure 1.21: Total hemp seeded area in Canada. Source: Laate 2012.

1.3.3.1.3 Flax and hemp straw morphology

In terms of morphology the flax stalk grows to a height of about 0.5 to 1.25 m and the hemp up to 4.5 m, with stem diameters ranging from 1.6 to 3.2 mm and 4 to 20 mm respectively (Batra 2007). The structures of the flax and hemp stalk are similar consisting of an outer waxy bark serving as a protective layer, a layer of bast fibers, a cambium layer and an inner woody tissue surrounding a pith cavity as illustrated in Figure 1.22 (Munder et al. 2005). Hemp has a pith cavity half the size of the stalk diameter compared to flax. The bast layer contains fiber bundles with hemp having longer and coarser fibers than flax. Basically, these fiber bundles are a collection of technical fibers (glued together by a weak pectin and lignin interface) which on a finer scale are also composed of 10 to 40 elementary fibers (Singleton et al. 2003) (Figure 1.21).

The woody core tissue known as the flax shive for the flax crop and hemp hurd for the hemp crop provides support for the plant during growth and is a porous material which comprises up to 75% of the stalk (Bismarck et al. 2005). Figure 1.23 shows a cross section of the flax shive and hemp hurd tissue as seen under a scanning electron microscope.

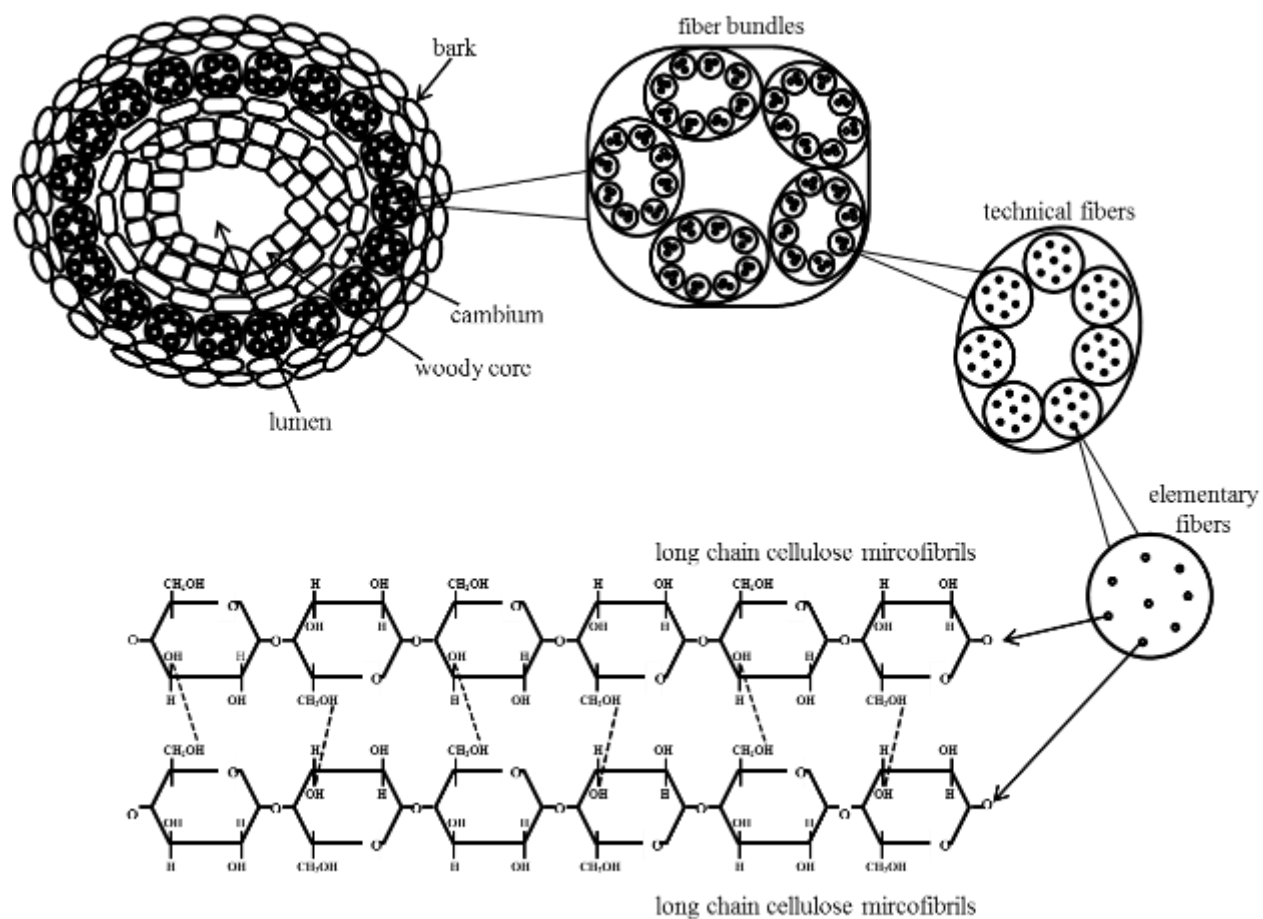


Figure 1.22: Schematic of the cross-section of a flax stalk showing the arrangement of bast fibers and woody core tissue. Modelled after Van Den Oever et al. 2000, Zimmermann et al. 2004 and Munder et al. 2005.

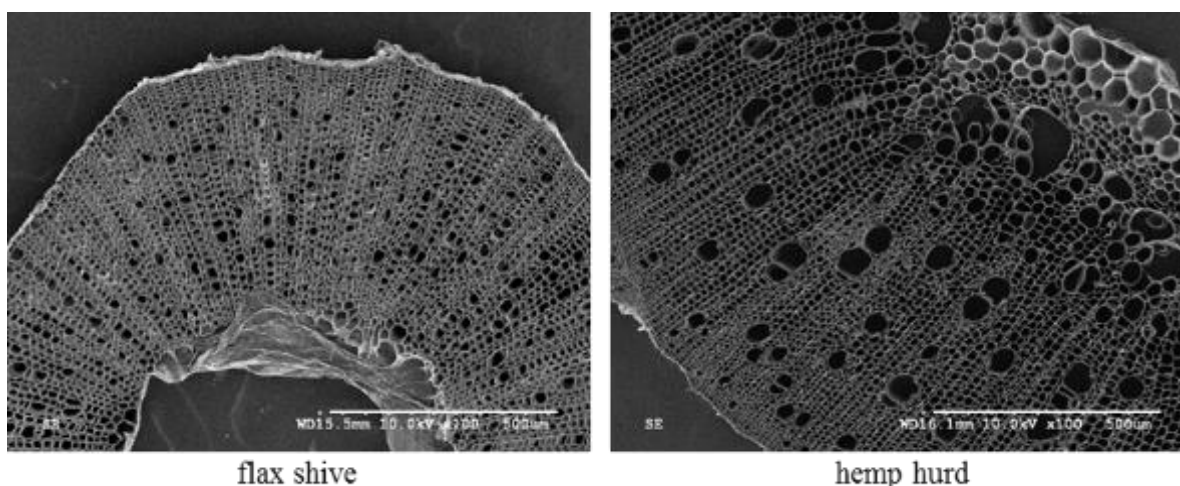


Figure 1.23: SEM image of the cross-section of flax shive and hemp hurd woody tissue. Source: Sam-Brew 2016.

The flax shive and hemp hurd residues like most lignocellulosic residues are composed of cellulose, hemicellulose and lignin. Other chemical components found in the residues include ash and extractives. The typical chemical properties for the flax shive and hemp hurd residues are presented in Table 1.4

Table 1.4: Chemical components of flax shive and hemp hurd residues.

Component	flax shive	hemp hurd
Cellulose (%)	53.27	44.2
Hemicellulose (%)	13.62	30.3
Lignin (%)	20.53	24.4
Ash (%)	3.53	1.4
Source	Rensten 2010	Stevulova&Schwarzova 2014

1.3.3.1.4 Processing of flax and hemp straw

Traditionally to obtain the bast fiber and woody residue the flax and hemp stalks are harvested and retted—a controlled degradation of the harvested stems (Goodman et al. 2002). Retting can be accomplished naturally through cold water (anaerobic bacterial activity) and dew retting (fungal

activity) on the fields, or chemically using dilute acids or bases (sulphuric acid or naphtha) (Batra 2007). Commonly used is the cold-water retting process where the stalks are submerged in cold water and through the natural decay process anaerobic bacteria cultures soften the pectin in the stalks allowing the separation of the fiber bundles in the bast from the woody tissue and bark material (Munder et al. 2005). The retted materials are then dried, open air or with dryers, and stored for a short period.

In recent years, the loosening of the bond between the fibers, bark and the inner woody portion has been accomplished through a mechanical process known as decortication. Decortication uses fluted rollers to break off the brittle woody core tissue and removes bark material (Munder et al. 2005). The breaking process also opens the fiber bundle to obtain technical fibers. At this stage, not all the woody core tissue is removed and the decorticated bast fibers also contain short fiber fractions (termed the tow). Depending on the end use and the level of purity required the stem maybe processed further through scutching (beating with blades) and hackling (combing to untangle and align fibers) to clean the fibers and open the technical fibers to obtain single elementary fibers (Van de Velde and Baetens 2001; Thomas et al. 2011).

The fractions obtained from processing the stalk comprise long staple fibers (up to 1 m) 20-30% by mass, 1-4% short tow fibers (0.1-0.5 m), 60-66% woody core tissue and 1-2% dust (Munder et al. 2005). The woody core tissue from the flax and hemp stalk, that is the flax shive and hemp hurd, as well as the long flax and hemp fiber portions can all be explored as raw material resources for particleboard production.

1.3.3.2 Resins for non-wood residues

With respect to issues surrounding resin cost, concerns about its effect on health and the environment, and its ability to bond non-wood residues, NAF and lignin-based resins have been identified and proposed as means of addressing this problem.

1.3.3.2.1 No-added formaldehyde resins (NAF)

Since the implementation of the ATCM regulations, several resins have been formulated and advertised to produce low emissions of formaldehyde or contain no formaldehyde. This research identifies one such resin, Acrodur® 950L, produced by BASF (a major chemical manufacturing company) which has been effectively proven to bond a wide range of agricultural fibers including kenaf, sisal, flax and hemp fiber in the automotive industry. This resin is proposed and evaluated in this research as an alternative to UF resin in effectively bonding non-wood residues such as flax shive and hemp hurd.

1.3.3.2.2 Lignin-based resins

Several studies have been published using different bio-based resins produced from biodegradable polymers such as cashew nut shell liquid, castor oil, soybean, wheat protein and tannins; these resins are used either alone or in combination with UF or PF resins for particleboard panels (Mo et al. 2001; Theis and Grohe 2002; Mao et al. 2011; Tabarsa et al. 2011; Valarelli et al. 2014). The main aim of these bio-based resins is to reduce the cost of production by substituting the expensive petroleum-based resins with less expensive naturally occurring materials. The concept of substituting proportions of pMDI resin with a cheaper natural binder such as lignin can also be explored as a means of making pMDI which is a non-formaldehyde emitting resin, a more cost-effective option for particleboard production from non-wood residues.

1.4 Research objectives

The primary aim of this research is to:

1. Investigate flax and hemp crop residues that are readily available in Canada as alternate natural resources which can supplement wood in particleboard production. The idea is to evaluate the viability of using both the woody core tissue and bast fiber portions obtained from processing the flax and hemp stalks for particleboard manufacture. The woody core tissue will contribute to addressing residue supply problems and the bast fibers will serve as a means of improving the strength properties of particleboard.

2. Evaluate resin options which are capable of efficiently bonding agricultural and plant residues to meet/exceed specified ANSI particleboard performance standards and environmental regulations.
3. Determine if there is room for further reduction of the current quantity of pMDI resin being used for particleboard production by itself or by substitution of a percentage of the resin with other natural polymers.

The overall motivation is to produce low density particleboards with improved strength properties by taking advantage of the low density and intrinsic strength properties of the flax and hemp crop residues. A decrease in board density for panel manufacturers hints at lower production costs in two major ways. Foremost is reduced energy savings as less energy will be required to compress the low density raw materials. Secondly, there will be transportation savings on the final product since transport of the boards from the mill to customers is by weight and not volume therefore more low density particleboards can be transported at a time.

1.5 Research structure

Subsequent chapters of this research describe experimental procedures through which the outlined research objectives were achieved.

Chapter 2 presents a comprehensive description of the residues and resins used in this research, methods of residue (flax shive, hemp hurd) processing and the particleboard manufacturing sequence. The chapter also includes detailed explanations on thermal analysis evaluations, short-term physical and mechanical property tests and information on the data analysis approach employed in the research.

To successfully use the Canadian grown flax shive and hemp hurd residues to supplement wood in particleboard production, Chapter 3 entailed the characterization of the flax shive and hemp hurd residues, preliminary experiments on the feasibility of fabricating 3-layered particleboards from these residues and analysis of the short-term particleboard properties in comparison to wood particleboards.

Chapters 4 and 5 explored the possibility of consolidating the flax shive and hemp hurd residues with a formaldehyde-free resin, Acrodur[®] 950L, and a pMDI-lignin resin formulation respectively, to produce particleboards that conform to current environmental regulations on formaldehyde emissions.

To improve particleboard strength properties, Chapter 6 evaluated the option of incorporating flax and hemp bast fibers in continuous mat form as reinforcements in particleboard by taking advantage of the fiber's high tensile strength properties.

Chapter 7 presents a summary of the significant findings for the research and its importance to the particleboard industry. Also, included in the chapter are the research limitations and recommendations for future work.

The list of references cited in the study are provided in Chapter 8 and Appendices with additional information related to statistical data analysis are presented.

2. Materials and methods

This chapter describes the residues and adhesives as received and used in this research. The chapter includes detailed descriptions of the processes that were performed on the residues to obtain the portions required for particleboard manufacture and how these residues were characterized physically and thermally to provide insight into the behaviour of the material. The method used for particleboard manufacture in this study consistent with the manufacturing sequence commonly employed in industry is also described, as is a thorough explanation of all physical and mechanical property tests that were performed on the manufactured particleboards per the American Society for Testing and Materials (ASTM) standards. Finally, the chapter ends with the process of data analysis and evaluation of the board properties against those specified by the American National Standards (ANSI A208.1) for particleboard.

2.1 Raw materials used in the study

The non-wood natural resources explored in this research include both the woody core tissue (flax shive and hemp hurd) and bast fibers (flax and hemp fiber) obtained from the flax and hemp crop.

2.1.1 Flax shive and hemp hurd residues

Flax shive and hemp hurd residues were purchased from the Alberta Innovates Technology Futures' pilot decortication facility in Vegreville, Alberta; these consisted of woody tissue and short fiber residues (Figure 2.1).



Figure 2.1: Flax shive and hemp hurd residues. Source: Sam-Brew 2016.

The flax shive and hemp hurd materials were sieved into size fractions via a laboratory mechanical shaker using 4 standard sieves with openings 4.6 mm, 2 mm, 1 mm and 0.5 mm (Figure 2.2).



Figure 2.2: Mechanical shaker with 4 sieves for residue fractioning. Source: Sam-Brew 2016.

After sieving, oversize (on 4.6 mm screen) and undersize (through 0.5 mm screen) residues were discarded. Figure 2.3 shows the various size fractions obtained for both flax shive and hemp hurd residues after sieving.

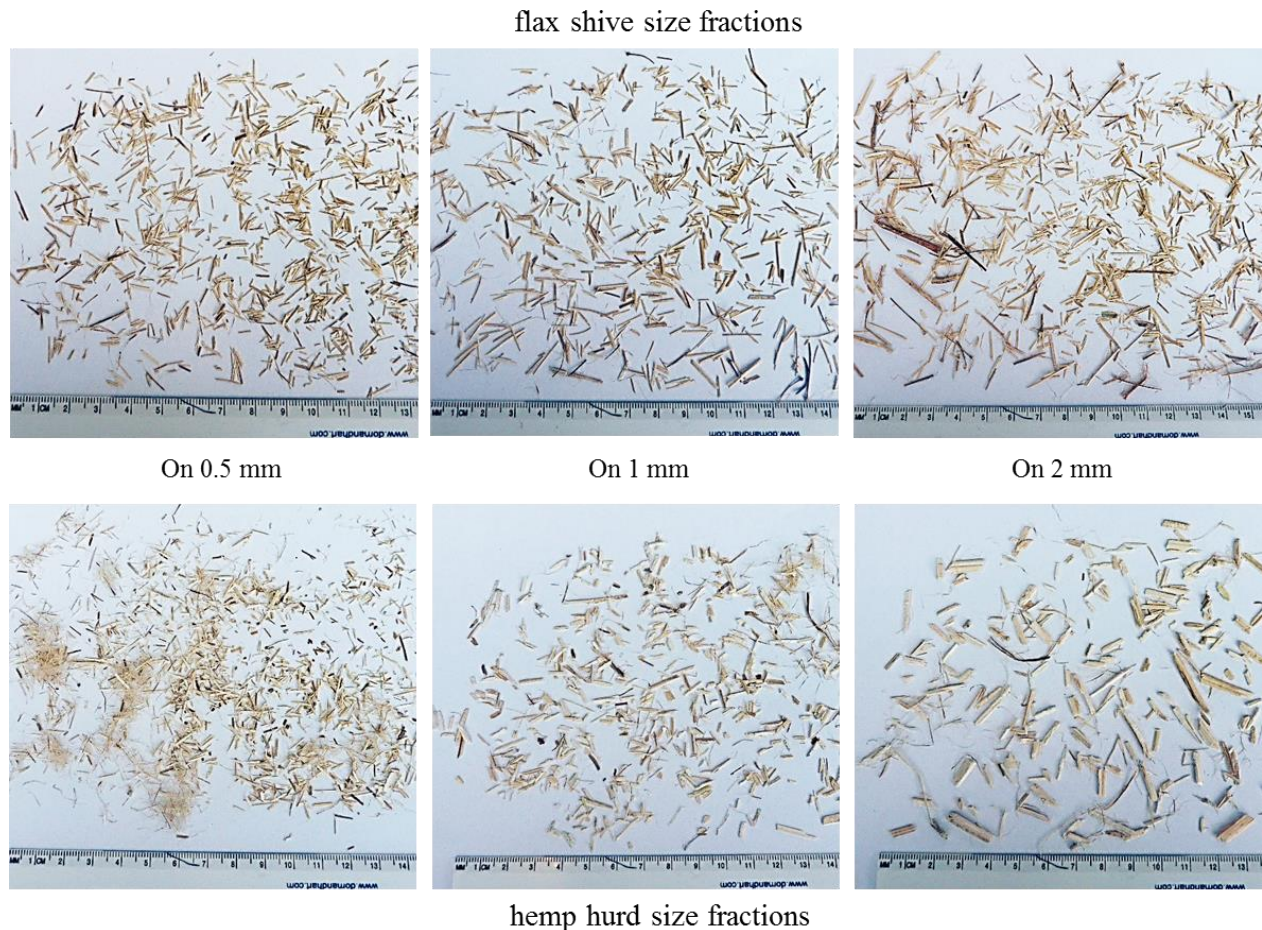


Figure 2.3: Flax shive and hemp hurd residues collected on the 0.5 mm, 1 mm and 2 mm sieves after screening. Ruler on bottom is demarked in cm. Source: Sam-Brew 2016.

Particles collected on the 2 mm sieve (100%) were used as core furnish only. A 50:50 mixture of particles on the 1 mm and 0.5 mm sieve were used as face furnish, with the exception of the hemp hurd residue where only particles on the 1mm sieve (100%) was used because those on the 0.5 mm sieve contained a larger proportion of short hemp fibers which tended to agglomerate; this is not

desired because during board manufacture these fiber balls do not mix well with the woody particles nor the resin (Figure 2.4).

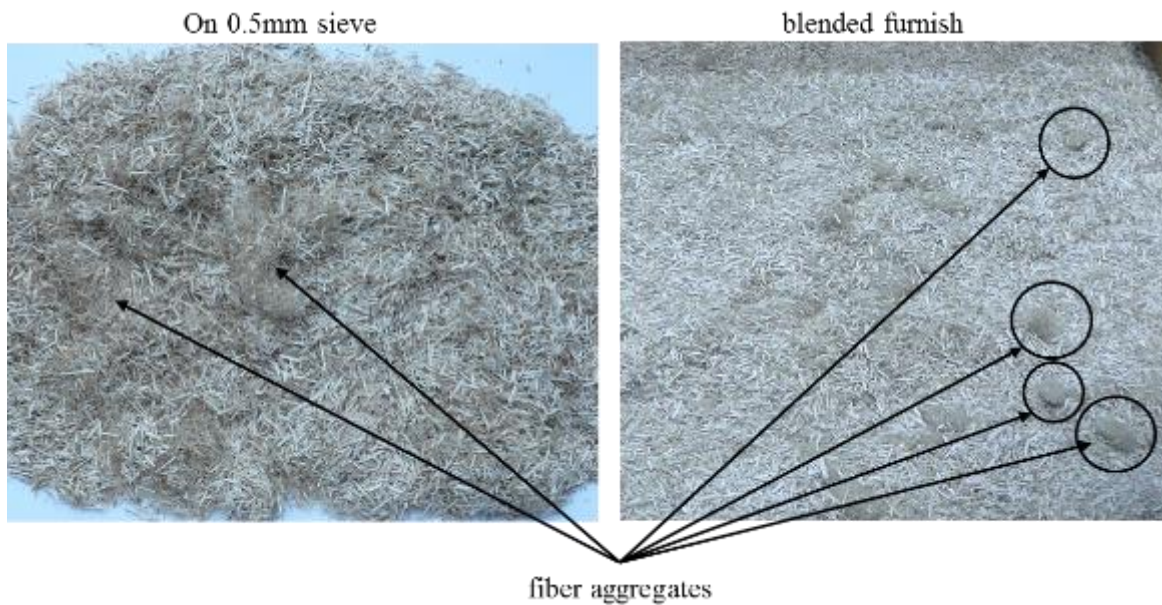


Figure 2.4: Hemp fiber aggregates as seen in hemp hurd furnish collected on 0.5 mm sieve and blended with resin. Source: Sam-Brew 2016.

2.1.2 Wood residues

For purposes of comparison with non-wood residues, industrial face and core wood particles consisting of softwood species mostly Spruce, Firs and Pines were supplied by the Roseburg Forest Products Company in Dillard, Oregon (Figure 2.5).

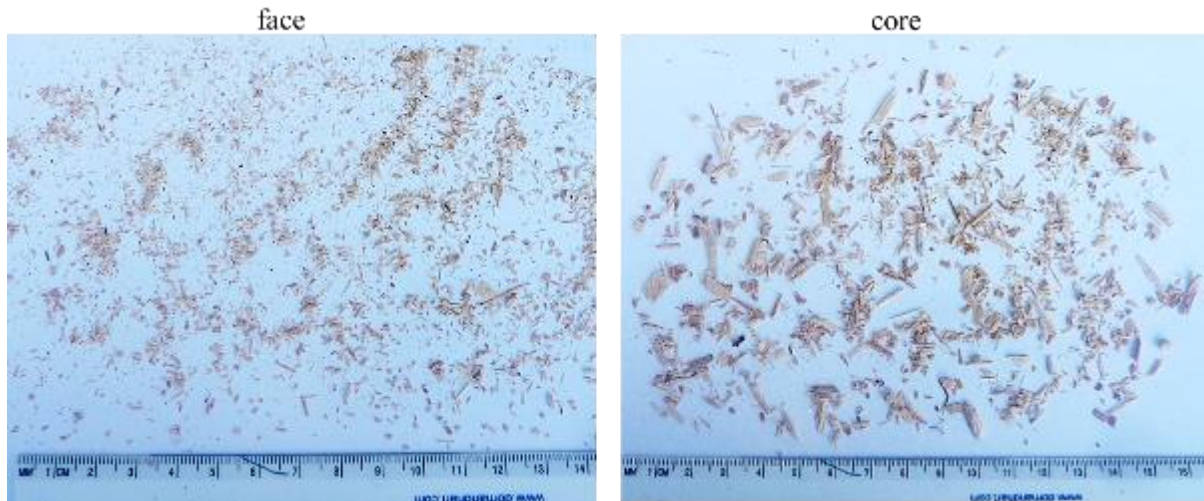


Figure 2.5: Industrial wood residue. Ruler on bottom is demarked in cm. Source: Sam-Brew 2016.

2.1.3 Flax and hemp fiber

The flax and hemp fibers which cost \$1.20/kg were also purchased from Alberta Innovates Technology Futures. The fibers consisted of a mix of single strand fibers and fiber bundles, with the latter being the majority (Figure 2.6).

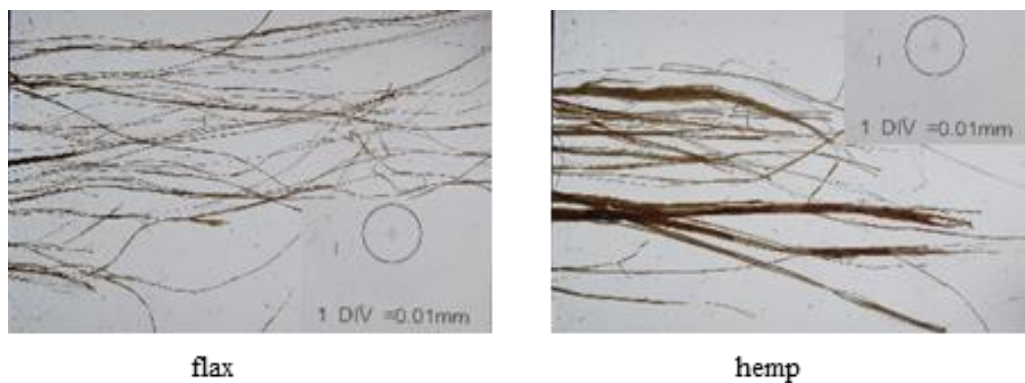


Figure 2.6: Flax and hemp fiber bundles. Source: Sam-Brew 2016.

Using a prototype Fiber Quality Analyzer developed by the Pulp and Paper Center, UBC, in accordance with the apparatus described in ASTM D7879-13, mean fiber diameters ranging from

0.09 mm to 0.18 mm for flax and 0.25 mm to 0.47 mm for hemp were recorded for 7 samples sets. A carding board (72 teeth per inch) and hand carder (54 teeth per inch) were used to align the hemp and flax fibers into mats approximately 635 mm by 203 mm by 3 mm (Figure 2.7).

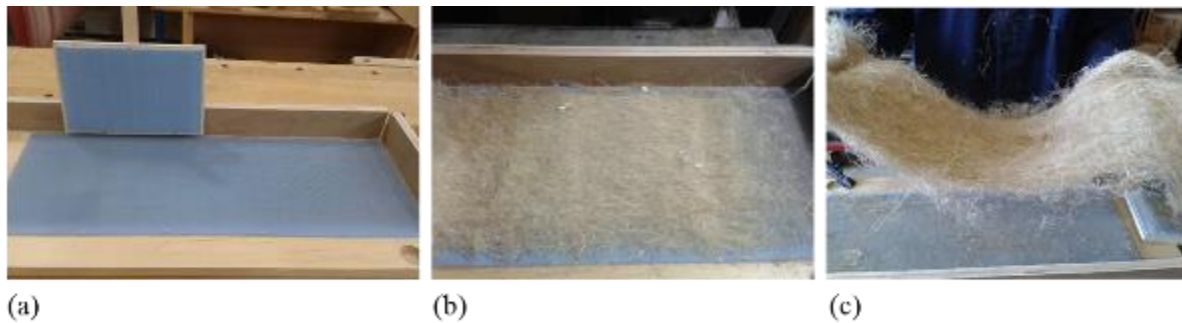


Figure 2.7: Aligning of hemp fiber using carding board and hand carder: (a) custom made carding board and hand carder, (b) carding of hemp fiber, and (c) aligned hemp fiber mat. Source: Sam-Brew 2015.

2.1.4 Resins

Two main types of resin were used for particleboard manufacture in this study, a commercial grade pMDI resin Lupranate[®] M20 and an acrylic based resin Acrodur[®] 950L, both provided by BASF North America. A third resin called Casco-Resin[™]—a commercial grade urea formaldehyde (UF) resin—from Hexion Inc. was used for only differential scanning calorimetry studies. Table 2.1 presents information on the physical and chemical properties of the resins used in this research.

Table 2.1: Physical and chemical properties of resins used in the research.

Resin type	pMDI	Acrylic	UF
Brand name	Lupranate® M20	Acrodur® 950L	Casco-Resin™
Color/ physical state	dark amber liquid	yellowish liquid	white-hazy liquid
Solids content	100%	50%	65%
NCO content	31.5%	n/a	n/a
pH value	n/a	3-4	7.2-8.4
Density	1.22 g/cm ³	1.2 g/cm ³	1.27-1.30 g/cm ³
Viscosity	200 mPa.s @ 20 °C	900-2500 mPa.s @ 23 °C	100-340 mPa.s

2.2 Description of experimental methods employed

2.2.1 Residue and fiber characterization

Bulk density measurements were taken for the flax shive, hemp hurd and wood residues by freely pouring the residues into a box (of known volume) without compaction and subsequently weighing the box (Figure 2.8). Using image analysis software, Image J (National Institutes of Health Image), particle length and width (aspect ratios) as well as total surface area were also measured for the face and core furnish of all 3 residue types.

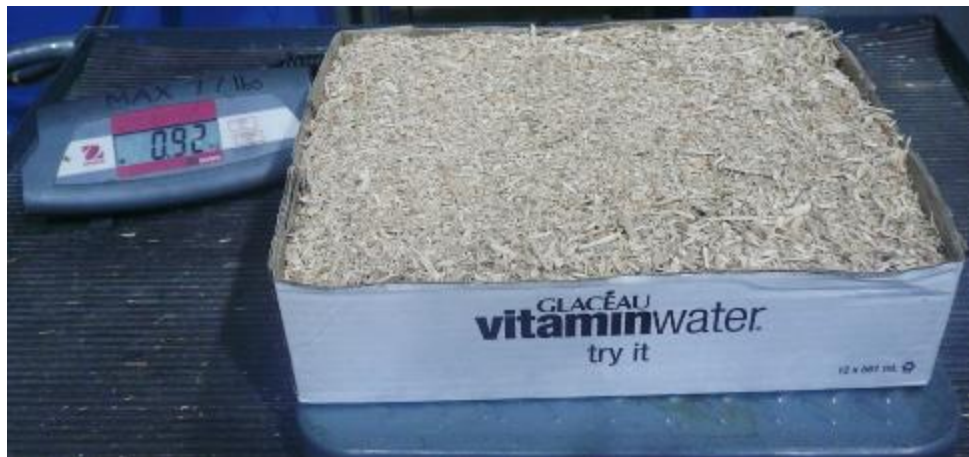


Figure 2.8: Bulk density measurement of hemp hurd residue. Source: Sam-Brew 2016.

Using a stage microtome, samples 20 μm thick were cut from cross-sections of the flax and hemp stalks and viewed under a Hitachi S3000N scanning electron microscope (SEM), to provide information on the woody core tissue.

2.2.2 Differential scanning calorimetry (DSC)

DSC is a technique that is used to measure the difference in temperature and heat flow observed when a material undergoes a transition (physical or chemical change) or chemical reaction. For instance, when thermoset resins such as pMDI are heated they go through an irreversible exothermic chemical reaction where the resin components evolve heat during curing. Using thermal techniques such as the DSC the heat evolved can be observed and measured as a large exothermic peak. DSC was therefore employed to observe the effect that the different residues (i.e. wood, flax shive and hemp hurd) have on the curing behavior of pMDI, Acrodur® 950L and UF resins. The information provided can be used to optimize the pressing parameters in particleboard manufacture.

The residues were ground into powder using a ball mill, Retsch PM 200 (Figure 2.9), and sieved through an ASTM no. 60 sieve with 0.250 mm opening. The powder that went through the sieve was oven dried and used for the DSC samples.



Figure 2.9: Ball mill for grinding residues. Source: Sam-Brew 2016.

High-volume stainless steel hermetic sample pans which are commonly used with liquid and solids with volatile contents (such as water, formaldehyde) were used for the DSC measurements. The presence of moisture has been shown to increase the curing of pMDI resin by accelerating cure (He and Yan 2005, 2007). Moisture was therefore considered as a factor in the analysis of the thermal properties of the wood, flax shive and hemp hurd residues to replicate the exact conditions used in hot pressing the particleboards. To avoid any erroneous effects of moisture and ensure detailed observations of only the effects of different residues on the curing behaviour of pMDI, all the residues were oven dried at 103 ± 2 °C for 24 hours and a specified quantity of distilled water added to bring all samples to a similar MC before testing. The specified quantity of distilled water was based on a mass ratio of 1:10 residue and or resin. The wood, flax shive and/or hemp hurd powder were thoroughly mixed with the resin and distilled water at a 10:10:1 mass ratio respectively immediately prior to testing. For samples without residues, the pMDI resin was mixed with distilled water at a mass ratio of 10:1.

Sample sizes ranging from 15.0 to 15.5 mg were placed in the high-volume sample pans and tightly sealed with a rubber O-ring seal and lid. The dynamic scan was conducted using a single heating

rate of 10 °C/min within a temperature range of 40 °C to 230 °C for samples with the pMDI resin and a range of 40 °C to 200 °C for the Acrodur[®] 950L and UF resins in a TA Instruments DSC Q1000 machine. Three replicates were conducted per each sample with less than 4% deviations in peak height between samples. All analysis and comparisons were conducted using TA Universal analysis software.

For each sample the onset temperature, the peak temperature and reaction heat/ reaction enthalpy were extracted from the DSC curves as shown in Figure 2.10. The extracted onset temperature indicates the starting temperature of the cure reaction, and speaks to the reactivity of the resin. The peak temperature obtained from the peak of the exothermic reaction indicates the temperature at which the cure rate reaches a maximum. Finally, the reaction heat represented by the area under the curve defines the amount of energy released or produced during the reaction and speaks to the chemical bond formation between the resin molecules also known as resin crosslinking.

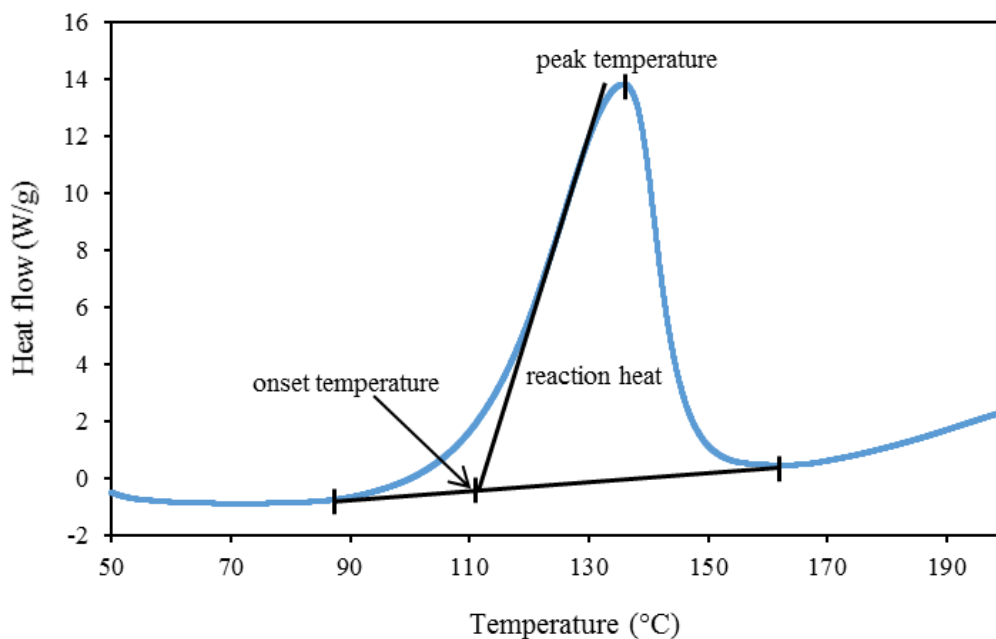


Figure 2.10: DSC curve illustrating reaction heat, onset and peak temperature data extraction.

2.2.3 Particleboard manufacture

For each experiment 3-layered particleboards consisting of 50% face and 50% core furnish were manufactured as illustrated in Figure 2.11. The moisture content (MC) of all the residues at the time of board manufacture were determined per ASTM D1348-94.

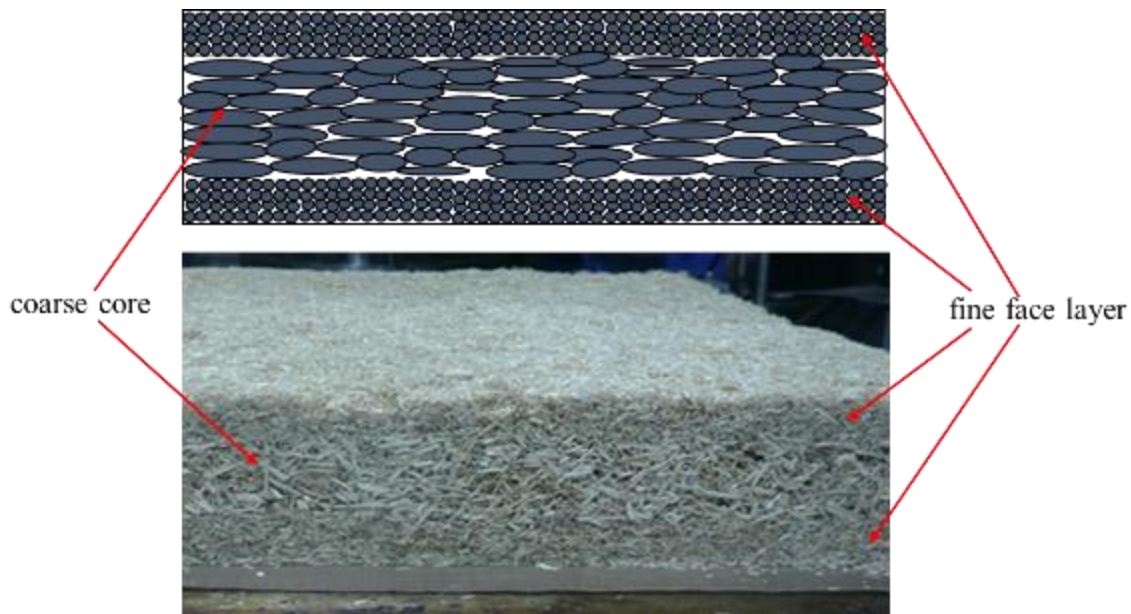


Figure 2.11: Three-layered hemp hurd particleboard with fine face and coarse core furnish. Source: Sam-Brew 2016.

The face and core furnish were blended separately with the chosen quantity of resin termed the resin load, based on the oven dry weight of the residues. Using a Drais® paddle blender fitted with an air atomizer the particles were uniformly blended with resin for 5 minutes (Figure 2.12).



Figure 2.12: Drais paddle-type particleboard blender. Source: Sam-Brew 2016.

Caul plates of dimensions 710 mm by 710 mm and a wooden forming box with inside dimensions 635 mm by 635 mm were used for mat formation. Teflon sheets were used as release agents to prevent direct contact between the caul plates and the resinated furnish to allow for easy removal of the final board after pressing. The resinated furnish were formed by hand according to the schematic shown in Figure 2.11 and evenly pre-pressed with a wooden board to remove air, reduce the mat thickness and ensure contact between the particles (Figure 2.13).

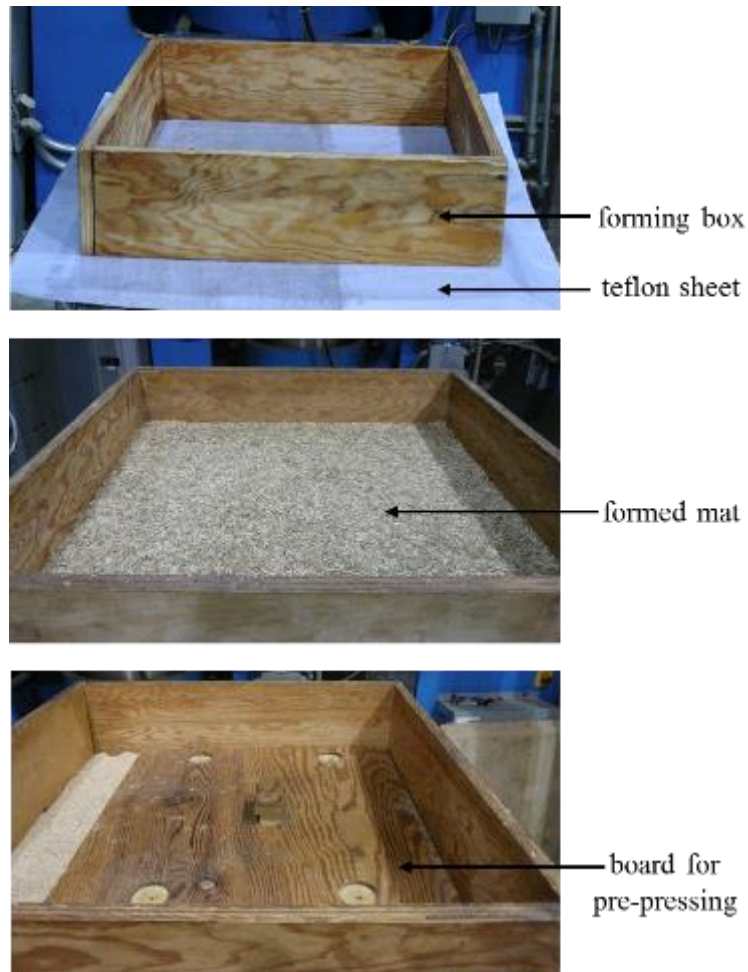


Figure 2.13: Mat forming and prepressing process during particleboard manufacture. Source: Sam-Brew 2016.

The wooden frame was removed, a teflon sheet and caul plate were placed unto the top of the mat before transferring it into a Pathex hot press (model 338T) (Figure 2.14). The mat was pressed to a target board density of 620 kg/m^3 and panel thickness of 12.7 mm. The press cycle based on the mat displacement/thickness included 16 s closing time, 415 s holding period at 2 MPa and 140°C , and 200 s degassing time before transferring to pressure control for a 50 s opening.

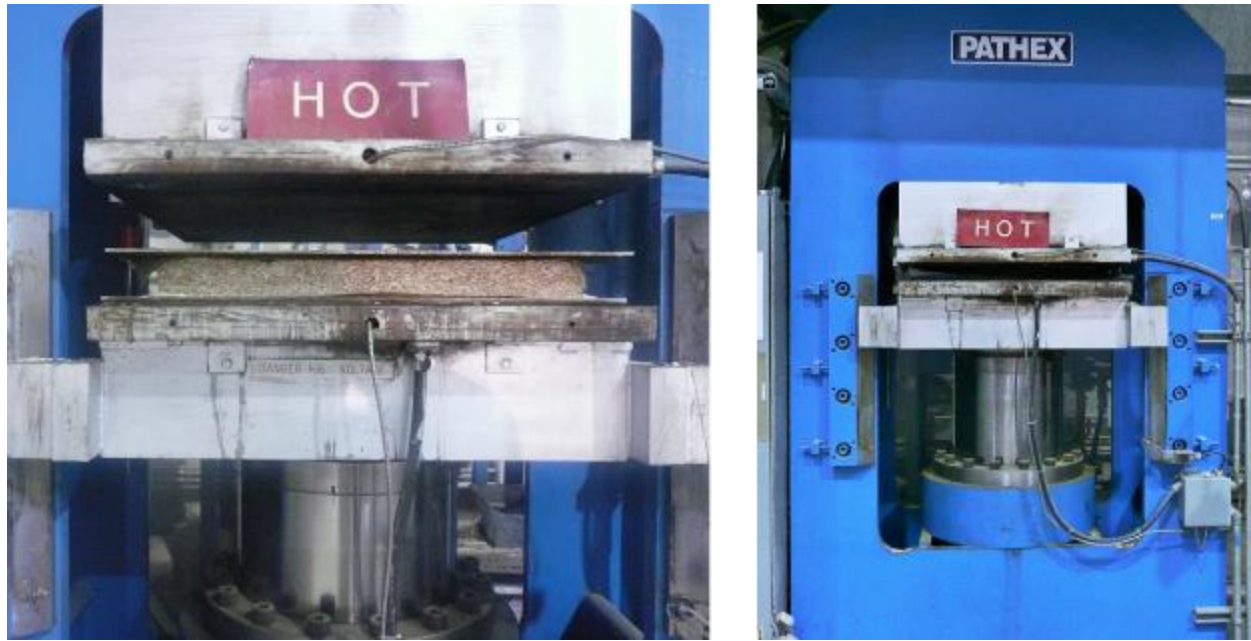


Figure 2.14: Hot pressing operation. Source: Sam-Brew 2016.

2.2.4 Short-term property testing

After manufacture the boards were cooled down at room temperature for 24 hours and trimmed by approximately 25.4 mm to eliminate edge effects. The boards were sanded with a wide belt sander to remove pre-cured surfaces and provide even board surfaces for testing. Samples were cut up for mechanical and physical property tests comprising internal bond, thickness swell, water absorption, static bending and linear expansion. As much as possible the test samples were randomly obtained from the particleboards using cutting patterns, a typical example is given in Figure 2.15. The samples were stacked in a chamber maintained at a $65 \pm 5\%$ relative humidity and $20 \pm 3^\circ\text{C}$ and conditioned to constant weight and MC for a minimum of 2 weeks before testing (Figure 2.16). The samples were evaluated in accordance with American Standards for particleboard (ANSI A208.1-1999, A208.1-2009 and ASTM D1037-06a).

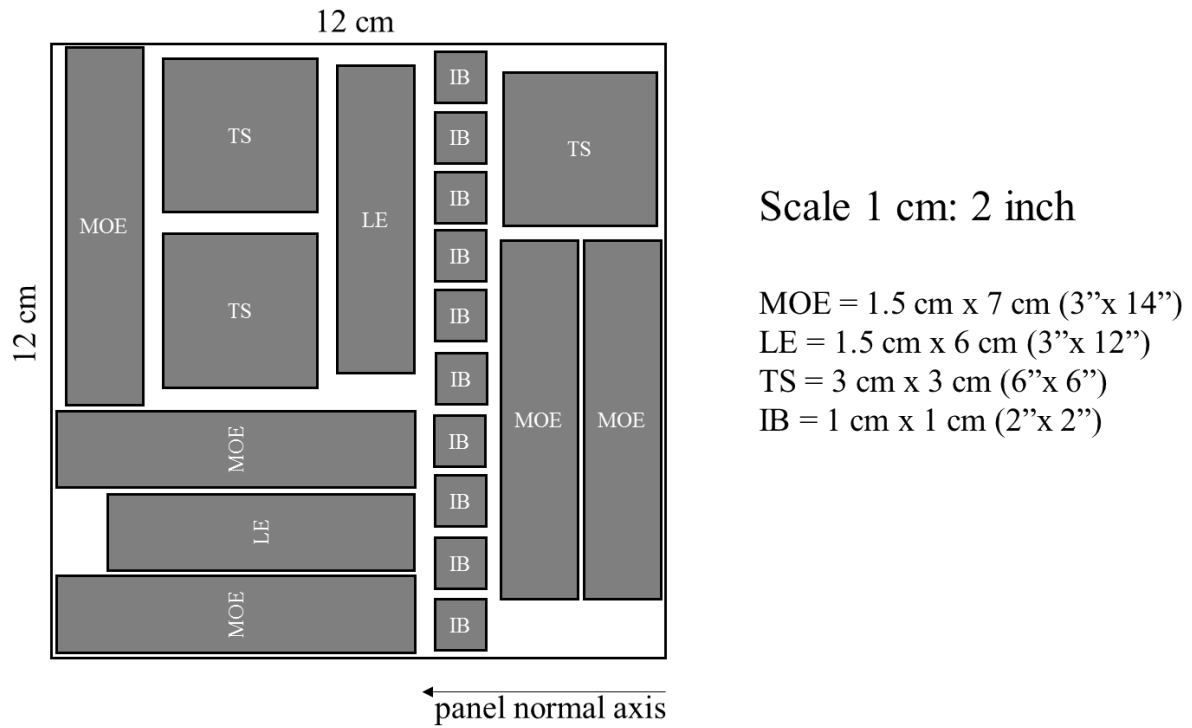


Figure 2.15: Sample cutting pattern used to obtained test samples for physical and mechanical property tests. Source: Sam-Brew 2016.



Figure 2.16: Test samples stacked in a constant humidity chamber. Source: Sam-Brew 2016.

2.2.4.1 Vertical density profile (VDP)

The differences in density through the thickness of the particleboard because of the particle characteristics and pressing parameters is termed the vertical density profile (VDP). This was measured with an X-ray machine, Quintex Measurement Systems (model QDP-01X) using samples measuring 50 mm by 50 mm (Figure 2.17). Length, width, thickness and weight measurements were taken for each sample prior to testing. The samples were placed vertically in sample holders and scanned individually.



Figure 2.17: X-ray machine for vertical density gradient measurements. Source: Sam-Brew 2016.

2.2.4.2 Internal bond (IB)

The internal bond (IB) test method was used to determine the cohesive strength of the particleboard by applying a tensile force perpendicular to the board face. This was conducted on samples of similar size as those used for the VDP and since the VDP was a non-destructive test the same samples were tested for the IB strength. The test samples were bonded to aluminum blocks of size 50 mm by 50 mm with hot melt glue and the blocks attached to the loading head of a Sintech 30D universal testing machine (Figure 2.18). The samples were tested at a uniform loading rate of 1.016 mm/min until failure occurred as per the ASTM D1037-06a standard. The IB was calculated as a ratio of the maximum load applied to the surface area over which it was applied:

$$IB = \frac{P_{max}}{ab}$$

(ASTM D1037-06a)

where:

a = length of the test specimen (mm)

b = width of the test specimen (mm)

P_{max} = maximum load (N)

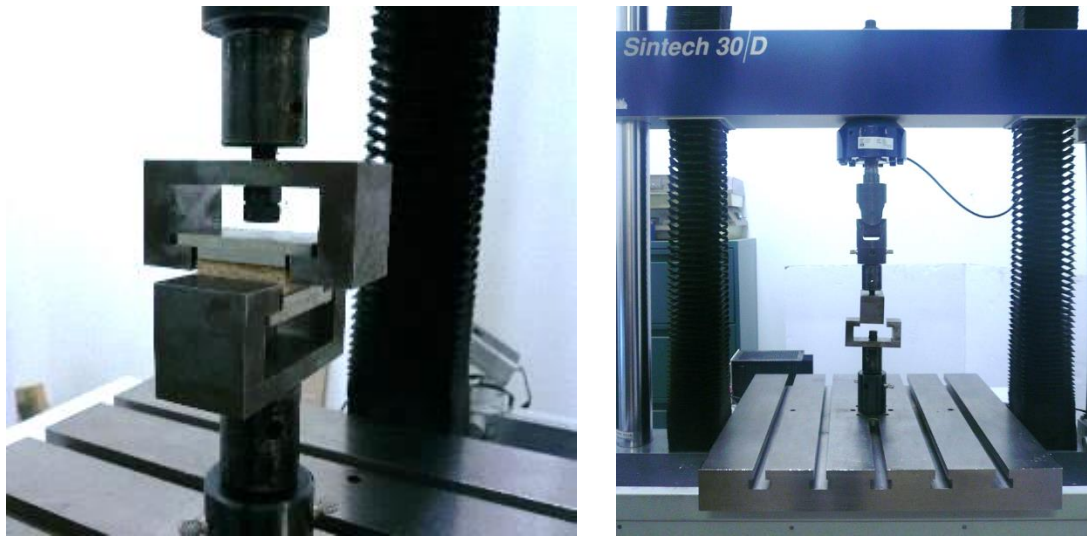


Figure 2.18: Internal bond test with a universal testing machine. Source: Sam-Brew 2016.

2.2.4.3 Static bending - modulus of rupture and elasticity (MOR/MOE)

Three-point bending tests were conducted to determine the modulus of rupture (the maximum load carrying capacity also known as the bending strength) and the modulus of elasticity (the recoverable deformations produced referred to as the bending stiffness) in bending per ASTM D1037-06a. Test samples measuring 76 mm by 356 mm (3 inch by 14 inch) were obtained both parallel and perpendicular to the forming direction (normal axis) of the particleboard. The initial sample mass, length, width and thickness were measured and used to calculate the average board density of the particleboards. The samples were then mounted on supports and loaded mid-point using a force perpendicular to the length of the samples at a loading rate of 6.35 mm/min (Figure 2.19).

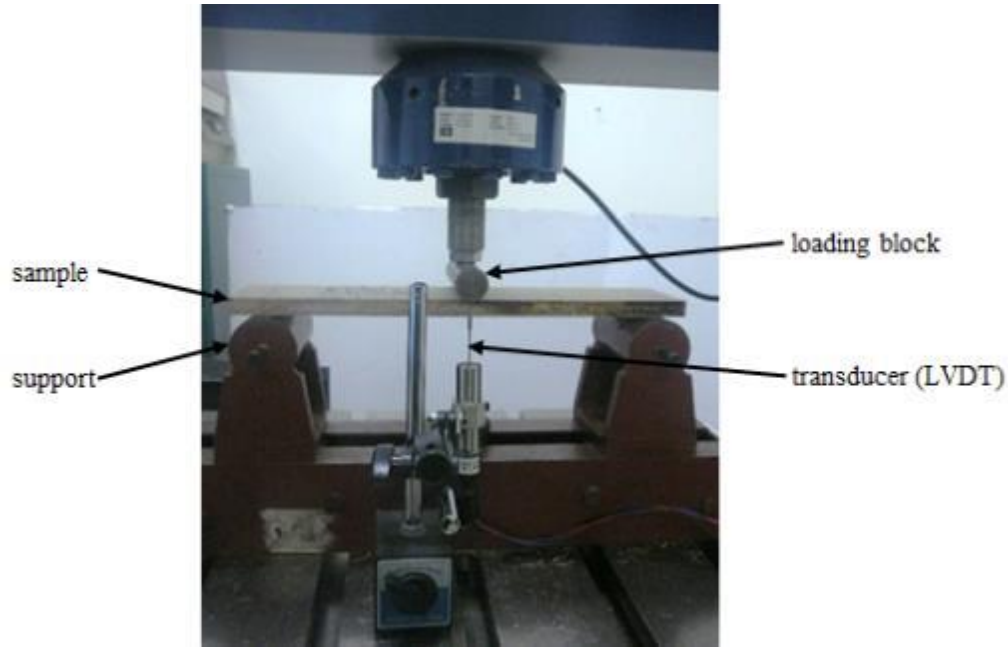


Figure 2.19: Three-point bending test conducted on hemp hurd particleboard. Source: Sam-Brew 2016.

The load-deflection data was obtained via a linear voltage differential transducer (LVDT) attached to the test sample at center span directly beneath the loading head. In accordance with ASTM D1037-06a, the Young's modulus (E) was calculated from the slope of the straight-line portion of the load-deflection curve, that is from 10% to 40% of the maximum load attained and used in the calculation of the apparent modulus of elasticity (MOE) according to the following equation:

$$MOE = \frac{L^3}{4bd^3} \frac{\Delta P}{\Delta y} \quad (\text{ASTM D1037-06a})$$

and the modulus of rupture (MOR) calculated as:

$$MOR = \frac{3P_{max}L}{2bd^2} \quad (\text{ASTM D1037-06a})$$

where:

b = width of the test specimen (mm)

d = thickness of test specimen (mm)

L = span length (mm)

$\frac{\Delta P}{\Delta y}$ = slope of load deflection curve (N/mm)

P_{max} = maximum load (N)

In contrast to conventional fiber-reinforced composites (example glass or carbon fiber reinforced plastics), the mechanical properties of wood composites cannot be accurately predicted using the traditional rule of mixtures. This is because in wood composites (particleboard, MDF, OSB) the wood fibers/strands are discontinuous and the small amount of resin used (less than 10 weight %) is sprayed on as droplets forming a discontinuous deposit of non-uniform thickness which partially covers the fiber/strand. Thus there is non-uniform stress transfer from one point to the other. Moreover, the wood fibers/strands have orthotropic characteristics, a complex microstructure and time-dependent properties which further complicates its performance.

2.2.4.4 Linear expansion (LE)

The linear expansion (LE) measures the dimensional changes in a sample as the MC of its environment changes. Samples measuring 76 mm by 305 mm were cut parallel and perpendicular to the forming direction of the particleboard. Consistent with ASTM D1037-06a the test samples were kept in a constant humidity chamber maintained at a $50 \pm 2\%$ relative humidity and 20 ± 3 °C for 2 weeks and the length of each sample measured with a linear expansion gauge consisting of a Mitutoyo Digimatic caliper fixed on a board (Figure 2.20). Length measurements were taken always with the direction parallel to the forming direction of the particleboard pointing to the left of the gauge (Figure 2.20a) or to the top of the caliper for perpendicular samples (Figure 2.20b).



Figure 2.20: Linear expansion measurements for wood particleboard measured a) parallel and b) perpendicular to the forming direction of the board. Arrow indicates the forming direction of the original board. Source: Sam-Brew 2016.

The test samples were then transferred to another chamber maintained at a $90 \pm 2\%$ relative humidity and $20 \pm 3^\circ\text{C}$ for an additional 2 weeks before the length measurements were taken again for each sample. The percentage change in length (i.e. the LE) between the lower and higher relative humidity was calculated based on the length obtained from the 50% relative humidity measurements. The results obtained were then presented for each particleboard type per ANSI

A208.1-2009 standard as a calculation of the upper or maximum 95th percentile for the range of LE data recorded by:

$$U_{95\%} = \bar{\bar{x}} + t(S\bar{x}) \quad (\text{ASTM D1037-06a})$$

where:

$\bar{\bar{x}}$ = grand mean of test sample averages

$S\bar{x}$ = standard deviation between test sample averages

t = single-sided 5% t-value of a normally distributed sample

2.2.4.5 Thickness swell (TS) and water absorption (WA)

This test was used to determine the water absorption characteristics of particleboard. Test samples measuring 152 mm by 152 mm after conditioning were measured for their weight, thickness, length and width values. These were then submerged horizontally in a tank that was filled with fresh water and held 25.4 mm below the water surface for 2 hours (short term) and a subsequent 22 hours (long term) per ASTM D1037-06a as shown in Figure 2.21.



*Figure 2.21: Submerged particleboards in thickness swell and water absorption measurements.
Source: Sam-Brew 2016.*

After soaking for 2 hours the samples were taken out and the excess water allowed to drain off for a few minutes. The samples were weighed to determine the water absorbed (WA) which was expressed as a percentage by volume and weight. The thickness of the samples was measured from four points mid-way along each edge to evaluate the changes in thickness (TS) with reference to the initial sample thickness before submersion. The same procedure was repeated after soaking the samples for 22 hours. The test samples were then dried in an oven set at 103 ± 2 °C for 24 hours to determine their oven dry mass. Using the samples' initial mass and oven dry mass the MC for each particleboard was calculated. In analysis of the TS and WA characteristics, the long term 2-plus-22 hour soaking period is simply referred to as a 24 hours or long term submersion.

2.2.5 Data analysis

Using an analysis of variance test (ANOVA) and the Tukey-Kramer honestly significant difference test, the test data obtained were analyzed for potential differences between particleboard types. All test data were checked to ensure they met the assumptions regarding the error term (i.e., independent observations, normal distribution and equal variance) and in cases where the test data did not meet these assumptions, transformations such as logarithm, reciprocal or square root were applied. All statistical analysis was conducted with JMP 11 software package at a 5% significance level.

For some results, strength properties are graphically presented according to ANSI A208.1-1999 standards which permits the calculation of least significant differences between board types unlike A208.1-2009 which is presented based on the lower 5th or upper 95th percentile limit, otherwise the two ANSI versions, i.e. 1999 and 2009 are essentially equivalent.

3. Flax shive and hemp hurd residues as alternate raw material for particleboard production¹

The work presented in this chapter is based on experimental studies carried out to evaluate flax shive and hemp hurd residues as alternative furnish for particleboard manufacture. These residues as indicated in the introductory Chapter 1, are readily available in Canada and if proven to be capable of substituting wood for board production will help address wood residue shortage experienced by several particleboard mills. The chapter includes background information on the reasons and key objectives for conducting the study, a summary of the design of experiment indicating the factors and or combination of factors of interest, and detailed analysis and discussion of results of the particleboard properties tests conducted and their relevance to the industry.

3.1 Background

The major costs in particleboard production are the wood residue and the resin. The costs incurred from the resins are mainly due to the overall increase in crude oil and natural gas prices. The increasing cost of wood residues on the other hand is because of scarce supply with numerous sawmill closures, the cost of transporting the residue over significant distances and competition with other sectors (example the bio-energy industry) for available sawdust and shavings. For the particleboard mills this limited wood residue supply means finding material from several locations near and far to prevent production curtailment. This results in non-uniform residue resources composed of a variety of wood species, increased transportation and furnish costs, and invariably increased production costs.

Most of the time to counteract the negative effect that a wide furnish variability has on particleboard strength properties, more resin is incorporated during panel manufacture further increasing costs. A promising alternative to this issue is to investigate other lignocellulosic

¹ A version of this chapter will be submitted for publication

residues which are readily available and can efficiently substitute wood residue in particleboard production. Accordingly, agricultural crops and plant residues have been of interest in recent years. Numerous articles have been published concerning the use of the branches, leaves, seeds, husks, roots and or fruits of agricultural crops and plant residues for particleboard manufacture. Examples include guar and sorghum stalks (Gabir et al. 1990), sunflower stalks (Khristova et al. 1996), waste tea leaves (Yalnkilic et al. 1998), castor stalks (Grigoriou and Ntalos 2001), coconut shell (Almeida et al. 2002), durian peel and coconut fiber (Kheduri et al. 2003), almond shell (Gürü et al. 2006), tissue paper solid waste and corn peel (Lertsutthiwong et al. 2008), wild rye (Li et al. 2009), rice straw (Li et al. 2010), corn cob (Pavia et al. 2012), macadamia shell (Wechsler et al. 2013) and poppy husk (Keskin et al. 2015).

Particleboard has conventionally been manufactured with urea formaldehyde (UF), and in some cases phenol formaldehyde (PF). But most agricultural crop and plant residues have waxy outer stalk surfaces that hinders its bonding to conventional resins resulting in poor interfacial interactions (Mwaikambo and Ansell 2002; Wasycliw 2005). This interfacial interaction is important because the waste residues may have high strength and stiffness properties but if the bonding between them is poor, the inherent strength of the residue counts for nothing and the resulting composite exhibits poor mechanical properties (Zhang et al. 2005; Ndazi et al. 2006). Polymeric diphenyl methane diisocyanate (pMDI) resin has been shown to successfully bond agricultural crops and plant residues (such as miscanthus, wheat straw, corn pith, rice straw) together to produce panels that meet the required standards for specific applications (Tröger et al. 1998; Wang and Sun 2002; Mo et al. 2003; Halvarsson et al. 2010; Zhang and Hu, 2014). Isocyanate resin cures rapidly and is used in lower quantities (usually 3-6% mass resin load) in comparison to UF and PF resins (6-14% mass resin load) (Frihart 2013), and with the current concerns about formaldehyde emissions from conventional UF and PF resins this adhesive type is a practical choice.

In Canada, flax (*Linum usitatissimum*) and hemp (*Cannabis sativa* L.) crops are cultivated for oil from their seeds and fiber from the stalk. Retting and decortication of the stalks leads to 3 main fractions – long staple fibers, short (tow) fibers and woody core tissue. The woody tissue obtained from the flax crop is termed flax shive and that from the hemp crop known as hemp hurd.

Considering the wood residue shortage that plagues some particleboard mills, the flax shive and hemp hurd residues which are readily available have been identified as alternate raw materials for particleboard production.

This study comprehensively evaluates the properties of low density 3-layered particleboards manufactured from 100% flax shive and hemp hurd residues using pMDI resin. Given the relatively higher cost of isocyanate resin it is important (for economic reasons) to identifying the minimum amount of resin that can sufficiently bond the flax shive and hemp hurd residues. A low resin consumption of 2.5% (based on oven dry weight of residue) and an upper limit of 5% is used for board production. The study consists of two main parts–

- (1) characterization of the flax shive and hemp hurd residues, and
- (2) evaluation of the physical and mechanical strength properties of the flax shive and hemp hurd particleboards against those of wood particleboards.

3.2 Experimental design

The main factors of interest for this experiment were the effect of residue type (wood, flax shive, hemp hurd) and quantity of resin (2.5%, 5%) on particleboard properties. Based on the experimental design outlined in Table 3.1, 3-layered particleboards as illustrated in Figure 2.11 were made from 100% flax shive, hemp hurd and wood residues through the manufacturing sequence outlined in Chapter 2-section 2.2.3. The moisture content (MC) of the flax shive and hemp hurd residues at the time of board manufacture were determined (per ASTM D1348-94) to be approximately 10% each for the 2.5% resin load boards and 9.8% each for the 5% resin load boards. The MC of the wood particles was 9.1% for the 2.5% resin load boards and 8% MC for the 5% resin load boards. Four replicates were manufactured per each combination of resin and residue type for a total of 24 particleboards.

Table 3.1: Design of experiment for low density wood, flax shive and hemp hurd particleboards.

Factors		Levels	Response	Total specimens
Variables	Residue type	wood	MOR/MOE	12
		hemp hurd	IB	32
		flax shive	TS	12
	Resin load (%)	2.5	LE	20
		5		
Constants	Density (kg/m ³)	620		
	Thickness (mm)	12.7		
	Resin type	pMDI		
	Replicates	4		

3.3 Results and discussion

3.3.1 Particle geometry and bulk densities

Particleboard properties such as the bending strength (MOR), bending stiffness (MOE), tensile strength perpendicular to the face (IB), surface characteristics and changes in dimensions in response to moisture (LE, TS and WA) are affected by the size characteristics of its constituent residues. Table 3.2, Figures 3.1 and 3.2 presents the length to width ratio (aspect ratio) of both face and core furnish for the flax shive, hemp hurd and wood residues.

Table 3.2: Mean aspect ratio of wood, flax shive and hemp hurd residues. $n=50$ for each mean. Values in parenthesis are standard deviations.

Material		Aspect Ratio
Face	wood	2.52 (1.25)
	hemp hurd	4.48 (2.18)
	flax shive	8.13 (4.28)
Core	wood	3.48 (2.36)
	hemp hurd	4.88 (1.89)
	flax shive	10.76 (5.53)

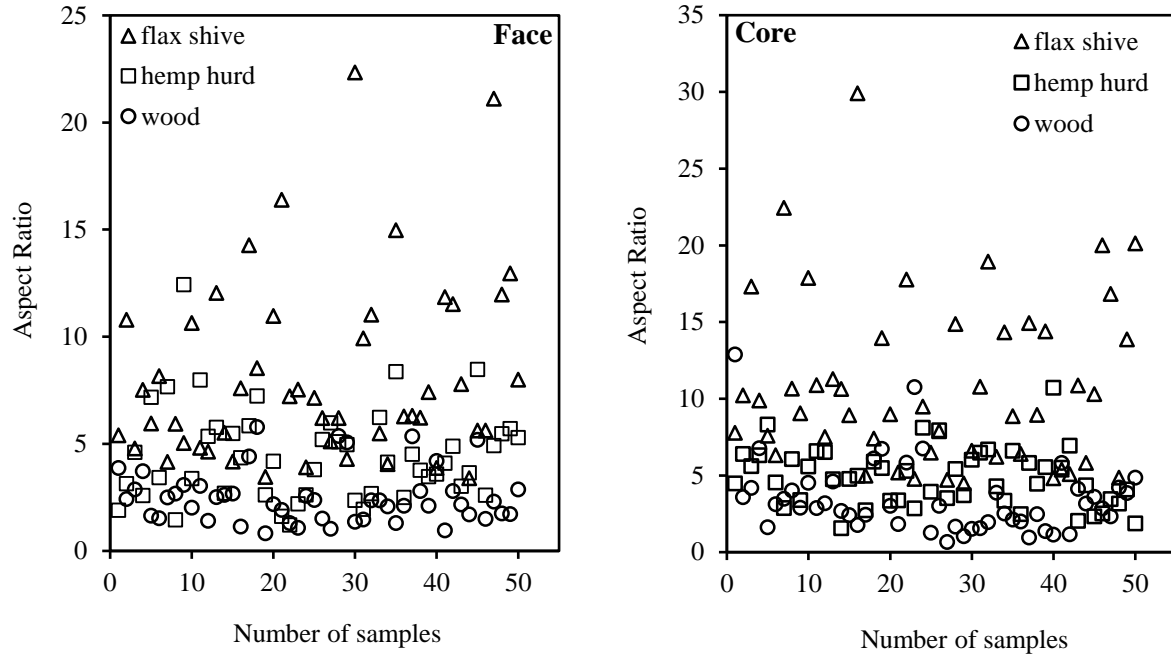


Figure 3.1: Aspect ratio of face and core wood, flax shive and hemp hurd residues. $n=50$ for each residue type.

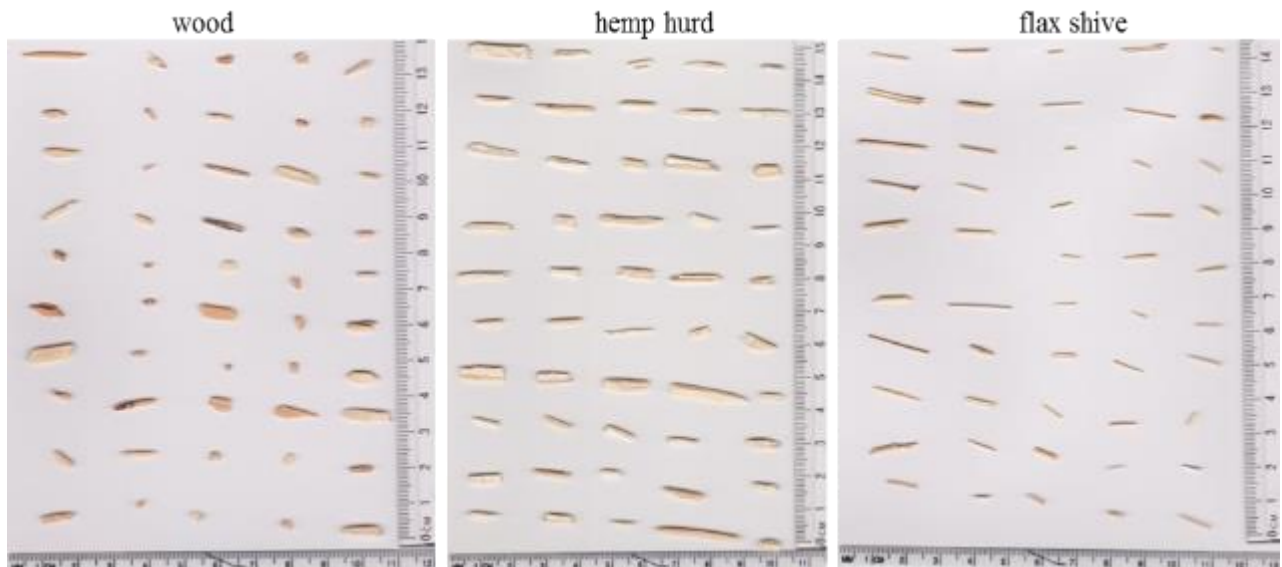


Figure 3.2: Visual comparison of core wood, flax shive and hemp hurd residues. Ruler on bottom is demarked in cm. Source: Sam-Brew 2016.

From the table and figures, the flax shives are characterised by higher aspect ratios with longer, narrower and needle-like particles, compared to the hemp hurd which are medium sized and more rectangular in form and the wood residues which are the shorter and thicker. Numerous studies conducted over the years on the effect of particle geometry on board properties have found that higher bending strength properties are obtained from longer particles because of the greater surface area it provides in terms of contact between particles (Maloney 1993, Juliana et al. 2012). Additionally, the shorter and thicker particles tend to improve internal bond strength because they have the tendency to pack themselves better during mat formation. Besides, theoretically at a set resin content the shorter and thicker particles are expected to have more resin coverage per unit area compared to the longer thinner particles owing to the decrease in specific surface area compared to the latter. It is anticipated that the flax shive and hemp hurd residues will produce particleboards with greater bending strength and the wood residues better internal bond strength.

Table 3.3 lists the bulk densities of the wood, flax shive and hemp hurd residues which was measured using the procedure given in section 2.2.1. In terms of core furnish the hemp hurd residues yielded the lowest bulk density significantly so ($p < 0.0001$) from the flax shive and wood core (Appendix B). The wood core had a much higher bulk density. Similarly, for the face furnish the wood residues yielded a significantly ($p < 0.0001$) higher bulk density compared with the hemp hurd and flax shive residues (Appendix B). For panel manufacturers, this means on a weight basis more flax shive and hemp hurd residues will be required compared to wood residues to produce a particleboard of similar thickness.

Table 3.3: Bulk density of wood, hemp hurd and flax shive residues. $n=3$ for each mean. Values in parenthesis are standard deviations.

Material		Bulk density (kg/m³)
Face	wood	200.90 (4.65)
	hemp hurd	140.01 (1.61)
	flax shive	88.13 (1.61)
Core	wood	167.05 (2.99)
	hemp hurd	89.87 (2.11)
	flax shive	99.38 (0.67)

3.3.2 Mat core temperature

During hot pressing the mat core temperature was monitored using a thermocouple positioned approximately at the center of the mat. Boards were produced using the same pressing cycle outlined in section 2.2.3. The rate of heat transfer through the core of the flax shive, hemp hurd and wood residues used in combination with the 5% resin load for particleboard manufacture are presented in Figure 3.3.

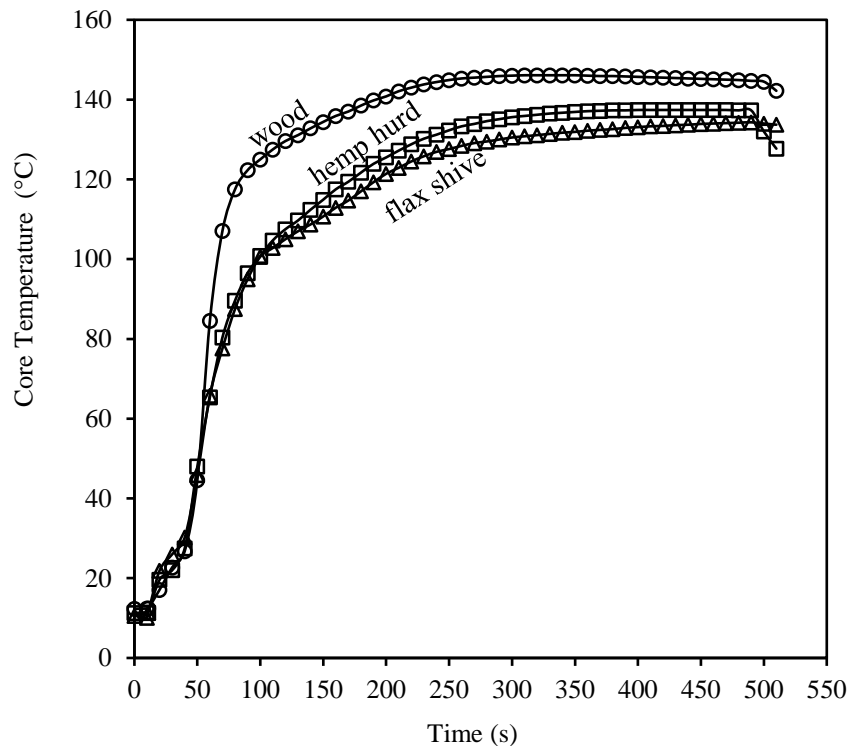


Figure 3.3: Differences in mat core temperature during hot pressing of the 5% pMDI boards.

In the first 50 seconds, there appears to be no difference between all 3 particleboard types as the core temperature steadily rises. The core of the wood mat quickly heats up to 100 °C after 70 seconds compared with a slower heating of the flax shive and hemp hurd mats which reaches 100 °C after approximately 100 seconds. The core temperature then steadily increases until it reaches a maximum of 134 °C and 137 °C for the flax shive and hemp hurd mats respectively while that

of the wood mat was slightly higher at about 146 °C (i.e. approximately the press platen temperature).

This slower rate of heat transfer to the core for the hemp hurd and flax shive mats can be attributed first to the initial differences in MC between the wood residues (8%) and the hemp hurd/flax shive residues (9.8%) and secondly to their lower bulk densities (Kelly, 1977; Papadopoulos et al. 2002; Papadopoulos and Hague 2003; Dai et al. 2004; Papadopoulos et al. 2004). For granular-type particles a positive linear relationship has been shown to exist between the press time and mat MC before the core mat temperature extends above 100 °C: that is the lower the MC the shorter the press time needed for the temperature to exceed 100 °C, therefore the faster heating rate in the wood core.

Furthermore, the lower bulk densities (increased void fraction) of the hemp hurd and flax shive residues coupled with the slightly higher MC (9.8%) compared to the wood residues delayed the rate of heat flow to mat core. This notion is corroborated by Papadopoulos et al. (2002, 2003) who working on wood, coconut stem chips and flax shive mats all of which had 10% MC and pressed to the same board thickness, observed similar trends in heat flow in the core where the wood chips yielded a faster rate of heat transfer in the core compared to the flax shive and the coconut chips.

Though the internal gas pressure was not measured during hot pressing for the particleboards in this study, it is believed that the internal gas or vapor pressure which speaks to the permeability of the mat influences the maximum core temperature achieved in the mat. A higher core temperature is indicative of a low mat permeability meaning a higher buildup of internal gas pressure within the mat (Rofii et al. 2014). Inferring from this the flax shive and hemp hurd mats in which the lowest core temperatures were observed is likely to have produced lower internal vapor pressures because of their porous nature which allowed easy escape of the gases to the edges of the mat.

This information is of importance as it influences the press time required to ensure the polymerization/hardening of the resin in the core of the particleboard.

3.3.3 Moisture content, average board density and vertical density profile

The average board densities were calculated based on the initial mass and volume of the static bending samples while the moisture content (MC) was computed from the thickness swell samples using their initial and oven dry mass (Table 3.4). Statistically significant differences ($p < 0.0001$) were observed in MC between panel types for both the 2.5% and 5% resin load boards (Appendix B). This difference in MC for both resin loads which was more pronounced in the hemp hurd and flax shive boards was the result of the moderately higher initial MC's of the residues compared to the wood residues.

Table 3.4: Average board density and moisture content of low density particleboards. Each board density mean was computed from 12 samples. Mean moisture content was calculated from 12 samples for the 2.5% boards and 11 for the 5% boards. Values in parenthesis are standard deviations.

	wood	hemp hurd	flax shive
2.5% resin load boards			
Density (kg/m ³)	555 (36.72)	532 (25.02)	533 (17.69)
MC (%)	10.24 (0.13)	10.94 (0.08)	11.56 (0.40)
5% resin load boards			
Density (kg/m ³)	638 (35.60)	631 (26.53)	657 (18.15)
MC (%)	10.22 (0.11)	10.53 (0.12)	10.86 (0.12)

The average board densities for the 2.5% resin load boards ranged from 532 to 555 kg/m³ and were not significantly different from each other (Appendix B). For the 5% resin load boards on the other hand, average board density was significantly different ($p = 0.0401$) between panel types, specifically between the flax shive and hemp hurd (Appendix B). From Table 3.4 its evident that the average board density was lower for the 2.5% resin load boards compared to the 5% resin load boards. It is important to point out that all board manufacture was based on mass calculations to the same target density of 620 kg/m³ and thickness of 12.7 mm. This difference is in part the result of the differences in residue moisture and resin content.

Figure 3.4 shows the peak face (F1, F2) and core (C) densities through the thickness of the wood, hemp hurd and flax shive panels for each resin content. On average, all board types portrayed the normal vertical density profile (VDP) observed in particleboards—high density faces and low core densities. In the hot-pressing operation, the surface layers of the mat in contact with the press platen heats up, moisture is readily converted to water vapor and heat transferred to the core mostly by convection. The compressive strength of the surface layers is therefore lowered first (through the relaxing effect of moisture and temperature) and the particles compressed to a greater extent than those in the mat core before the target thickness is reached. This density gradient dictates the portion of the board with higher face densities which enhance particleboard bending strength and stiffness properties, and the lowest density (i.e. the core) which invariably influences the internal bond strength and screw withdrawal resistance.

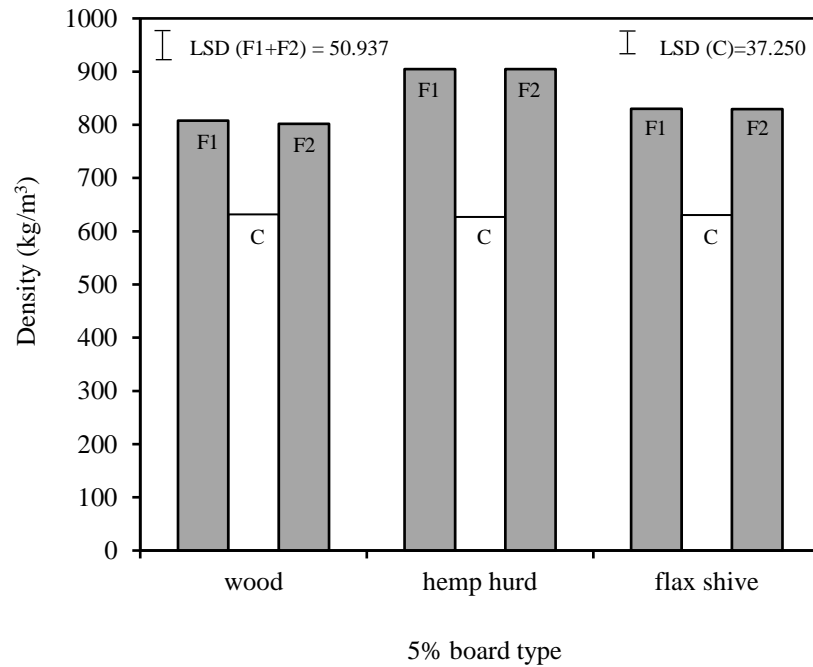
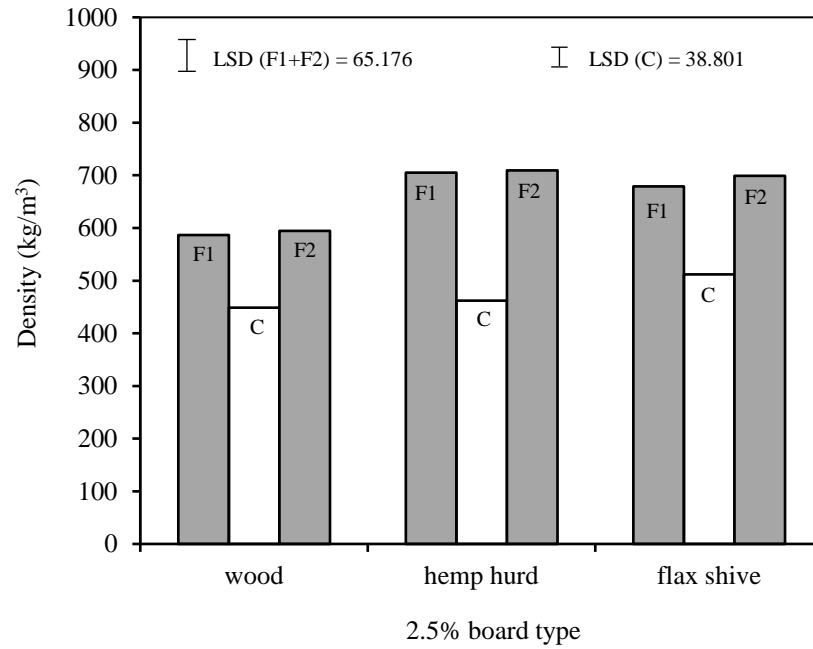


Figure 3.4: Face and core density profiles of low density particleboards, expressed as peak face (F1, F2) and core densities (C). $n=6$ for each mean. Error bars represent least significant difference (LSD) between means.

For the 2.5% resin load boards, significant differences were observed in the VDP between the wood, hemp hurd and flax shive panel faces ($p=0.0006$) ranging from 586-709 kg/m³ and the core ($p=0.0017$) from 448-512 kg/m³. The lowest face and core densities were observed in the wood panels. The VDP of the 5% resin load boards revealed no significant differences between panel types in terms of their core densities which ranged from 627-632 kg/m³; of the 3 panel types the hemp hurd yielded the lowest core density. There was however a significant difference ($p=0.0014$) in peak face densities ranging from 802-905 kg/m³; the highest face density was observed in panels manufactured from hemp hurd, with no difference between the wood and flax shive panels.

Moisture is one of the factors that influences the formation of a density gradient in particleboard. This is because it serves as a means of heat transfer from the mat faces in contact with the press platen to the mat core to assist polymerization of the resin (curing/hardening) and consolidation of the mat. Prior to the 2.5% and 5% resin load board manufacture the MC determined for both the hemp hurd and flax shive residues was 10.10% and 9.8% respectively. Irrespective of having a similar MC for the same resin load (i.e. 2.5% or 5%), higher face densities were consistently observed in the hemp hurd particleboards compared with the flax shive particleboards. A look at the maximum mat pressures observed for the particleboards during the hot-pressing operation reveals the hemp hurd boards yielded the highest mat pressure (Figure 3.5) even though the same press cycle was used for manufacturing all particleboard types. This indicates that the hemp hurd and flax shive residues have more resistance to compaction compared with the wood residues which are more easily compressed. A comparison of the face and core vertical density variations for all the particleboard types is presented in Table 3.5. From the table, it is evident that hemp hurd boards have a higher face to core density variation through its board thickness compared to the flax shive and wood boards because of its higher face and lower core densities.

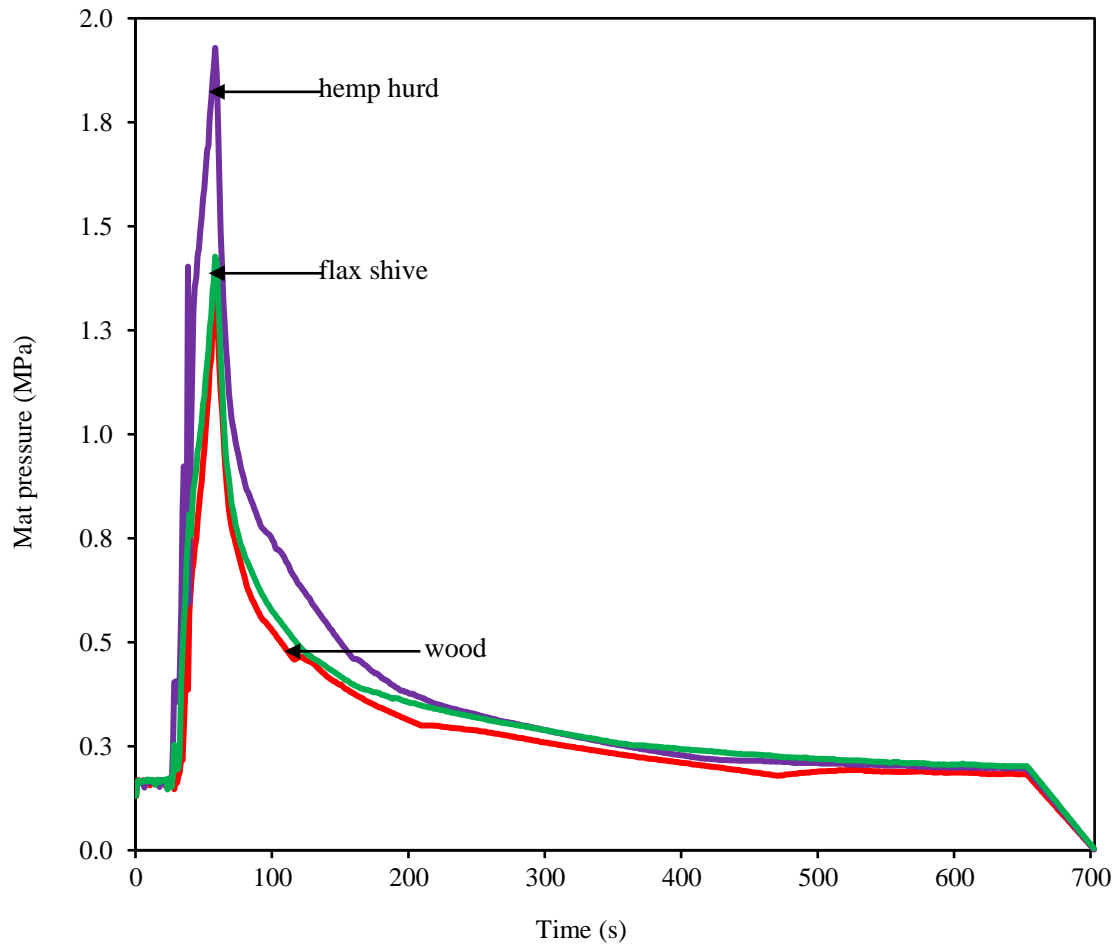


Figure 3.5: Differences in compressive force plotted as a function of time during hot pressing.

Table 3.5: Face and core layer density variations for 2.5% and 5% resin load boards.

Board Type	wood		hemp hurd		flax shive	
	2.5%	5%	2.5%	5%	2.5%	5%
average board density	555	638	532	631	533	657
face/ board density	1.06	1.26	1.33	1.43	1.29	1.26
core/ board density	0.81	0.99	0.87	0.99	0.96	0.96
face: core variation	0.26	0.27	0.46	0.44	0.33	0.30

To eliminate the effect that differences in MC have on the formation of the VDP, samples of wood, hemp hurd and flax shive residues were conditioned to 11% MC in a chamber held at $65 \pm 5\%$

relative humidity and 20 ± 3 °C for 3 weeks and subsequently used for board manufacture with 5% pMDI resin load. Results of the face and core vertical density variations are presented in Table 3.6 and Figure 3.6.

Table 3.6: Face and core layer density variations for particleboards manufactured with residues conditioned to 11% moisture content.

Board Type	wood	hemp hurd	flax shive
average board density	595	613	603
face/ board density	1.25	1.42	1.41
core/ board density	0.93	0.94	0.96
face: core variation	0.32	0.48	0.44

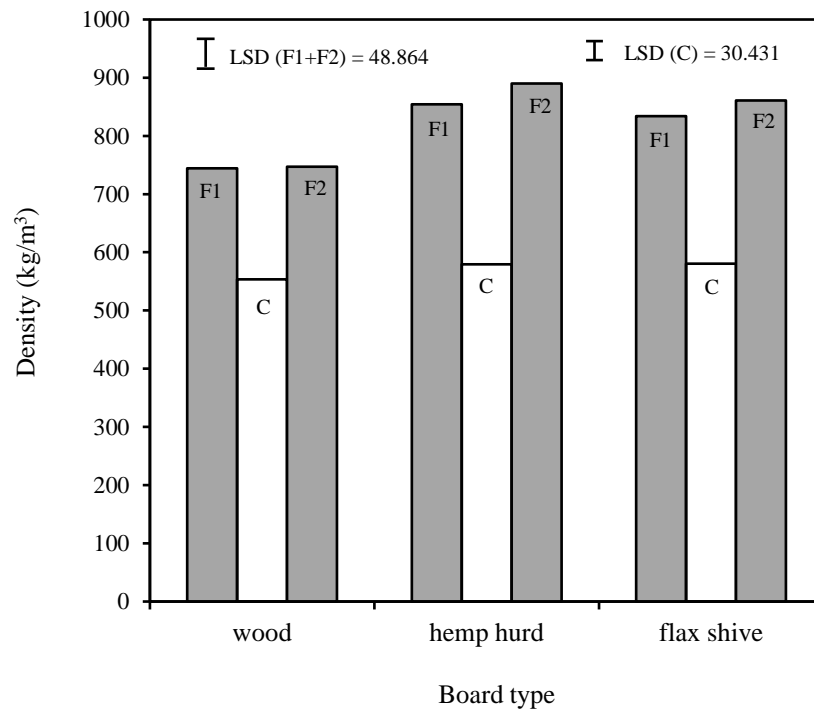


Figure 3.6: Face and core density profiles of particleboards manufactured with conditioned residues, expressed as peak face (F1, F2) and core densities (C). $n=6$ for each mean. Error bars represent least significant difference (LSD) between means.

There were no significant differences in average board density between the particleboard types manufactured from the conditioned furnish. The hemp hurd particleboards once again yielded significantly higher average face density (872 kg/m^3) resulting in a larger face to core density variation (Table 3.6), closely followed by the flax shive particleboards (848 kg/m^3) and the lowest occurring in the wood particleboards (746 kg/m^3). In terms of core density, a high value was observed in the flax shive boards (580 kg/m^3) but this was not significantly different from that of the hemp hurd (579 kg/m^3) and wood boards (553 kg/m^3).

These results suggest that there are other factors aside MC which are influencing the density gradient within the different particleboard types. In fact, other factors such as the particle configuration within the layers of the formed mat, cellular structure and the compressive strength of the constituent residue all impact the formation of the density gradient (Dai and Steiner 1993). A detailed look at the cell structure of the hemp hurd tissue compared to the flax shive (Figure 3.7) shows thicker cell walled vessels in the hemp hurd which explains the greater force required (Figure 3.5) to compress its mat to the same board thickness as the flax shive residue.

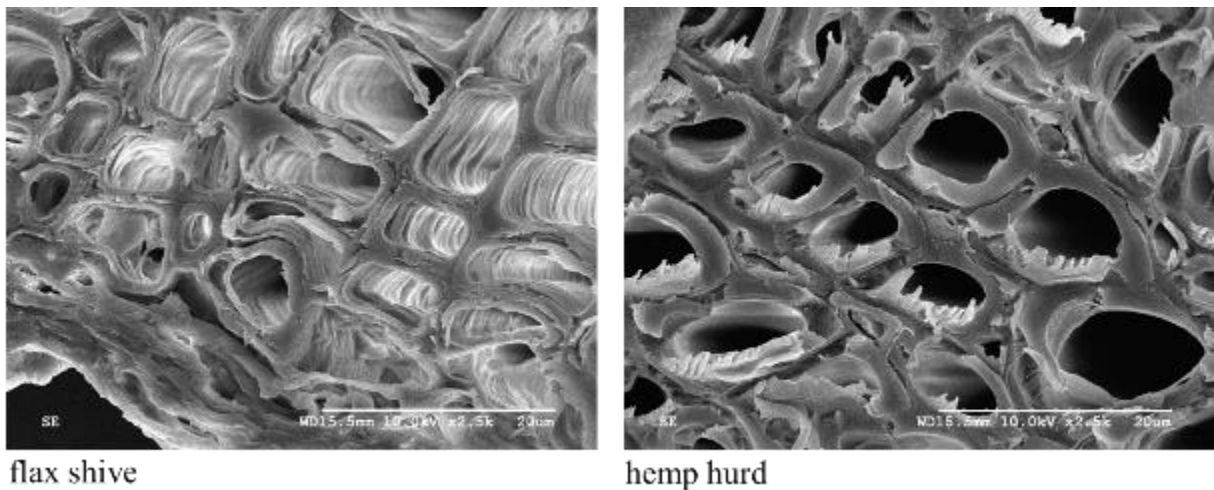


Figure 3.7: SEM image of vessels in flax shive and hemp hurd residues. Source: Sam-Brew 2016.

3.3.4 DSC analysis

Figure 3.8 presents the complied DSC graphs for a combination of wood, hemp hurd and flax shive residues with pMDI resin where the distinct single exothermic peak observed represents the curing reaction. Figure 3.8a represents the oven dry residues and pMDI resin mixture in the absence of moisture and Figure 3.8b represents the residues and resin mixture combined with distilled water. Results of the average onset temperatures, peak temperatures and reaction heats extracted from both graphs are listed in Table 3.7 as ‘moist’ for samples with distilled water and ‘dry’ for samples without water. A dynamic scan of pMDI resin alone without distilled water (dry) displayed no peak within the temperature range 40 °C to 230 °C indicating the resin did not undergo any exothermic reaction.

From table 3.7, samples of the oven dry residue and pMDI resin mixture (dry) yielded higher temperatures for the onset of the curing reaction and reaction rates, and lower heat of reactions in comparison to the same residues combined with pMDI resin and distilled water (moist). The results indicate a lower degree of cure in the pMDI resin with the residues in the absence of moisture. These results are in line with observations made by He and Yan 2005 for aspen flour reacted with pMDI resin under oven dry, 6% and 12% moisture content. It is important to note that the term “degree of cure” does not refer to the extent of resin cure but rather to the quantity of possible bond formation between the resin molecules, because the temperature range used in testing ensures the resin is 100% cured by the end of the cycle.

Generally, the predominant mechanism of adhesion of pMDI resin with wood containing moisture has been proven to be through hydrogen bonding and mechanical interlocking by the formation of polyurea networks between the resin and the water in the wood (Wendler and Frazier 1996; Bao et al. 2003; Smith 2004; He and Yan 2007). The second minor method of adhesion that is known to occur between pMDI resin and wood is a chemical covalent bonding which theoretically occurs between the hydroxyl groups of the wood components such as the cellulose, hemicelluloses and lignin, and the pMDI resin to form urethane bonds (Pizzi and Owens 1995). This method of bonding is however dependent on the accessibility and reactivity of the hydroxyl groups to the pMDI resin. Pizzi and Owens (1995) in their study demonstrated this by reacting dry cellulose and dry wood flour separately with pMDI resin. In both cases two exothermic reaction peaks were identified in the same temperature range (128-172 °C and 241-295 °C) which was higher than that

observed in pMDI:water samples (87-117 °C). The authors attributed this to the formation of covalent bonds between the resin and dry wood/dry cellulose in the absence of water.

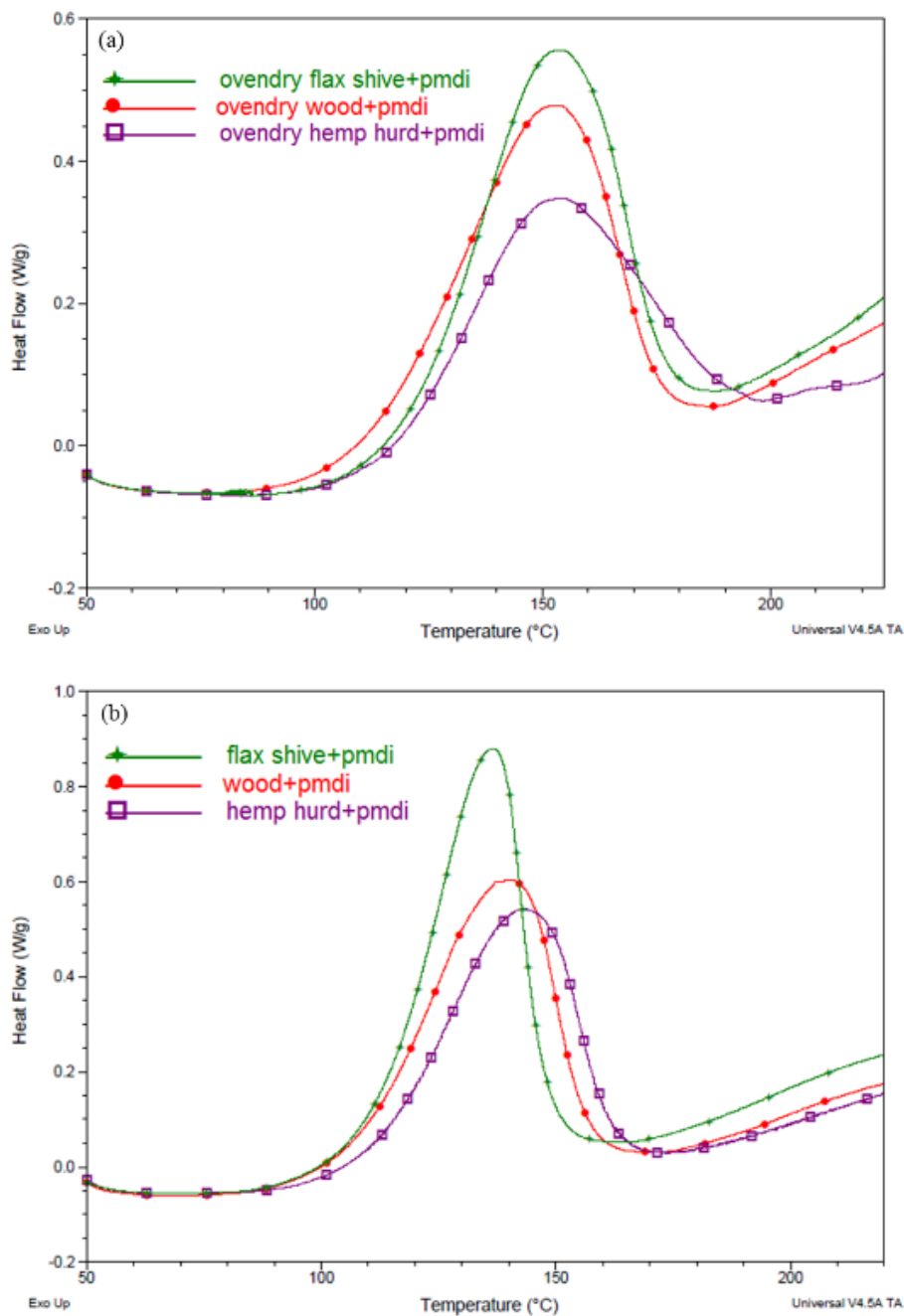


Figure 3.8: DSC curves of the curing reaction between (a) oven dry wood, hemp hurd, flax shive residues and pMDI resin and (b) similar residues combined with pMDI resin and distilled water.

Table 3.7: Thermal properties of curing reaction between pMDI resin and residues.

Sample	Onset Temp. (°C)		Peak Temp. (°C)		Reaction heat (J/g)	
	moist	dry	moist	dry	moist	dry
pMDI	106.7	—	130.5	—	235.3	—
wood+pMDI	107.9	113.1	138.5	151.6	118.7	108.3
hemp hurd+pMDI	112.7	118.1	142.5	151.9	112.8	89.6
flax shive+pMDI	111.2	121.1	136.2	153.1	125.0	115.0

Also interesting from their study was the fact that the pMDI:dry wood samples attained lower activation energies in contrast to the pMDI:dry cellulose samples, suggesting a lower activation energy of the curing reaction between the pMDI and dry wood flour compared to the dry cellulose samples. These results point to the fact that if cellulose was the only component that activated the curing reaction, then with lower quantities of the cellulose in the pMDI:dry wood system it should have yielded higher activation energies for both exothermic peaks, which was not the case. Indicating that the presence of other components such as hemicelluloses and lignin in the wood with reactive hydroxyl groups encouraged the faster reaction values (lower activation energies) observed. Discussions in subsequent paragraphs will therefore focus on the combination of the different residues, pMDI resin and distilled water samples which are representative of the conditions observed in wood composite formation.

The effect of residue type on pMDI curing reaction is significant ($p < 0.0001$) compared with neat pMDI resin (Appendix B). The addition of the different residues to the pMDI resin significantly reduces the reaction heat. Why is this so? One would expect that in the presence of moisture and with the introduction of the residues which each contain cellulose, hemicelluloses and lignin constituents with reactive hydroxyl groups, a greater number of bonds is likely to be formed between the residue and pMDI mixtures than in the neat pMDI samples. Yet this is not the case. As explained earlier, the ability of the pMDI resin to react with the reactive hydroxyl groups will depend on their accessibility and reactivity. The hydroxyl groups in wood for instance have been reported to have a low mobility in their reaction with pMDI resin (He and Yan 2005). Since it has been established that the bonding mechanism between pMDI and wood flour is mainly by mechanical interlocking (when it penetrates the cellular structure) with quite minor covalent bond formation, then the reaction heat evolved from the DSC analysis must be from chemical bond

formations between the pMDI resin molecules. Therefore, the lower reaction heat values obtained for the residue and pMDI mixtures indicates a lower number of bond formation between the pMDI resin molecules. From table 3.7, there was a decrease in the percentage of reacted pMDI molecules by as much as 50% in the case of the wood, 52% for the hemp hurd and 47% for the flax shive residues. The significant decrease observed here could be the effect of the chemical components (extractives, silica, ash content) of the residues on the resin crosslinking and the ease with which the pMDI resin can access the reactive sites (cellulose, lignin, hemicellulose) in the residues, which is dependent on the intrinsic anatomy. For instance, Das et al. 2007 using solid-state NMR with nitrogen-labeled (N^{15}) pMDI resin bonded with 2 different wood species (southern yellow pine and yellow-poplar) reported a minor but statistically significant effect of the wood species on the cure chemistry. Bao et al. 1999 also reported that wood species (southern pine and aspen) influenced the rate of reaction of the pMDI as a function of temperature and moisture content.

The peak temperatures for the residue and pMDI mixtures also shifted to higher values and were significant ($p=0.0010$) in comparison to the neat pMDI sample signifying slower cure rates as well as providing information on the typical press platen temperatures that should be employed during the hot-pressing operation to ensure crosslinking of the pMDI resin (Appendix B). In terms of onset temperature, the neat pMDI (107 °C) and wood and pMDI (108 °C) samples had early onset of curing and were significantly different ($p=0.0014$) from the hemp hurd and pMDI (113 °C) and flax shive and pMDI (111 °C) samples (Appendix B). These onset temperatures suggest the curing reaction proceeds easily and at lower temperatures for the pMDI resin in the presence of the flax shive and wood residues.

With respect to the residues, based on the superimposed graphs (Figure 3.8b) and the values from table 3.7 (moist), the lower onset temperature value of 108 °C for the wood samples indicates an early start of curing of the pMDI resin in the presence of the wood residue, followed by the flax shive (111 °C) and the hemp hurd (113 °C) residues. The delayed onset of curing observed for the hemp hurd and flax shive residues maybe the result of their waxy surfaces. The reactivity of the pMDI resin in the presence of the residues is also confirmed by the peak temperatures which represents the rate of curing of the resin. Here lower temperatures and hence faster reaction cure rates were once again observed in the flax shive and wood samples, and the highest temperature indicating a slower cure rate was observed in the hemp hurd samples. A significant difference

($p=0.0029$) was observed in peak temperature between all 3 samples (Appendix B). The amount of heat produced at the end of the curing process (i.e. reaction heat) was highest in the flax shive samples and lowest in the hemp hurd samples; indicating a greater resin cure in the flax shive and pMDI mixture compared to the wood and hemp hurd samples. The hemp hurd and pMDI samples therefore exhibited the lowest degree of resin cure.

In summary, the DSC results indicate that of the 3 lignocellulosic residues, the hemp hurd has a significant prohibitive effect on the cure of pMDI resin. For particleboard manufacturers, this implies the need to increase press temperatures during particleboard production when using hemp hurd residues to ensure efficient bonding of the pMDI resin with the residue. The reaction heat results observed based on residue type remains unexplained and can be the emphasis for further studies.

3.3.5 Mechanical properties

3.3.5.1 IB

The internal bond (IB) strength for the different particleboard types are shown in Figure 3.9. Research has shown that for particleboard panels an increase in board density is associated with a corresponding increase in IB strength: the result of more contact between many particles and hence fewer voids in the mat as it is being compressed to a higher density in the pressing operation. For the 2.5% boards (Figure 3.9a) significant differences ($p<0.0001$) were observed between all panel types (Appendix B); within the same density range (500–600 kg/m³) the hemp hurd panels yielded the highest IB. There was a wide variation in IB strength for the wood panels correlating to the density variations within the panel and the low density core that was observed in its density gradient; approximately 19% of the wood samples yielded the lowest density and hence low IB strength. With the 5% boards on the other hand within a density range of 600–750 kg/m³ the wood panels yielded a significantly ($p<0.0001$) higher IB (Figure 3.9b); there were no statistically significant differences between panels manufactured from the flax shive and hemp hurd panels (Appendix B).

Results of the IB tests are not in line with the observations made in the DSC tests where the flax shive residues yielded significantly higher reaction heat values and hence a high degree of pMDI resin crosslinking. The lowest IB strength was consistently observed in the flax shive panels for

both the 2.5% and 5% boards. Although all the particleboard types exceeded the voluntary 0.15 MPa and 0.45 MPa minimum IB strength required by ANSI A208.1-1999 for low density (LD2) and medium density (M2) grade particleboard respectively, there is an indication that other factors aside the reactivity of the resin during the curing reaction (such as the rate of heat transfer to the mat core, residue cellular structure) are affecting the cohesive strength properties of the particleboard. Using image analysis (Image J) the core wood, hemp hurd and flax shive residues that had previously been measured for their aspect ratios were analyzed in terms of surface area – hemp hurd (20.58 mm²), wood (17.96 mm²) and flax shive (13.55 mm²). The medium sized rectangular hemp hurd particles yielded the largest surface area available for resin coverage while the shorter and thicker wood core particles came in second, hence the higher bond strengths for the hemp hurd and wood residues in the 2.5% and 5% boards. The wood residues because of their short and fine particle geometry as seen in Figure 2.5 also had the extra advantage of better packing as the particles easily filled the voids created during mat formation therefore ensuring greater particle-particle contact during mat densification (Maloney 1993, Sackey et al. 2008).

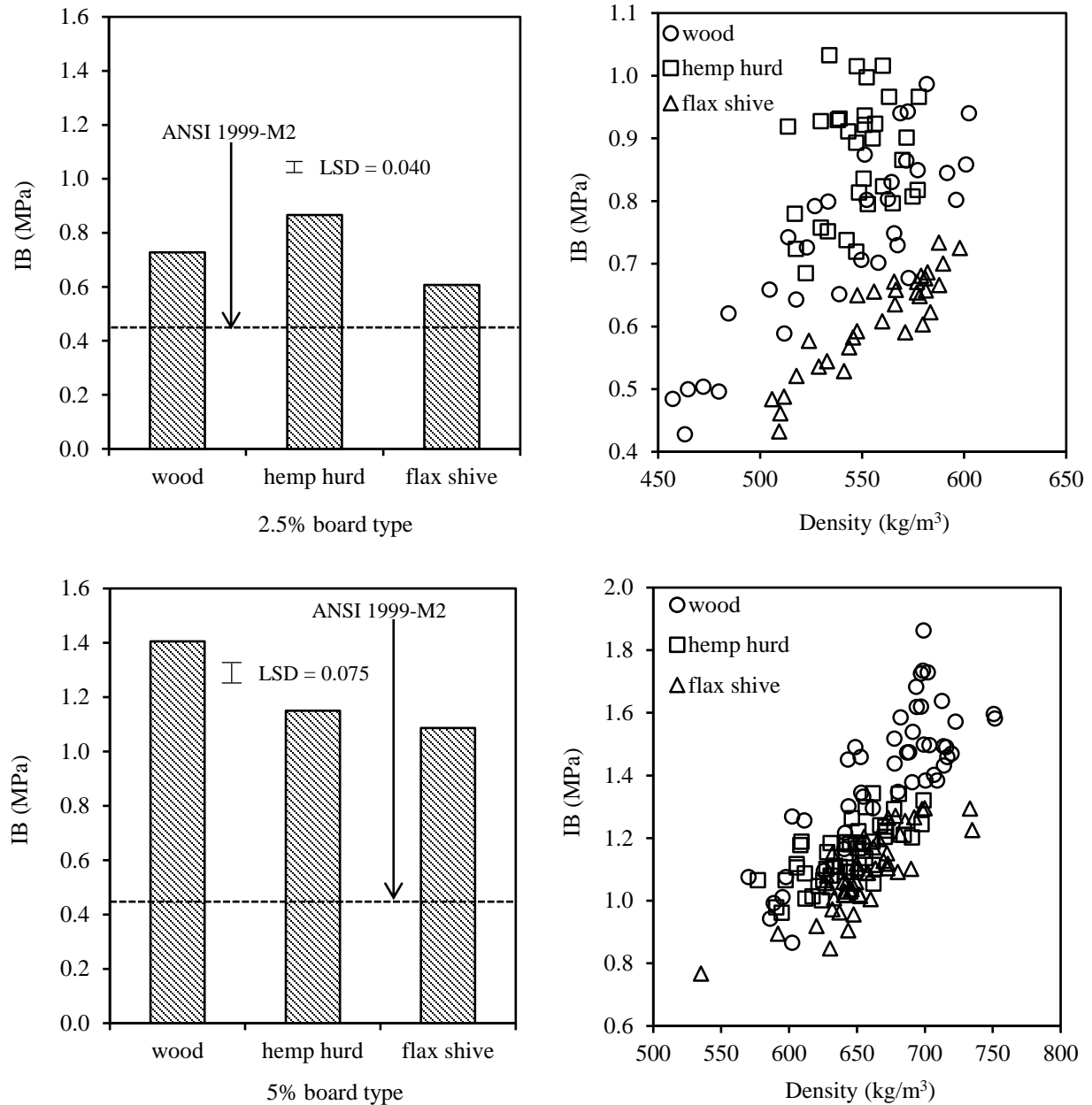


Figure 3.9: Internal bond strength of low density pMDI particleboards. Horizontal line indicates minimum value stipulated by ANSI A208.1-1999 for M2 boards. $n=32$ for each mean. Error bar represents least significant difference (LSD) between means.

As is expected with increase in resin content, the IB values were higher for the 5% resin load boards compared to the 2.5% resin load boards. Irrespective, values observed in the flax shive and hemp hurd boards were superior and or comparable to particleboards manufactured from similar

residues in other studies taking into consideration the differences in some processing parameters such as resin type, resin content, press time and board densities. Papadopoulos and Hague (2003) manufactured single-layer flax shive particleboards to a target density of 750 kg/m³ and thickness 17.5 mm using 13% UF resin; the boards yielded IB values of 0.09 MPa which is quite low. Balducci et al. (2008) working with hemp hurd also manufactured 16 mm thick single-layer boards with densities of 400 kg/m³ using 6% isocyanate resin and reported IB strength of 0.32 MPa which given the lower density when extrapolated (linear best fit) from the current study data for both 2.5% and 5% resin load pMDI boards (Figure 3.9) is still quite lower compared with the current results.

3.3.5.2 MOR and MOE

Particleboard is a random blend of residues which requires no material orientation during mat formation. In this study the mats were formed by hand in a square forming box and no bias towards the forming direction (i.e. parallel to the normal axis of the panel) was expected. As stated earlier in section 2.2.4, .3 test samples for the bending strength (MOR) and stiffness (MOE) properties were taken parallel and perpendicular to the forming direction. Analysis of the test data revealed no significant difference between samples from both parallel and perpendicular directions. The bending strength properties (MOR and MOE) presented in Table 3.8 and Figure 3.10 for the 2.5% and 5% resin load particleboard types are therefore pooled data of both sample directions.

Table 3.8: Static bending properties of pMDI-bonded particleboards. Data is based on ANSI A208.1-1999 panel averages. n=12 for each mean.

Board Type	MOR (MPa)		MOE (GPa)	
	2.5%	5%	2.5%	5%
wood	4.07 (1.54)	7.06 (1.56)	1.06 (0.29)	1.50 (0.26)
hemp hurd	12.40 (1.83)	17.90 (2.27)	2.09 (0.28)	2.72 (0.34)
flax shive	10.01 (2.32)	18.24 (1.83)	2.14 (0.32)	3.29 (0.24)
ANSI LD2	5.0		1.03	
ANSI M2	14.5		2.25	

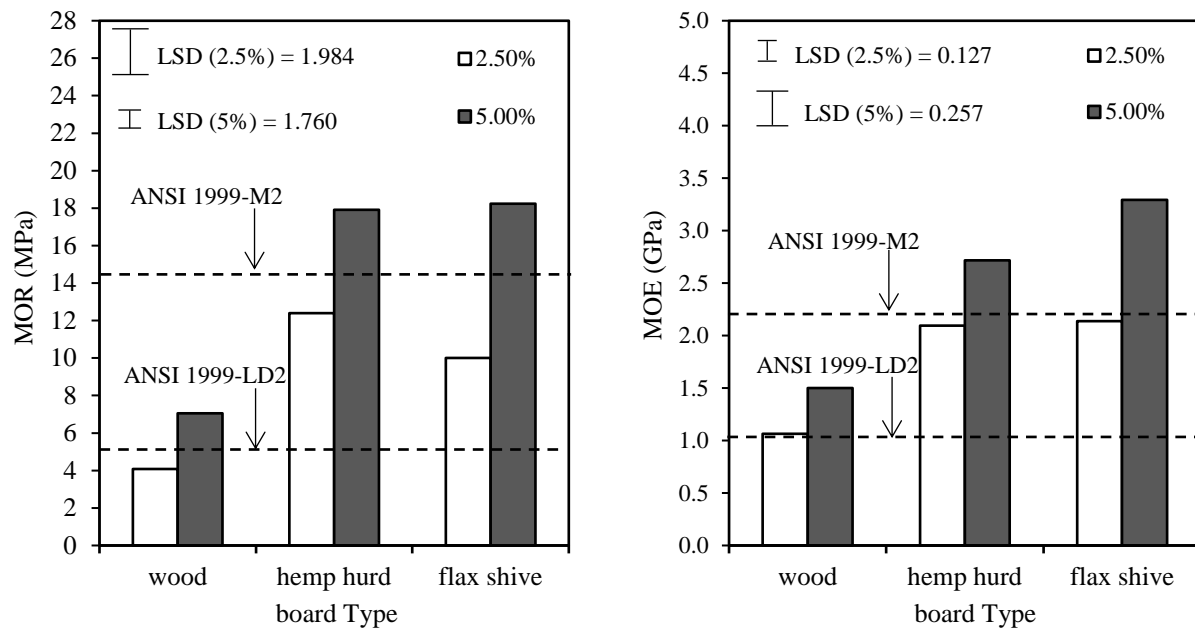


Figure 3.10: Bending strength and stiffness properties of particleboards manufactured with 2.5% AND 5% pMDI resin load. Horizontal line indicates minimum value stipulated by ANSI A208.1-1999 for LD2 and M2 grade particleboards. $n=12$ for each mean. Error bar represents least significant difference (LSD) between means.

Particleboard VDP and residue aspect ratio have been reported to significantly influence the MOR and MOE of particleboard panels (Kelly 1977, Maloney 1993). High density faces observed in the vertical density analysis of a board denote highly compact regions that were developed because of plasticization and compression of the residues. As the face density increases the higher the compaction within the board (implying greater inter-particle contact) and the better its ability to resist bending stresses that are applied to it. Also, residues which are longer and thinner as discussed in section 3.3.1 tend to have more potential bonding sites and therefore provide greater contact area/overlap between particles for bonding; this helps resist the stresses developed during bending resulting in stronger and stiffer particleboards.

The hemp hurd and flax shive particleboards consistently exhibited higher face densities (Figure 3.4) compared to the wood particleboards even when all furnish types were conditioned to the same MC (Figure 3.6). This coupled with their high length to width ratios resulted in boards with

significantly ($p < 0.0001$) greater bending strength, approximately 60%, compared to the wood boards within the same resin load category (Appendix B). The difference in MOR and MOE values for similar particleboard types (wood, hemp hurd or flax shive) in the 2.5% and 5% resin load boards can further be attributed to the increase in board density (Table 3.4) which generally results in an increase in bending strength and stiffness properties as previously explained.

For the reduced pMDI resin load of 2.5% only the flax shive and hemp hurd panels exceeded the standards for LD2 grade particleboard in terms of MOR. For MOE, all panel types met the ANSI 1999 requirements for LD2 particleboard. Both hemp hurd and flax shive panels for the 5% resin load boards surpassed the stipulated ANSI specifications for both LD2 and M2 grade particleboard. Panels manufactured from the wood residues on the other hand only met the standards for LD2 particleboard in terms of MOR and MOE.

3.3.6 Physical properties

3.3.6.1 LE

Particleboard when exposed to high humidity absorbs moisture and expands in volume. These dimensional changes are the result of the hygroscopic nature of its constituent materials and the release of the compressive stresses which were set in the boards during the hot-pressing operation. These changes are not entirely reversible upon drying, becoming a great concern in applications where particleboards will be exposed to large changes in moisture – liquid water or water vapor.

The linear expansion (LE) with changes in moisture content between 50% and 90% relative humidity at 20 ± 3 °C for the wood, hemp hurd and flax shive boards with different resin contents are presented in Figure 3.11. Particleboards manufactured from the wood residues yielded the highest linear expansion, significantly so ($p < 0.0001$) compared with the flax shive and hemp hurd panels (Appendix B). This is to be expected as linear expansion is more dependent on the particle geometry and particle alignment than on board density. As such an increase in particle length typically results in a decrease in linear expansion (Miyamoto et al. 2002), emanating from the fact that the longer particles are more likely to have longer longitudinal sections where less dimensional

changes occur. The geometry of the flax shive particles as seen in Figure 3.2 confirm its lower LE values.

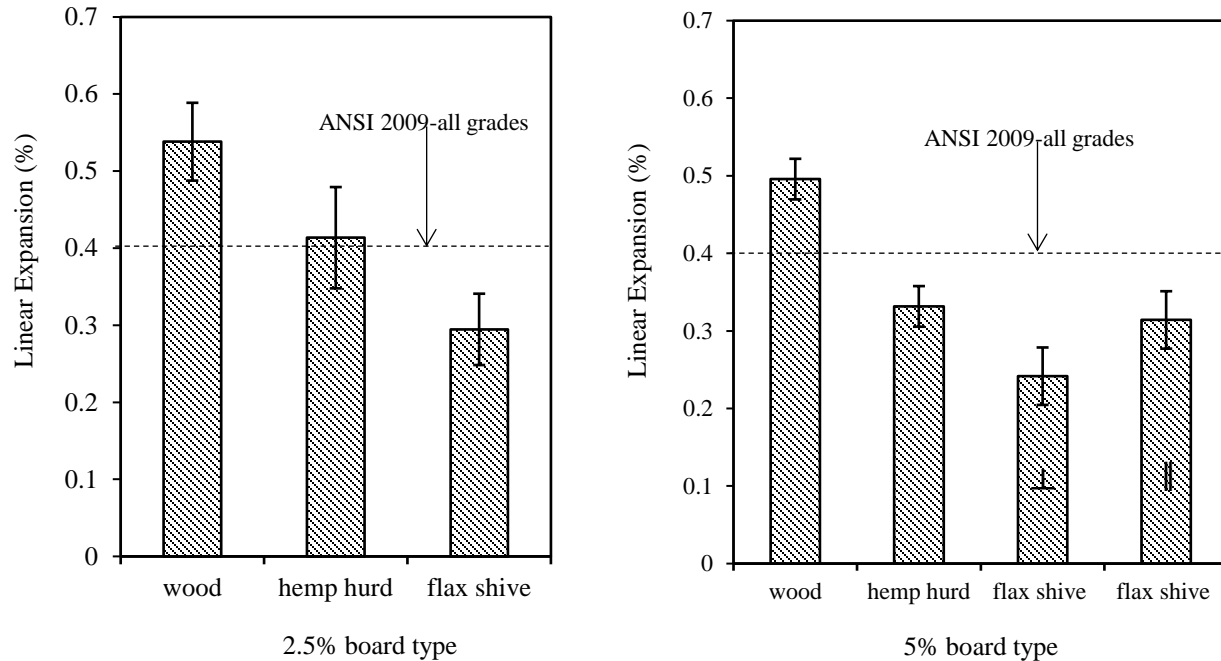


Figure 3.11: Linear expansion with changes in moisture content between 50% and 90% relative humidity: parallel (||) and perpendicular (⊥) to forming direction. Horizontal line indicates maximum value stipulated by ANSI A208.1-2009. Each mean was computed from 16 samples for the 2.5% boards, 20 samples for the 5% wood and hemp hurd boards, and 10 samples for the 5% flax shive (||) and (⊥) boards. Error bars represent 95% confidence intervals.

Since mats were randomly formed the linear expansion is expected to be equal in the directions parallel and perpendicular to the forming direction. Comparison of test samples from both directions for each particleboard type revealed no significant differences for the 2.5% and 5% resin load boards with exception of the flax shive particleboards manufactured with 5% resin load where changes in length were greater in the direction parallel to the forming direction than in the perpendicular direction. This is not surprising as it has been suggested that despite the random orientation employed during mat formation in particleboard manufacture some particles tend to orient themselves at angles to the forming plane (Kelly, 1977), hence greater changes in length occur in the plane parallel to the forming direction. It is important to note that the LE in

particleboard when compared to solid wood is generally greater in the longitudinal direction and much less in the radial.

Increasing the resin content of the particleboard types appears to have a slight but not significant reduction effect on their LE values. Boards manufactured with the 5% resin load compared with the 2.5% resin load boards have LE values reduced by approximately 5% for the wood and hemp hurd particleboards and 10% decrease in the case of the flax shive particleboards parallel to the normal axis of the board. Linear expansion values observed in the flax shive and hemp hurd particleboards manufactured with the 5% resin load were within the limits stipulated by the ANSI A208.1-2009 standards. In the case of the 2.5% resin load boards only the flax shive particleboards (a pooled effect of samples parallel and perpendicular to the forming direction) yielded values within the ANSI limits while the hemp hurd boards at 0.41% just exceeding the stipulated 0.40% maximum average percent. The values observed for the wood particleboards in both the 2.5% and 5% resin load boards exceeded the maximum permitted LE value outlined in ANSI A208.1-2009.

3.3.6.2 TS and WA

Table 3.9 and Figure 3.12 shows the 2 and 24 hours thickness swell (TS) and water absorption (WA) properties of wood, hemp hurd and flax shive particleboards. Notice from the figure that the error bars are not equal for some responses though all 3 particleboard types had an equal number of samples: this is because the data set were transformed (logarithm and reciprocal transformations) to ensure that all analysis met the assumptions regarding the error term as discussed in section 2.2.5.

For the 2.5% resin load boards, the wood particleboards yielded significantly ($p < 0.0001$) lower TS (approximately 60% less) and WA (approximately 45% less) values for both 2 and 24 hour measurements compared to the flax shive and hemp hurd particleboards which had relatively similar values (Appendix B). The high TS values attained by the flax shive and hemp hurd particleboards on the other hand can be attributed to their highly absorbent nature which has been well documented by Papadopoulos and Hague 2003, Nguyen et al. 2009 and Arnaud and Gourlay 2012.

Table 3.9: Thickness swell and water absorption properties of pMDI-bonded particleboards. Each mean was computed from 12 samples for the 2.5% boards and 11 for the 5% boards.

Board Type	2TS (%)		24TS (%)	
	2.5%	5%	2.5%	5%
wood	2.53 (0.57)	7.02 (3.28)	6.67 (0.77)	10.87 (2.72)
hemp hurd	6.11 (0.54)	5.78 (1.28)	16.54 (0.76)	14.13 (1.83)
flax shive	6.11 (1.59)	5.86 (0.63)	18.44 (3.97)	16.67 (1.25)
Board Type	2WA (%)		24WA (%)	
	2.5%	5%	2.5%	5%
wood	7.02 (0.69)	28.02 (18.54)	26.75 (2.63)	39.48 (18.30)
hemp hurd	15.85 (0.89)	12.01 (2.60)	48.47 (1.87)	33.51 (3.95)
flax shive	15.57 (2.58)	11.46 (1.03)	45.76 (7.76)	33.80 (2.16)

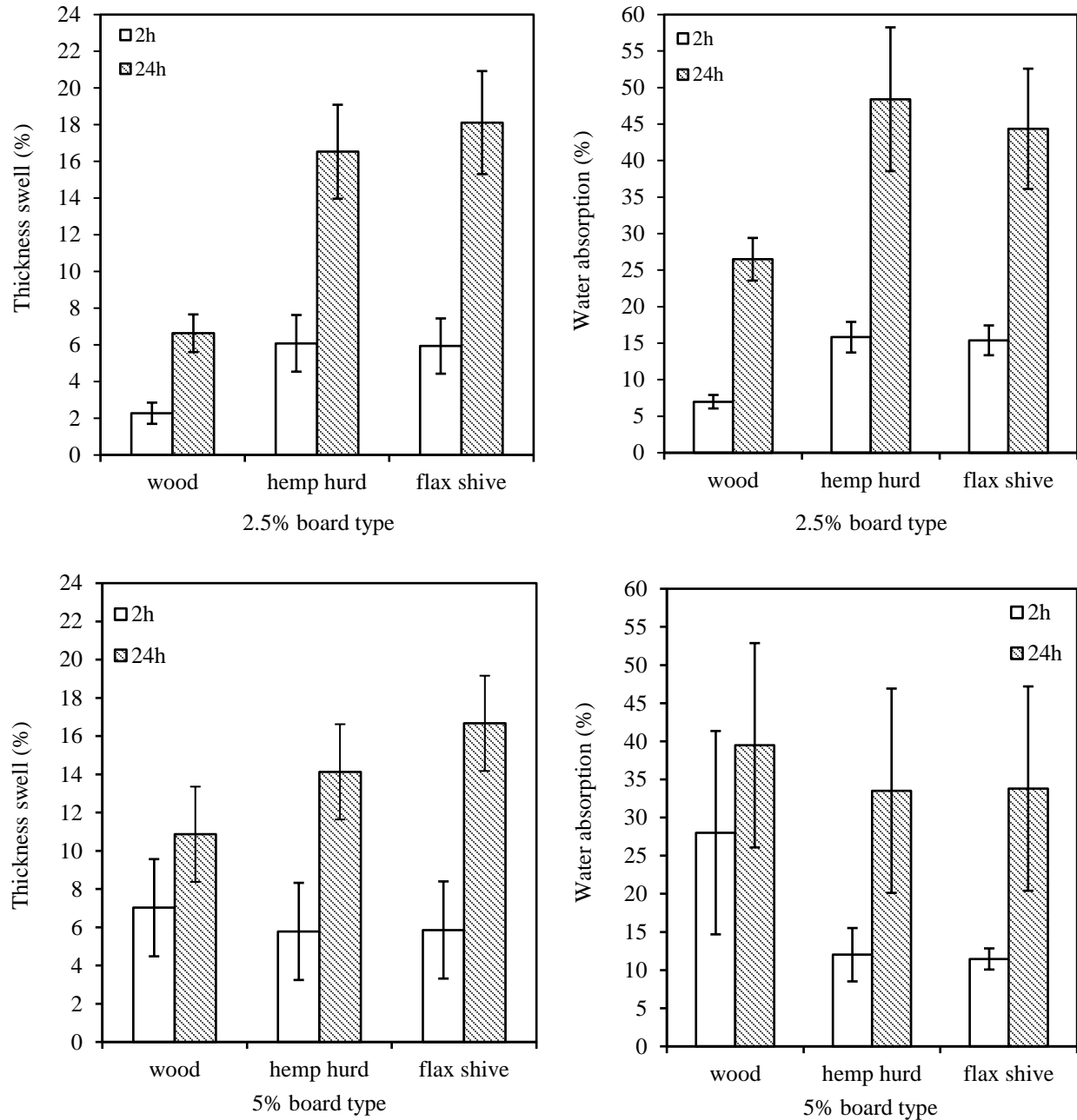


Figure 3.12: Thickness swell and water absorption characteristics of 2.5% and 5% pMDI resin load particleboards for 2h (2 hours) and 24h (24 hours). Each mean was computed from 12 samples for the 2.5% boards and 11 for the 5% boards. Error bars represent 95% confidence intervals.

In boards manufactured with 5% resin load, the wood particleboards swelled the most in thickness (7%) in the first 2 hours of submersion, absorbing twice the amount of water (28%) significantly

($p=0.0014$) so compared to the flax shive and hemp hurd particleboards (11.5 and 12% respectively) (Appendix B). After 24 hours of submersion the flax shive particleboards record the highest TS (16.7%); with significant ($p<0.0001$) differences observed between all 3 particleboards types. Overall no significant differences were observed between the percentages of water uptake by the wood, hemp hurd or flax shive particleboards for the 5% resin load boards. The unusually high variability observed within the wood particleboards is unexplained and is likely the result of non-uniform particle distribution during hand forming of mats.

It is important to note that no wax was used in board manufacture. A comparison of the 2.5% and 5% resin load boards revealed that with exception of the wood particleboard, particleboards made from hemp hurd and flax shive residues with 2.5% resin load yielded the highest TS and WA values. This is likely due to boards with the higher 5% resin load having better inter-particle bonding and therefore enhanced dimensional stability. But this is also contrary to conventional understanding that boards of a higher average density (in this case the 5% resin load boards as seen from Table 3.4) tend to have higher compressive stresses set within the panels during manufacture and these stresses are released upon submersion into water, resulting in greater spring back. This theory is certainly true for the wood particleboards which consistently yielded higher TS values for the 5% resin load boards compared with the lower 2.5% resin load boards.

The ANSI standards do not stipulate maximum values for thickness swell or water absorption for medium or low density particleboard, so there was no benchmark for evaluation. The results obtained are however much less than those observed by Balducci et al. (2008) who reported 28.3% thickness swell for a 6% isocyanate-bonded hemp hurd particleboard of a lower density (400 kg/m^3) than particleboards from the current study. Considering the fact that boards with relatively higher densities tend to have more relaxation of compressive stresses and spring back upon submersion into water (Kelly 1977), one would expect the hemp hurd boards from this study with lower resin contents (2.5% and 5%) and higher densities ranging from $540\text{--}662 \text{ kg/m}^3$ to show higher thickness swell values compared to the results reported by Balducci et al. (2008) but this is not the case.

3.3.7 Comparison of flax shive and hemp hurd particleboard properties with particleboard from other non-wood residues

A comparison of the mechanical and physical properties obtained for the flax shive and hemp hurd particleboards produced in this study with those from other non-wood or agricultural residues is presented in Table 3.10. From the table, though the flax shive and hemp hurd particleboards in the current study have a lower density, the boards with the lower 2.5% pMDI have MOR values similar to coconut chip particleboard produced with 2% pMDI to a higher density and thickness. Likewise, the IB values obtained for the hemp hurd and flax shive particleboards manufactured from both 2.5% and 5% pMDI resin load are higher than those reported for the coconut chip particleboards produced with 2-8% MDI resin.

Generally, the information provided in the table suggests that flax shive and hemp hurd residues like other well know agricultural residues can be used to produce particleboards with comparable physical and mechanical strength properties.

Table 3.10: Comparison of the strength properties of particleboards manufactured from isocyanate resin and non-wood residues.

Residue type	Thickness (mm)/layer	Density (kg/m ³)	Resin (load %)	MOE (GPa)	MOR (MPa)	IB (MPa)	TS-24h (%)
flax shive	12.7/3	533-657	pMDI (2.5, 5)	2.14-3.29	10-18.2	0.61-1.09	16.7-33.8
hemp hurd	12.7/3	532-631	pMDI (2.5, 5)	2.09-2.72	12.4-17.9	0.87-1.15	14.1-33.5
bagasse Wu, 2001	7.2-7.4/1	800-940	pMDI (5, 8)	2.3-3.79	19.1-27.9	1.6-2.7	11.9-15.6
coconut chips Papadopoulos et al. 2002	17.5/1	720-760	MDI (2,4,6,8)	—	10.6-20.9	0.38-1.21	7.1-36.8
wheat straw Mo et al. 2003	6.5/1	700	MDI (4)	2.28	18.1	0.64	27.3
rice straw Pan et al. 2006	6.5/1	700	pMDI (4)	1.76	15.5	0.32	27.5

3.3.8 Cost competitiveness of wood versus flax shive/hemp hurd

The findings indicate that based on performance the flax shive and hemp hurd particleboards have the potential value to compete with wood particleboards. However, there are several factors that hinder the use of the flax shive and hemp hurd residues for particleboard production.

The first major issue being the high cost of the residues which makes it difficult to use the residues economically for the manufacture of particleboard in comparison to wood. As mentioned previously, flax shive and hemp hurd residues are by-products of the flax/hemp straw primary processing to obtain flax and hemp fiber. This primary processing is capital intensive in terms of the processing machinery employed and facilities required. Thus, the flax shive and hemp hurd residues obtained are sold at relatively higher price to help recoup some of the production costs involved. Currently, flax shive and hemp hurd residues are being sold on average at \$1/kg (with variations in price depending on individual producers) and the cost of transporting the residues ranges from \$28-\$34 per tonne. These prices are far higher than wood shavings and sawdust which are being sold for \$50/bone dry ton (approximately \$0.1/kg for dry wood residue) and transported at approximately \$25 per tonne.

The second issue is to do with the supply and storage of the flax shive and hemp hurd residues. Flax and hemp are both annual plants with life cycles of 90-125 days. Therefore, residue supply is confined to specific periods of the year and adequate quantities must be stored to sustain particleboard production in the off-seasons. The long-term large storage facilities are another significant cost related to the use of these residues for particleboard manufacture.

Lastly, another issue associated with the use of flax shive and hemp hurd residues for particleboard is the type of resin used for bonding and its cost. As mentioned earlier, flax and shive residues like most agricultural residues can efficiently be bonded with pMDI resin, a more expensive resin compared to the traditional UF resin which is commonly used with wood residues for particleboard. The current market price for pMDI is between \$1.8-\$2.2/kg and that of liquid UF resin is cheaper at \$0.9-\$1.1/kg.

Table 3.11 estimates the cost of production of a 4 foot by 8 foot particleboard from wood and flax shive/hemp hurd based on the following assumptions:

1. particleboard density 620 kg/m³
2. thickness 12.7 mm
3. 8% UF resin (65% solids content) at \$0.9/kg for wood
4. 2.5% pMDI resin (100% solids content) at \$1.8/kg for flax shive/hemp hurd residues.
5. \$25/tonne for transporting wood and \$30/tonne for flax shive/hemp hurd
6. \$1/kg for flax shive/hemp hurd residues and \$0.05/kg for wood residue

Since the manufacturing process involved in producing particleboard from flax shive/hemp hurd remains the same as that for wood, the labor cost, electricity, overhead and maintenance costs are essential equal. Hence, the major production costs incurred from using the flax shive/hemp hurd residues (evident from Table 3.11) are from the high cost of the residue and its transportation. Therefore, the cost of producing a 12.7 mm (0.5 inch) flax shive/hemp hurd particleboard of density 650 kg/m³ using 2.5% pMDI resin is more than 5 times the cost of producing the same particleboard from wood residues using 8% UF resin.

Table 3.11: Estimated production costs for a 4 foot by 8 foot particleboard panel.

	wood	flax shive/hemp hurd
residue	\$1.21	\$24.11
transportation	\$0.60	\$0.72
resin	\$2.57	\$1.04
Total	\$4.38	\$25.87

Based on this one will say that the cost savings for using flax shive or hemp hurd waste residues in place of wood residue disappear. It is essential to note that this argument is valid in regions where wood residue is cheap, readily available and accessible with no immediate competition for the resource. But in regions where the flax shive and hemp hurd residues are the most abundant lignocellulosic biomass, competitive pricing can drive the residue cost low. This will make the residues practical and cost-effective allowing their conversion to high value products which command premium prices per performance.

3.4 Conclusions

Generally, in the choice of raw material for furniture-grade particleboard production a balance needs to be struck in terms of residues that will provide adequate bond strength under compaction and high strength properties in bending. This chapter evaluated the feasibility of using hemp hurd and flax shive residues which are direct by-products of the hemp and flax stalk decortication process for particleboard production. The experimental work focused on the use of low quantities of pMDI resin — 2.5% and 5% — for the manufacture of 3-layered low density 620 kg/m³ particleboards.

Hemp hurd and flax shive residues were characteristically slender with lower bulk densities compared with wood residues. Their particle geometry positively influenced bending strength and stiffness properties and the lower bulk densities resulted in a slower rate of heat transfer to the particleboard mat core. Dynamic scanning calorimetry of the pMDI resin and wood, hemp hurd and flax shive residues indicated significant differences in the way the residues affected resin cure. In terms of the onset temperature and rate of curing, the pMDI resin was identified to have a greater initial reactivity in the presence of the wood residues. However, at the end of the curing process the heat of reaction was lowest in the hemp hurd samples signifying a low degree of pMDI cure. Of the 3 lignocellulosic residues tested, the hemp hurd had a substantial prohibitive effect on the curing reaction of pMDI resin. Surprisingly a greater number of chemical bonds were formed between the pMDI resin molecules in the presence of the flax shive residues compared to the wood residues, indicative of greater pMDI resin crosslinking in the flax shive samples. This degree of bonding was however not reflected in the internal bond strength of the particleboards manufactured and was attributed possibly to the effect of the differences in chemical and anatomical characteristics of the residues on the pMDI cure. This is therefore an area for further studies.

The vertical density profiles conducted on the 3 particleboard types showed the hemp hurd particleboards consistently having higher face densities, the result of their thick cell wall structure which required higher compaction to produce particleboards of a specified thickness. This in turn translated into greater flexure properties. Cohesion tests on hemp hurd and flax shive particleboards made with a low pMDI resin load of 2.5% revealed bond strengths comparable to those of wood particleboards with values that surpassed the minimum requirements of ANSI for

medium density M2 grade particleboards by as much as 92% and 35% respectively. Hemp hurd and flax shive particleboards manufactured with both 2.5% and 5% pMDI resin loads produced panels approximately 60% greater in bending strength and stiffness compared to their wood counterparts. Particleboards made with the 2.5% resin load exceeded the bending strength and stiffness requirements of the ANSI standards for low density (LD2) grade particleboard. While boards with 5% resin load exceeded the ANSI requirements for medium density (M2) grade particleboard.

Owing to their absorbent nature the flax shive and hemp hurd residues had an adverse effect on the particleboard thickness swell properties for both the 2.5% and 5% resin load boards, significantly so for boards manufactured with less resin. The narrow needle like nature of the flax shive and rectangular sections of the hemp hurd residues resulted in less linear changes with moisture variations between 50% and 90% relative humidity (at 20 ± 3 °C), which was likely due the residues consisting of a greater percentage of longitudinal sections.

These findings indicate that using lower isocyanate quantities of 2.5% (mass resin load) the flax shive and hemp hurd residues are capable of substituting wood residues based on performance, especially in the production of thinner (9.5-12.7 mm) low density grade particleboards for use in applications that have higher strength and longer span requirements. The performance enhancing characteristics of the hemp hurd and flax shive boards will add value to standard particleboard products and help expand them into new markets. Unfortunately, in Canada compared to wood residues, the current cost of the flax shive/hemp hurd residues and its transportation overtakes the cost savings element of using the residues as alternate biomass and quickly eliminates any profit potential.

4. Formaldehyde free acrylic-based resin—a binder for non-wood residue particleboards¹

The experimental work presented in this chapter assesses the performance of a new formaldehyde free acrylic-based resin, Acrodur[®] 950L, for particleboard production and its ability to bond agricultural crops/plant residues to produce board properties comparable to those of wood particleboards. The chapter provides information on the interest and general shift to non-formaldehyde emitting resins, details of the experimental design, and a comprehensive presentation and discussion of mechanical and physical strength properties obtained from short-term property tests of the manufactured particleboards. The chapter ends with a summary of the findings and its bearing on the use of Acrodur[®] 950L for particleboard manufacture from non-wood residues.

4.1 Background

The wood industry, specifically the panel products sector customarily uses amino and phenolic-based thermosetting resins such as urea formaldehyde (UF), melamine formaldehyde (MF) and phenol formaldehyde (PF) or a combination of these in the manufacture of particleboard, medium density fiberboard and oriented strand board (Dunky, 2003). The drawback of these resins from an environmental standpoint is their formaldehyde emissions. Lately with the increased demand and competition for residues for the panel products sector and others such as the biofuel and pulp industries, there has been growing research to identify and utilize other lignocellulosic biomass. Several agricultural crop and plant residues have been considered as raw material for panel products (Kozlowski and Helwig, 1998), with particleboard being the most investigated composite. Unfortunately, because of the waxy surfaces of these agricultural residues, UF resin which is primarily used for particleboard manufacture because of its low cost (approximately \$0.9-\$1.1/kg), low curing temperature and colorless glue line is unable to penetrate the residues to

¹ A version of this chapter will be submitted for publication

form mechanical interlocks or adequate bonds resulting in boards with poor bond strength (Markessini et al. 1997; Gerardi et al. 1998; Han et al. 1998).

Additionally, in 2007 the California Air Resources Board (CARB) instituted the Airborne Toxic Control Measure (ATCM) legislature to reduce formaldehyde emissions by specifying allowable formaldehyde emissions from wood composites such as particleboard, medium density fiberboard and hardwood plywood. This regulation coupled with growing interest in green buildings is encouraging most adhesive and composite manufacturers to make a shift to durable and environmentally friendly resins with low or no formaldehyde emissions.

In recent years, BASF, a chemical company, has developed a novel water-based formaldehyde free acrylic resin, Acrodur[®] 950L, as an alternative to the aminoplastic (UF and MF) and phenolic-based resins (PF). This resin has gained popularity in the automotive and furniture industries where it's currently being used in combination with natural fibers such as flax, hemp, kenaf, coir and sisal for various reinforcement applications because of its excellent bonding properties (BASF, 2015). Acrodur[®] 950L which on average costs approximately \$1.85-\$2/kg is a one-component resin based on the thermal crosslinking of polycarboxylic acids with a multi-functional alcohol, which produces water as a by-product of the curing process.

This study evaluates the physical and mechanical strength properties of low density particleboards (density < 640 kg/m³) manufactured from wood, flax shive and hemp hurd residues using Acrodur[®] 950L a formaldehyde-free resin, and compares it with the reported properties of other particleboards manufactured with conventional resins.

4.2 Experimental design

Using a low resin load of 7% Acrodur[®] 950L resin (oven dry weight basis), 3-layered particleboards were manufactured from hemp hurd, flax shive and wood residues as described in section 2.2.3. The approximate moisture content (MC) for the wood, hemp hurd and flax shive residues before board manufacture was 10 %. Crosslinking of the Acrodur[®] 950L resin is reported to occur in less than 2 minutes at a temperature range of 160-180 °C (Khalfallah et al. 2014). During the hot-pressing operation, the lowest temperature always occurs in the core of the mat. As

such to ensure the mat core temperature adequately reaches above 160 °C and allow complete crosslinking of the resin in the core, the press platen temperature was set to 190 °C after several board trials and the cooking period of the press cycle presented in section 2.2.3 extended to 565 s. A detailed experimental design for the study is provided in Table 4.1. Five board replicates were manufactured per each residue type for a total of 15 particleboards.

Table 4.1: Design of experiment for 3-layered particleboards using Acrodur® 950L resin.

Factors		Levels	Response	Total specimens
Variables	Residue type	wood	MOR/MOE	20
		hemp hurd	IB	32
		flax shive	TS	16
Constants	Density (kg/m ³)	620	LE	14
	Thickness (mm)	12.7		
	Replicates	5		

4.3 Results and discussion

4.3.1 Moisture content, average board density and vertical density profile

The MC and average board densities of the hemp hurd, flax shive and wood particleboards presented in Table 4.2 were calculated in a similar manner to that described in Chapter 3, section 3.3.3. Both MC ($p < 0.0002$) and average board density ($p < 0.0001$) yielded significant differences between all 3 particleboard types (Appendix C). The flax shive and hemp hurd particleboards yielded significantly high MC values in comparison to the wood boards. This was because the Acrodur® 950L resin was diluted with water to allow for easy atomization during the blending process, and the flax shive and hemp hurd residues with high absorption capacities absorbed more moisture in the process. The difference in average board density was solely due to the relatively higher wood particleboard density of 597 kg/m³ compared to the flax shive (577 kg/m³) and hemp hurd (569 kg/m³) boards which were not significantly different from each other.

Table 4.2: Average board density and moisture content of Acrodur® 950L bonded particleboards. n=20 for average board density means and n=16 for moisture content means. Values in parenthesis are standard deviations.

	wood	hemp hurd	flax shive
Density (kg/m ³)	597 (24.79)	569 (14.55)	577 (21.73)
MC (%)	10.81 (0.33)	11.49 (0.57)	12.41 (0.90)

The density profiles through the particleboard thickness are presented in Figure 4.1 as face (FI, F2) and core (C) densities. The face densities for the hemp hurd particleboards were significantly different ($p=0.0021$) from those of the wood and flax shive particleboards. The core densities ranging between 476-502 kg/m³ showed no significant differences. The density profile observed here for the different board types follows a similar trend to that detected in Chapter 3 (section 3.3.3) where the same residues were bonded with 2.5% and 5% polymeric diphenyl methane diisocyanate (pMDI) resin. In comparison to particleboards manufactured with 2.5% pMDI resin load (section 3.3.3), the pronounced density gradient observed here between the face and core layers for all 3 particleboard types was the result of the higher press temperature and moisture content of the residues used during the pressing operation which caused quick plasticization and greater densification of the faces prior to compaction of the core. The hemp hurd boards yielded the highest face density (853 kg/m³) and lowest core density (476kg/m³) and the wood boards with 759 kg/m³ and 493 kg/m³ yielded the next highest face and core densities respectively. In contrast with the 2.5% pMDI boards, the flax shive boards yielded the lowest face density at 731 kg/m³ but in a like manner attained the highest core density (502 kg/m³) of the 3 particleboard types.

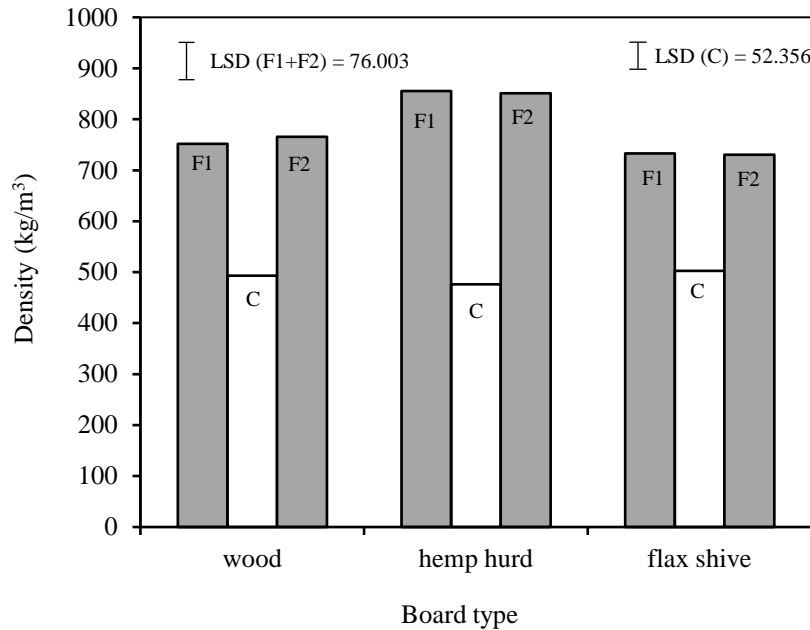


Figure 4.1: Density profile of particleboards; peak face (F1, F2) and core densities (C). $n=6$ for each mean. Error bars represent least significant difference (LSD) between means.

The large face and core density variations observed in Figure 4.1 and Table 4.3 cannot be solely attributed to the differences in board MC observed in Table 4.2. For if the VDP were only dependent on the MC differences then the flax shive particleboards with over 12% MC will be expected to have the greatest variation in density gradient with high face densities and low core density, which is not the case as seen in Table 4.3. These results further confirm the conclusions arrived at in Chapter 3 (section 3.3.3) that the cell structure of the residues affected its ability to resist compression during hot pressing. The density gradient observed is likely to influence the internal bond properties of the particleboards.

Table 4.3: Face and core layer density variations for Acrodur® 950L particleboards.

Board Type	wood	hemp hurd	flax shive
average board density	597	569	577
face/ board density	1.27	1.50	1.27
core/ board density	0.83	0.84	0.87
face: core variation	0.44	0.66	0.40

4.3.2 DSC analysis

Unlike the DSC runs for mixtures of pMDI resin with wood, hemp hurd, and flax shive residues where distilled water was added to enhance resin activation and the observation of distinct exothermic peaks, the DSC samples for the Acrodur® 950L and residue mixtures did not include water, the resin was used as received in combination with oven dry residues. DSC scans were also conducted in a similar manner with UF resin in combination with oven dry wood, hemp hurd and flax shive residues to allow for a comparison with the acrylic-based Acrodur® 950L resin.

4.3.2.1 Acrodur® 950L and UF resins

DSC scans of the neat Acrodur® 950L and UF resins are shown in Figures 4.2 and 4.3. The Acrodur® 950L resin undergoes a gradual endothermic glass transition or melting phase observed over the whole heating range of 50 °C to 200 °C (Figure 4.2). The endotherm observed cannot be attributed to vapor loss since the analysis was conducted in stainless steel high volume pans which prevent volatilization of materials.

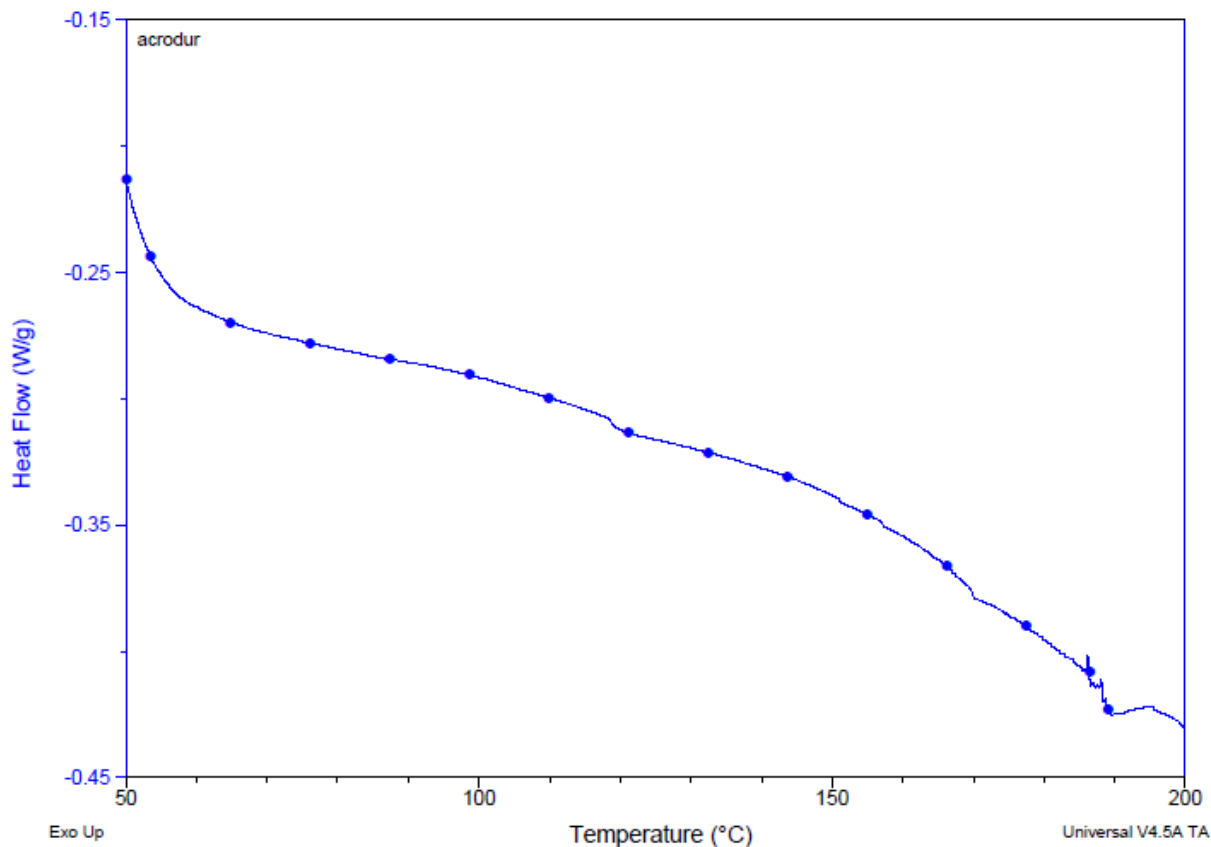


Figure 4.2: Dynamic scan of Acrodur[®] 950L resin.

In contrast, the UF resin undergoes an exothermic reaction between 90 °C and 160 °C and an endotherm after 165 °C (Figure 4.3). The first well defined exothermic peak represents the crosslinking of the UF resin while the second less prominent endotherm likely represents hydrolysis reaction of the UF resin also observed by Xing et al. 2005 while studying the curing behavior of UF resin as affected by various catalyst content. No catalyst was added to the UF resin in this research.

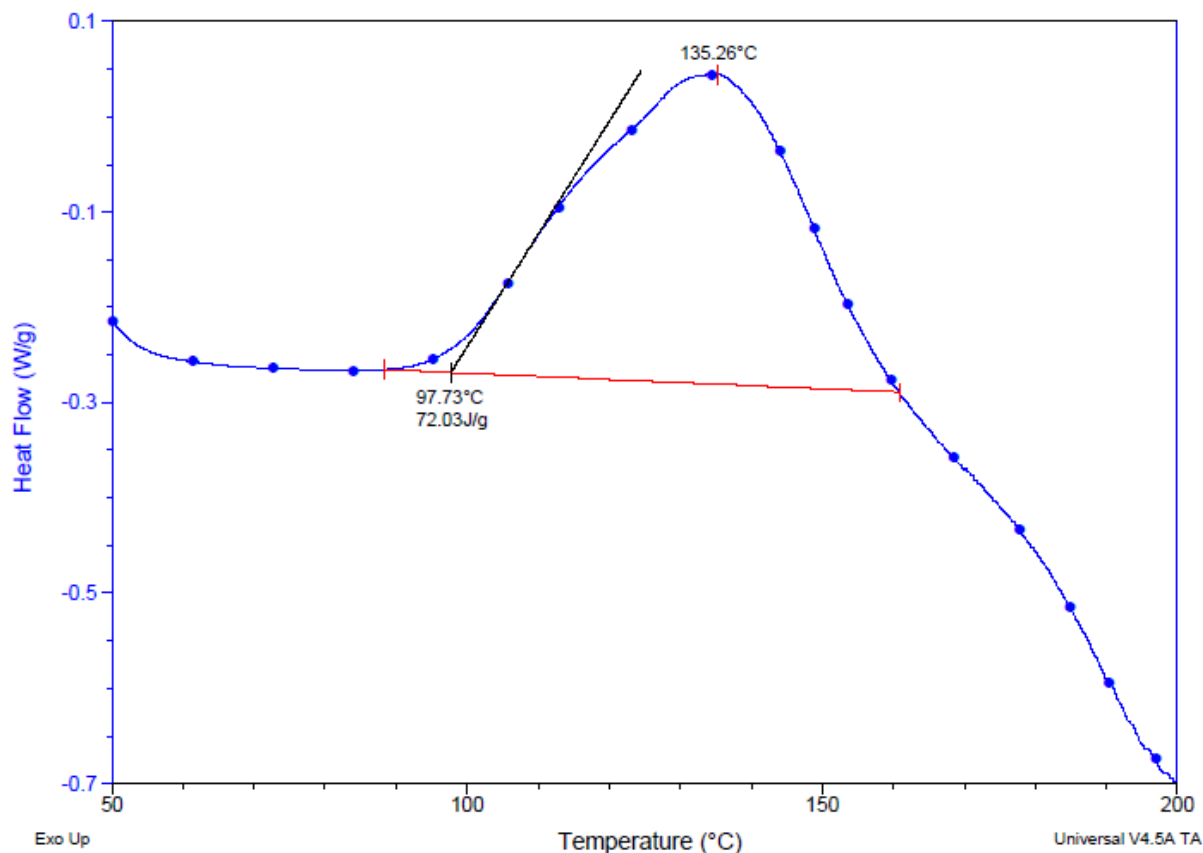


Figure 4.3: Dynamic scan of urea formaldehyde resin (Casco-Resin™).

4.3.2.2 Acrodur® 950L/ UF resin and lignocellulosic residue mixtures

A combined graph of the DSC results for mixtures of the different residues with Acrodur® 950L and UF resins is presented in Figures 4.4 and 4.5. A list of the average onset temperatures, peak temperatures and reaction heats extracted from the graphs are given in Tables 4.4 and 4.5.

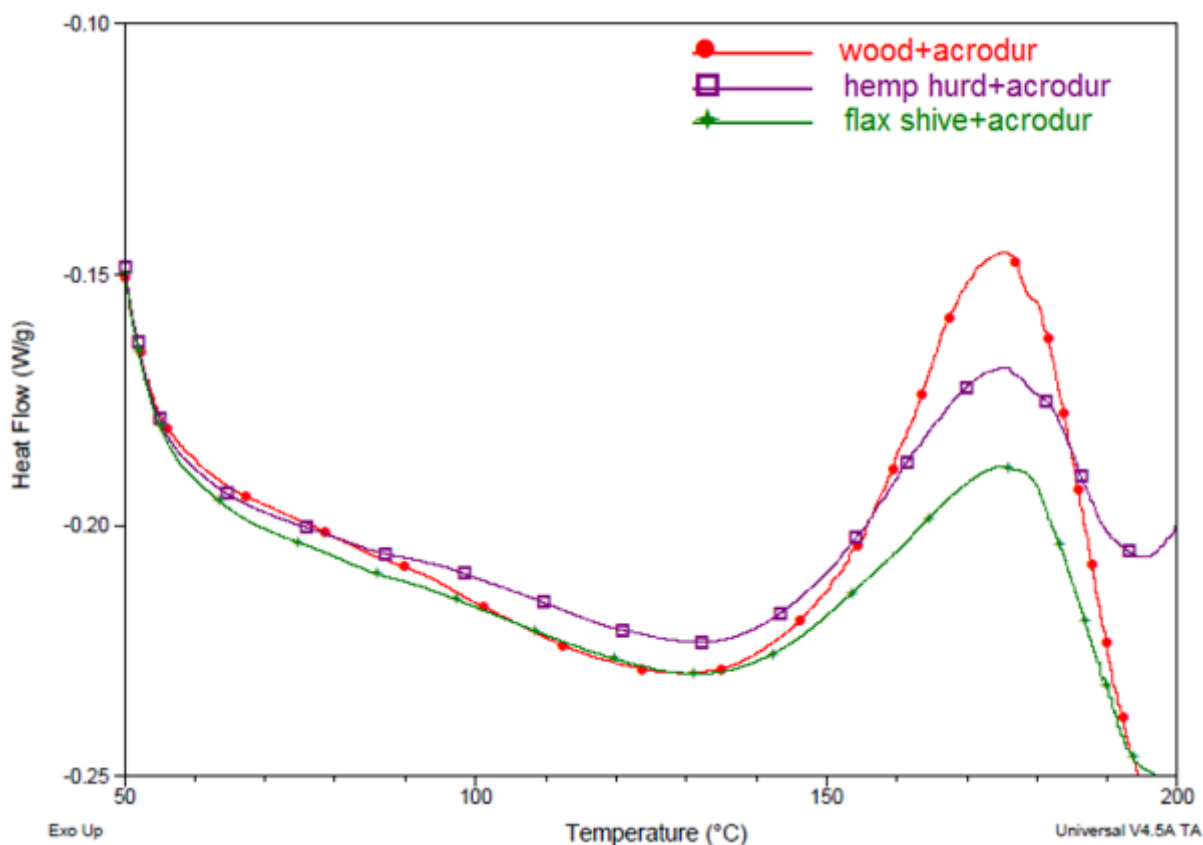


Figure 4.4: DSC curves of curing reaction between wood, hemp hurd, flax shive residues and Acrodur® 950L resin.

Table 4.4: Thermal properties of curing reaction between Acrodur® 950L resin and residues.

Sample	Onset Temp. (°C)	Peak Temp. (°C)	Reaction heat (J/g)
wood+acrodur	149.8	176.5	18.3
hemp hurd+acrodur	147.8	175.5	8.3
flax shive+acrodur	141.1	175.8	10.0

From Figure 4.4 it appears that residues combined with the acrylic-based Acrodur® 950L resin undergo an endothermic reaction possibly a melting process between 60 °C and 120 °C before a single exothermic peak representing the curing reaction takes place at higher temperatures between 140 °C and 200 °C, in keeping with the crosslinking temperatures stated for Acrodur® 950L resin.

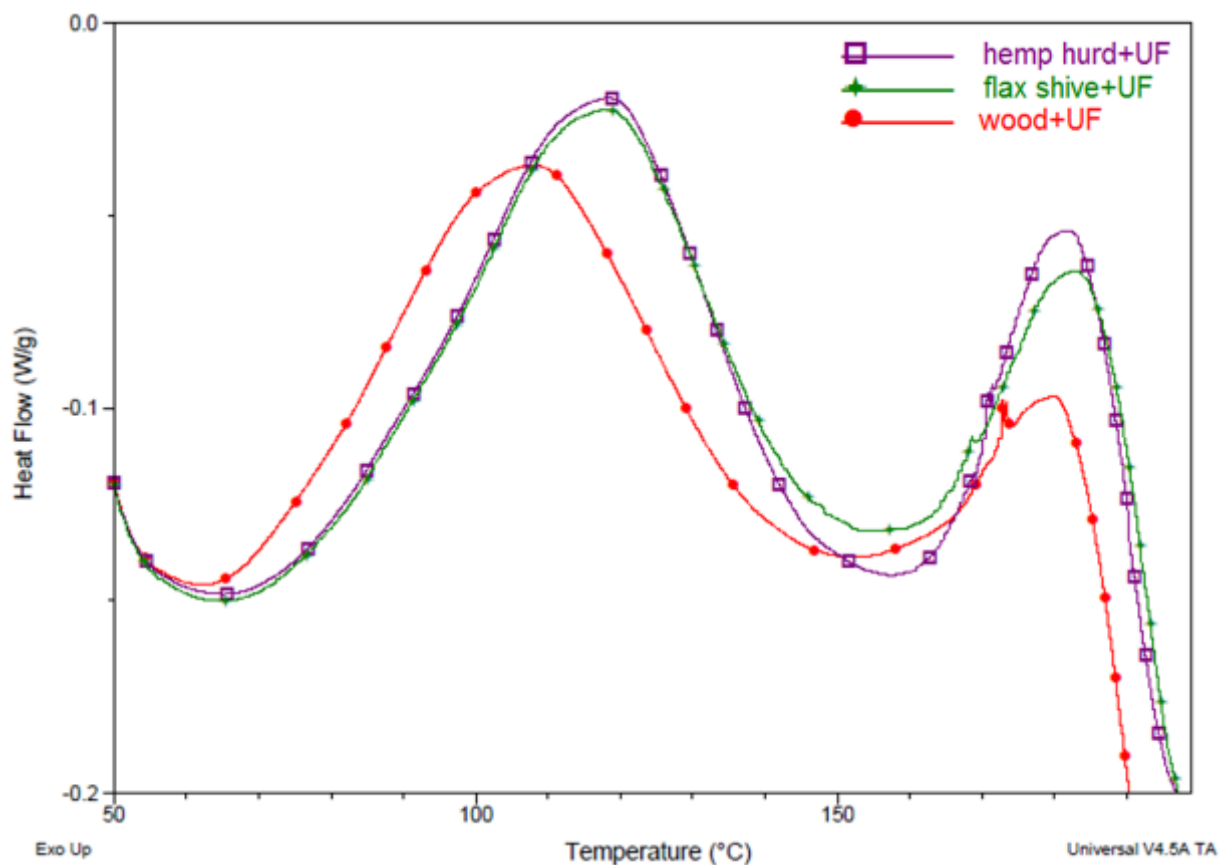


Figure 4.5: DSC curves of curing reaction between wood, hemp hurd, flax shive residues and urea formaldehyde (UF) resin.

Table 4.5: Thermal properties of curing reaction between urea formaldehyde (UF) resin and residues.

Sample	Onset Temp. (°C)	Peak Temp. (°C)	Reaction heat (J/g)
wood+UF	71.9	107.9	26.0
hemp hurd+UF	81.3	118.0	29.1
flax shive+UF	81.5	117.3	28.1

A look at the residues combined with UF resin (Figure 4.5) on the other hand displays two exothermic peaks, a first peak ranging from 70 °C to 150 °C representing the curing reaction of

the resin, followed by a second peak which was more pronounced with the hemp hurd and flax shive residues.

4.3.2.3 Effect of residue type on curing properties of resin

A comparison of the average onset temperatures for both the Acrodur[®] 950L and UF resins as seen in Figure 4.6 shows significantly ($p<0.0001$) early curing reaction for the UF resin at temperatures approximately 46% lower than that observed with the Acrodur[®] 950L resin (Appendix C). The start of the cure reaction for the Acrodur[®] 950L resin is slowest in the wood residues at 150 °C, followed by the hemp hurd at 148 °C and significantly ($p=0.0050$) faster in the flax shive at 141 °C. The inverse was observed with the UF resin where the onset of the curing reaction was significantly ($p<0.0001$) faster in the wood residue samples at 72 °C and slower in both the flax shive and hemp hurd residue samples at 81 °C (Appendix C).

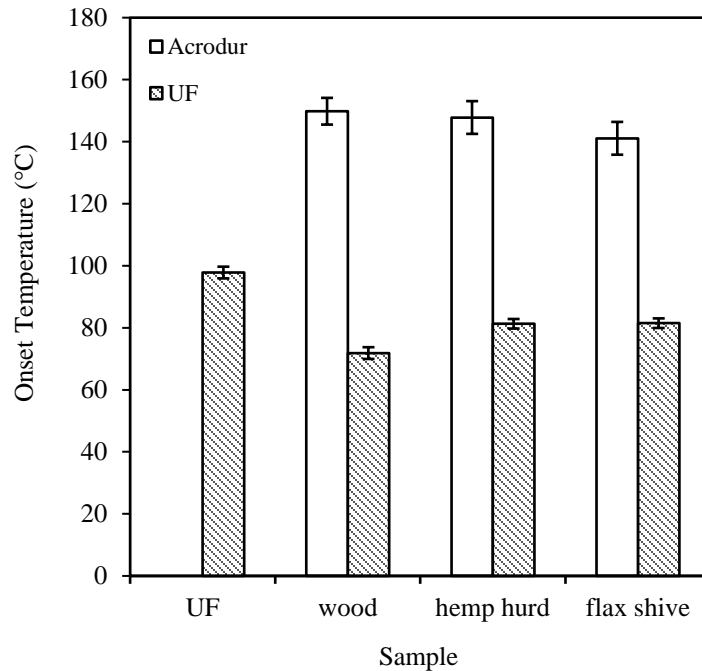


Figure 4.6: Onset temperature comparison between Acrodur[®] 950L and UF resin based on residue type. Error bars represent 95% confidence intervals.

Generally, the addition of the residues to the UF resin significantly ($p < 0.0001$) lowered the onset temperature of the resin cure by approximately 27% for the wood samples and 17% for both the hemp hurd and flax shive samples, indicating an accelerated curing rate (Appendix C). The significantly higher ($p < 0.0001$) temperature — approximately 10 °C — at which onset of curing begins for the UF and wood samples compared to the UF and hemp hurd or flax shive samples indicates the initial prohibitive effect of the non-wood residues on the UF curing process (Appendix C). The opposite was detected in the Acrodur® 950L resin combinations, where an earlier onset was observed for the curing reaction of the resin in the flax shive samples.

The peak temperatures for the curing reaction in the UF/ Acrodur® 950L resin and residue mixtures are shown in Figure 4.7. Once again the peak temperatures observed in the UF resin and residue mixtures were significantly lower ($p < 0.0001$) compared with the Acrodur® 950L resin and residue mixtures indicating a faster reaction rate for the UF resin in combination with residues (Appendix C). With the Acrodur® 950L resin there were no significant differences in peak temperatures between the wood (177 °C), hemp hurd (175 °C) or flax shive (176 °C) samples, demonstrating that irrespective of the initial onset temperature, the temperature at which the cure rate reached a maximum was similar for all the residues. This also indicates that the press platen during the pressing operation must be set to temperatures above 177 °C to guarantee temperatures in the core of the mat exceed this temperature and ensure effective bonding of the resin.

In contrast the peak temperatures observed for the UF and residue mixtures were significantly lower ($p < 0.0001$) than that for the neat UF resin indicative of a shorter reaction time with the addition of the residues (Appendix C). The UF and wood samples yielded significantly lower ($p < 0.0001$) temperatures (108 °C) compared with the UF in combination with the flax shive (117 °C) and hemp hurd (118 °C) samples (Appendix C); further confirming the greater reactivity of the UF resin in the presence of the wood residues.

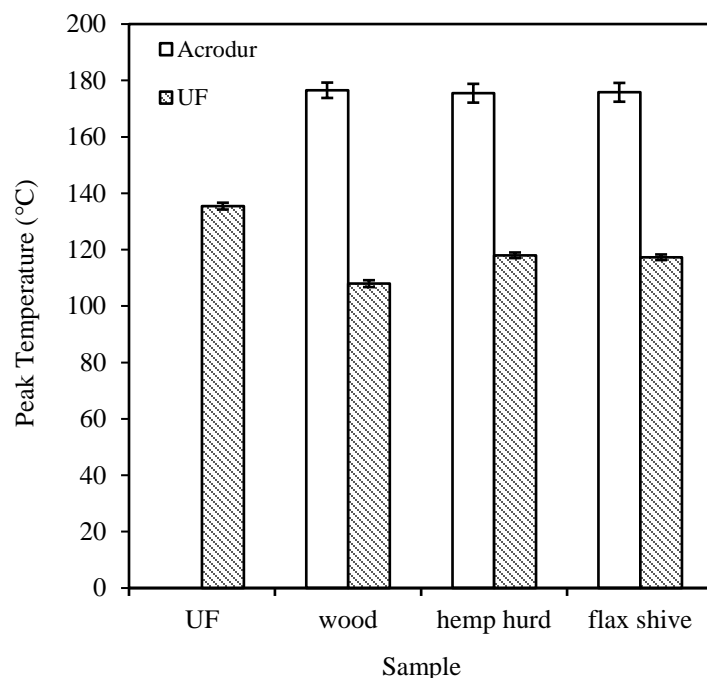


Figure 4.7: Peak temperature comparison between Acrodur® 950L and UF resins combined with different residue types. Error bars represent 95% confidence intervals.

As a thermoset resin cures heat is evolved (reaction heat). Higher reaction heat values are therefore indicative of more bonding between the resin molecules, i.e. resin crosslinking. The heat of reaction for both UF and Acrodur® 950L resins in combination with the wood, hemp hurd and flax shive residues are presented in Figure 4.8. A significantly higher ($p < 0.0001$) degree of cure was observed in the UF resin mixed with the residues in comparison to the Acrodur® 950L resin mixed with the same residues (Appendix C). The UF resin combined with the hemp hurd and flax shive samples attained the highest reaction heat at 29 J/g and 28 J/g respectively compared to the wood sample which was significantly different ($p = 0.0339$) at 26 J/g (Appendix C). These reaction heat values were significantly lower ($p < 0.0001$) compared to the neat UF resin reaction heat of 74 J/g suggesting less bonding between the UF resin molecules in the presence of the residues (Appendix C). For the Acrodur® 950L, the resin in combination with the wood samples at 18 J/g yielded significantly ($p = 0.0010$) higher reaction heat values of the 3 residue types—at least 40% greater than that for the hemp hurd (8 J/g) and flax shive (10 J/g) residues (Appendix C). These results

suggest more bonding at the end of the curing process between the Acrodur® 950L resin molecules in the presence of the wood residues in contrast to the flax shive and hemp hurd residues.

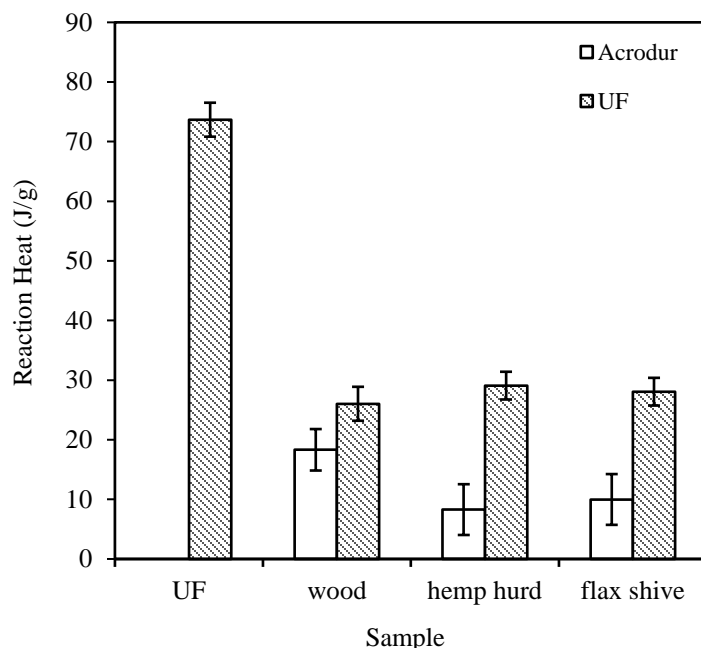


Figure 4.8: Reaction heat comparison between Acrodur® 950L and UF resin based on residue type. Error bars represent 95% confidence intervals.

Overall analysis of the DSC results demonstrates that in comparison to the UF resin the Acrodur® 950L resin has a slower onset of curing, slower rate of reaction and lower degree of cure when combined with wood, hemp hurd and flax shive residues. It is important to note that curing of UF resin is catalysed by acid, and the greater the acidity of the UF resin the higher the cure rate. Therefore, the pH and buffering capacity of the residues can affect the curing rate of the resin. The lower onset and peak temperatures observed for the UF resin in combination with the residues compared to the Acrodur® 950L resin could be the result of the acidity of the residues. Further analysis of the results indicates that despite the initial faster onset and reaction rates of the Acrodur® 950L resin in the presence of the flax shive and hemp hurd residues, at the end of the curing process the resin yielded a low degree of bonding. These results suggest that Acrodur®

950L resin may be unable to sufficiently bond flax shive and hemp hurd residues and thus may be prohibitive for particleboard production.

4.3.3 Mechanical properties

4.3.3.1 IB

The IB strength results obtained for the wood, hemp hurd and flax shive particleboards bonded with Acrodur[®] 950L resin are graphical presented in Figure 4.9. Within a density range of 550–670 kg/m³, the IB strength was significantly lower ($p < 0.0001$) for the flax shive boards compared to the wood and hemp hurd boards (Appendix C). The IB strength of all the particleboards exceeded the ANSI A208.1-1999 requirement of 0.15 MPa for low density (LD2) grade particleboard.

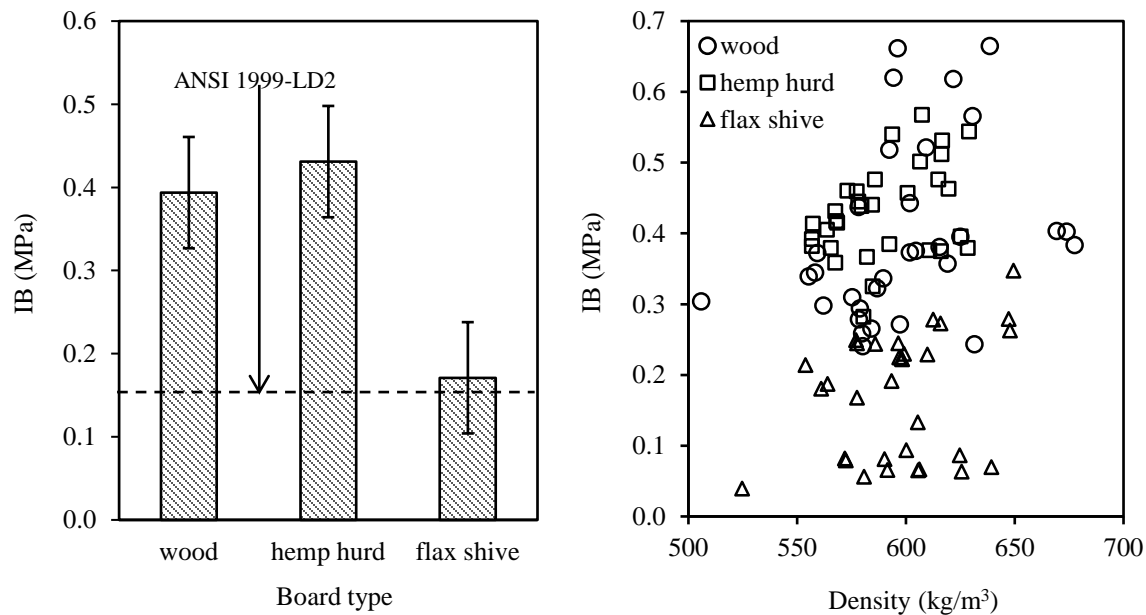


Figure 4.9: Internal bond strength of low density particleboards bonded with Acrodur[®] 950L resin. Horizontal line indicates minimum value stipulated by ANSI A208.1-1999 for M2 boards. $n=32$ for each mean. Error bars represent 95% confidence intervals.

Contrary to the DSC reaction heat results the hemp hurd particleboards bonded with Acrodur® 950L resin yielded the greatest internal bond strength compared to the wood and flax shive residues. The lower IB values observed in the flax shive particleboards can be attributed to a combination of the degree of cure of the Acrodur® 950L resin, the MC of the flax shive mats and the slower rate of heat transfer to the core of the mats as discussed in section 3.3.2. From the DSC results, in the presence of the flax shive residues the Acrodur® 950L resin yielded a low degree of cure which combined with the slower rate of heat transfer within the flax shive mats and higher mat MC during hot pressing resulted in poor bond strength at the end of the press cycle. Several samples for the flax shive particleboards were discarded because after numerous trials the core of the board was not completely bonded and fell apart.

Though the IB values observed for particleboards bonded with the Acrodur® 950L resin seem rather low it is important to note that the values obtained falls within the targeted low density (LD2) particleboard grade requirement. In 2003, Papadopoulos and Hague (2003) studied the feasibility of fabricating 17.5 mm single-layer particleboards from various proportions of wood and flax shive residues (approximately 10% MC) using 13% UF resin. They reported an average IB strength of 0.09 MPa for the pure flax shive particleboards of density 748 kg/m³. Despite their higher resin content and density- parameters that result in increased adhesion- the average IB strength of 0.17 MPa obtained for the flax shive particleboards in the current study are far greater.

Furthermore, studies conducted by Schöpfer et al. (2009) on light weight particleboards from hemp hurd residues using 10% UF resin reported IB values of 0.32 MPa and 0.57 MPa for the 450 kg/m³ and 550 kg/m³ particleboards respectively. With a mean IB strength of 0.43 MPa the hemp hurd particleboards in this study manufactured to 569 kg/m³ density with 7% Acrodur® 950L resin is lower compared to the 550 kg/m³ boards from Schöpfer et al. (2009) and can be related to the results observed in the DSC tests.

4.3.3.2 MOR and MOE

Significant differences ($p < 0.0001$) were observed between the wood, hemp hurd and flax shive particleboards in terms of their bending properties (MOR and MOE), with the hemp hurd and flax shive particleboards achieving the highest values (Figure 4.10, Table 4.6 and Appendix C). This

result is consistent with results obtained for the isocyanate-bonded particleboards discussed in section 3.3.5 (Chapter 3) where the hemp hurd and flax shive particles with high aspect ratios attained high bending properties. All the 3 particleboard types exceeded the minimum ANSI A208.1-1999 bending strength requirement of 5.0 MPa for low density (LD2) grade particleboard. The flax shive particleboard was the only board type of the 3 particleboards to meet the stiffness (MOE) requirements for medium density M2 grade particleboard.

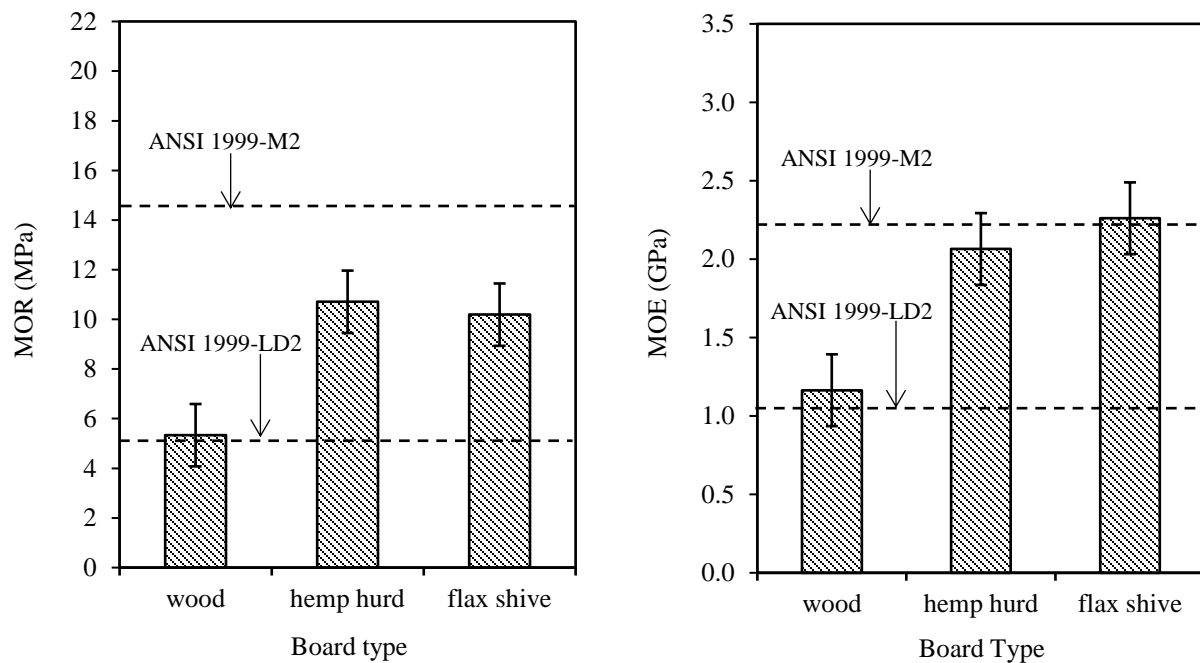


Figure 4.10: Bending strength and stiffness properties of particleboards manufactured with Acrodur[®] 950L resin. Horizontal line indicates minimum value stipulated by ANSI A208.1-1999 for LD2 and M2 grade particleboards. $n=20$ for each mean. Error bars represent 95% confidence intervals.

Papadopoulos and Hague's (2003) study on flax shive particleboards using a higher resin content of 13% UF and density 748 kg/m^3 , reports a higher MOR value of 11.72 MPa compared with a value of 10.19 MPa obtained for the flax shive boards in this study which were manufactured with a lower resin load of 7% Acrodur[®] 950L resin to a far lower density (577 kg/m^3).

Table 4.6: Bending strength and stiffness properties of Acrodur® 950L particleboards. Panel means are presented according to ANSI A208.1-1999. Values in parenthesis are standard deviations.

Board Type	MOR (MPa)	MOE (GPa)
wood	5.33 (1.30)	1.16 (0.23)
hemp hurd	10.71 (1.24)	2.06 (0.19)
flax shive	10.19 (1.63)	2.26 (0.33)
ANSI LD2	5.0	1.03
ANSI M2	14.5	2.25

In a study by Nikvash et al. 2010, 3-layered particleboards were produced from industrial wood chips as face material and hemp hurd residues in the core. Both wood and hemp hurd residues with MC of 3-4% were pressed at 200 °C to a target density of 700 kg/m³ and 20 mm thickness using 8% UF resin in the core and 10% resin in the face furnish. Results from the study yielded MOR and MOE values of approximately 16 MPa and 3.4 GPa respectively for the particleboards. The MOR and MOE results are greater than those obtained in this study (Table 4.6) for a pure hemp hurd particleboard bonded with 7% Acrodur® 950L resin (oven dry weight basis), and are the result of the differences in processing variables such as the residue types, MC, press temperature board thickness and density.

On the other hand, a comparison of the MOR and MOE results for the particleboards bonded with Acrodur® 950L and 2.5% pMDI resin load from Chapter 3 (section 3.35) revealed no significant differences in bending properties between the particleboard based on residue type (Figure 4.11). The analysis also indicated significantly ($p < 0.0001$) higher MOR and MOE values in both resin types for the hemp hurd and flax shive particleboards compared to the wood particleboards.

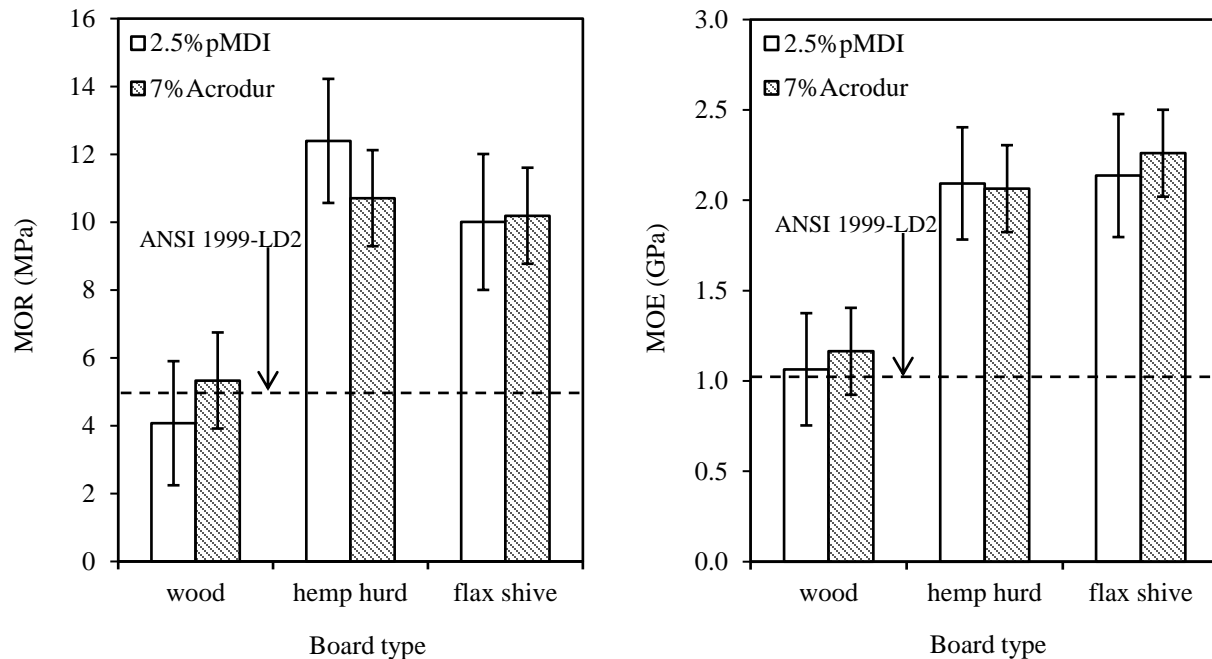


Figure 4.11: Comparison of bending strength and stiffness properties between 2.5% pMDI and 7% Acrodur® 950L particleboards. Horizontal line indicates minimum value stipulated by ANSI A208.1-1999 for LD2 grade particleboards. Error bars represent 95% confidence intervals.

4.3.4 Physical properties

4.3.4.1 LE

Figure 4.12 presents the linear expansion (LE) values between 50% and 90% relative humidity at 20 ± 3 °C for the wood, hemp hurd and flax shive particleboards. The values presented are the pooled effect of samples parallel and perpendicular to the forming direction after analysis revealed no significant differences between them. Compared to the hemp hurd and flax shive particleboards, the wood particleboards yielded significantly higher ($p < 0.0001$) LE values which exceeded the specified ANSI A208.1-2009 upper 95th percentile value of 0.40% (Appendix C). These results are in line with those obtained for the wood, hemp hurd and flax shive particleboards manufactured with 5% pMDI resin load (Figure 3.12) where LE values of 0.33% were observed in the hemp hurd particleboards, and 0.31% and 0.24% in flax shive particleboards parallel and perpendicular to the forming direction correspondingly. The differences in particle length as discussed earlier in

section 3.3.6 is responsible for the variations in LE observed here once again for the same residue types.

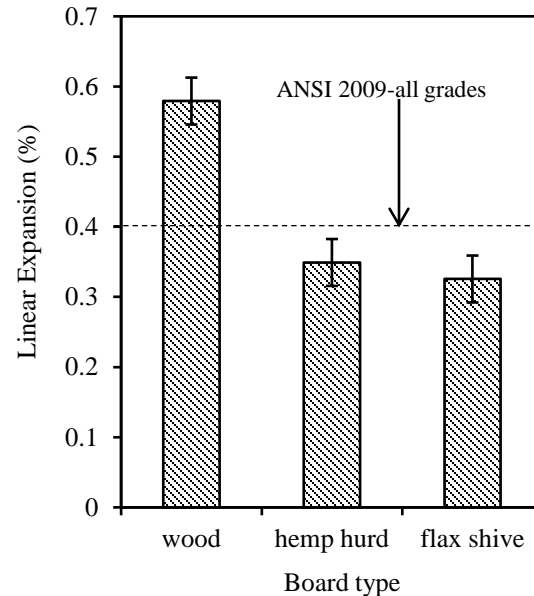


Figure 4.12: Linear expansion with changes in moisture content between 50% and 90% relative humidity. Horizontal line indicates maximum value stipulated by ANSI A208.1-2009. $n=14$ for each mean. Error bars represent 95% confidence intervals.

4.3.4.2 TS and WA

The thickness swell (TS) and water absorption (WA) properties of the wood, hemp hurd and flax shive particleboards are compared in Figure 4.13. The absorbent nature of the hemp hurd and flax shive residues (Hague 1998) are evident in the significantly higher ($p<0.0001$) thickness swell values observed for both the 2 and 24 hour submersion results (Appendix C). Surprisingly, analysis of the short term (2 hour) water absorption characteristics revealed no statistical differences between all 3 particleboard types; significant differences ($p<0.0001$) were only observed after 24 hours when the wood particleboards absorbed less water in comparison to the hemp hurd and flax shive particleboards (Appendix C).

Twenty-four hours TS values of 17% and 21% was reported by Schöpfer et al. (2009) for 450 kg/m³ and 550 kg/m³ hemp hurd particleboards bonded with 10% UF resin respectively. For the

same period the hemp hurd boards herein bonded with 7% Acrodur® 950L to a density of 569 kg/m³ yielded a relatively higher TS value of 26%, with the difference attributed to the higher resin content and internal bond of the particleboards from Schöpfer et al. (2009) study which allowed for greater adhesion between the particles in the board.

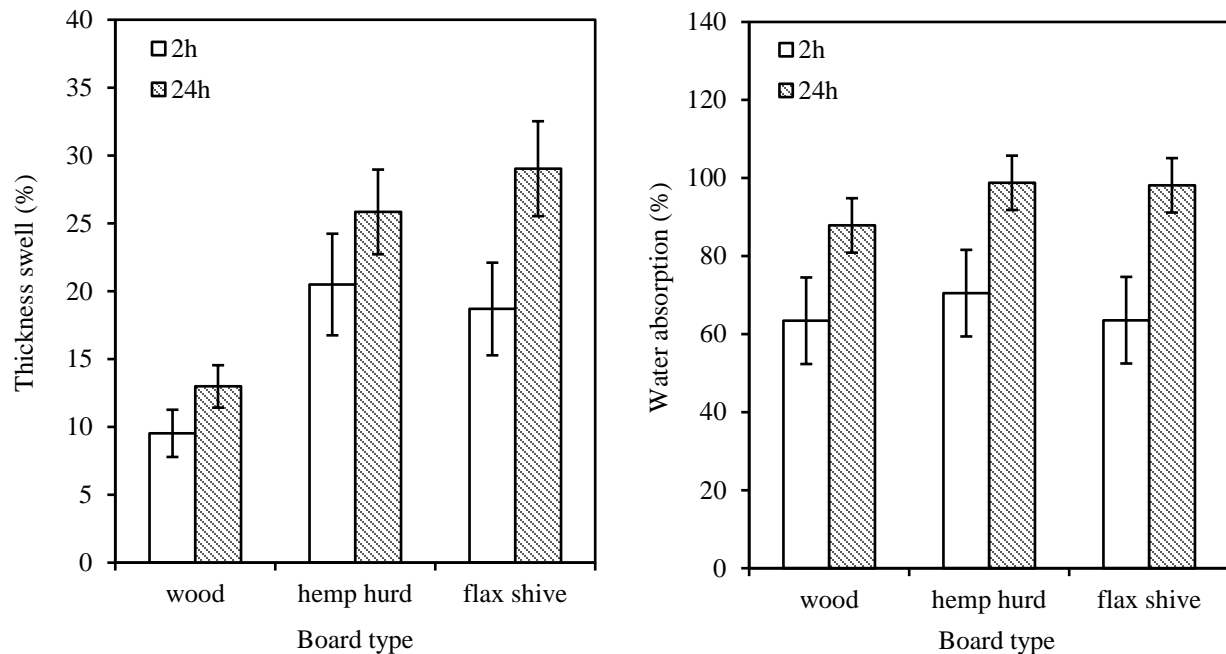


Figure 4.13: Thickness swell and water absorption properties of Acrodur® 950L particleboards; 2h (2 hours) and 24h (24 hours). $n=16$ for each mean. Error bars represent 95% confidence intervals.

The 24 hours TS value of 29% observed for the flax shive particleboards (577 kg/m³) are less than the 63% value reported by Papadopoulos and Hague (2003) for similar flax shive particleboards of a higher density 748 kg/m³. The difference in TS values is because of the differences in board density. Increasing board density correlates to increases in TS because denser boards tend to have higher residual compressive stresses set in the boards which are rapidly released once it interacts with water/moisture. Also, particleboards manufactured to a higher target density of 700 kg/m³ from a combination of wood and hemp hurd residues by Nikvash et al. (2010) using 8% and 10% UF resin yielded approximately 29% TS after 24 hours submersion, in comparison the pure hemp hurd particleboards in this study yielded a lower value of 26% TS.

The trend observed with particleboards bonded with Acrodur® 950L where higher TS values were observed in the hemp hurd and flax shive particleboards compared to the wood particleboards are in line with general observations that were made in other studies regarding the greater water absorption characteristics of the hemp hurd and flax shive residues when bonded with UF resin (Hague, 1998; Papadopoulos and Hague 2003; Schöpfer et al. 2009; Reh 2013).

A general comparison of the physical and mechanical strength properties of particleboards manufactured from other agricultural crop and plant residues using UF resin is given in Table 4.7. The information provided in the table gives an idea of the performance of the Acrodur®-based flax shive and hemp hurd particleboards against other residues manufactured with UF resin. A look at the IB values in contrast to the resin load, board densities and thicknesses listed suggests that foregoing other board parameters, Acrodur® 950L may be able to sufficiently bond other non-wood residues.

Table 4.7: Properties comparison of particleboards manufactured from non-wood residues.

Residue type	Thickness (mm)/layer	Density (kg/m ³)	Resin (load %)	MOE (GPa)	MOR (MPa)	IB (MPa)	TS-24h (%)
flax shive	12.7/3	577	Acrodur (7)	2.26	10.19	0.17	29
hemp hurd	12.7/3	569	Acrodur (7)	2.06	10.71	0.43	25.8
sunflower stalks Khrstova et al.1996	16/1	509-611	UF (12)	—	5.6-10.28	0.14-0.16	15.26-17.6
Date palm branches Nemli et al. 2001	20/3	650	UF (7-11)	—	14.16-18.94	0.35-0.83	7-18
wheat straw Mo et al. 2003	6.5/1	700	U (8)	1.8	6.36	0.11	63.9
cotton stalks Guler&Ozen 2004	20/3	400-700	UF (6-12)	—	3.31-17.95	0.11-0.59	18-35
kenaf stalks Kalaycioglu&Nemli 2006	20/3	600	UF (8-10)	—	13.99-16.34	0.36-0.43	10.16-19.1
tomato stalk Guntekin et al.2009	16/3	530-780	UF (8-12)	1.33-3.1	5.4-12.13	0.3-0.56	70-95
poppy husk Kessin et al.2015	18/3	680	UF (8-10)	0.58	3.24	0.21	22.48

4.4 Conclusions

Mostly non-wood residues obtained from plants or agricultural crops are hydrophilic and highly polar in nature due to the hydroxyl groups in the cellulose and hemicellulose which interacts with water through absorption or adsorption when exposed to moisture saturated environments (Abdul Khalil et al. 2000; Thomas et al. 2011). The presence of waxy substances on the residue surfaces however does not allow most polar resins to penetrate it and form mechanical interlocks or hydrogen bonds with the above-mentioned hydroxyl groups in the plants or agricultural crop cell wall. Hence a combination of the residues and conventional resins such as UF usually results in poor adhesive bonds.

The study presented in this chapter evaluated a water-based acrylic resin, Acrodur[®] 950L, which has no emissions of formaldehyde as a resin option capable of efficiently bonding non-wood residues to produce particleboards of comparable performance to conventional UF resins.

Thermal analysis demonstrated that the UF resin in the presence of the wood, hemp hurd and flax shive residues has an early onset of curing and faster reaction rates that were approximately 46% and 33% faster than that in the Acrodur[®] 950L resin respectively. Similarly, at the end of the curing process the UF resin yielded the highest reaction heat values (at least 30% higher) consistent with a higher bond formation between the resin molecules. Overall, the Acrodur[®] 950L resin demonstrated a low reactivity in the presence of the hemp hurd and flax shive residues.

Results of the short-term property testing of particleboards manufactured with 7% Acrodur[®] 950L resin revealed the internal bond strength of the low density (569-597 kg/m³) wood, hemp hurd and flax shive particleboards all exceeded the minimum values stipulated by ANSI A208.1-1999 for low density LD2 grade particleboard. The lowest bond strength was observed in the flax shive particleboards and was attributed to a combination of the low degree of Acrodur[®] 950L resin cure, the higher moisture content of the residue at the time of board manufacture and low heat transfer to the mat core which in turn caused insufficient cure of the resin in the core. All 3 particleboard types yielded MOR and MOE values that met the minimum low density LD2 requirements by ANSI A208.1-1999. The highest MOR and MOE values were continually observed in the flax shive and hemp hurd particleboards because of the residues greater aspect ratios which accorded them more overlap in bonding and bending. A comparison of the bending strength and stiffness

results with that of similar residues manufactured with 2.5% pMDI showed no significant differences.

The linear expansion values observed between 50% to 90% relative humidity at 20 ± 3 °C for the hemp hurd and flax shive particleboards bonded with Acrodur® 950L were within the range stipulated by ANSI A208.1-2009 and were comparable to similar particleboards fabricated with 5% pMDI resin. Over 2 and 24 hours' period the thickness swell values for the hemp hurd and flax shive particleboards were significantly higher than that of the wood particleboard. Similar results have been reported in other studies concerning the negative effect of the hemp hurd and flax shive residues on thickness swell and water absorption characteristics of particleboard because of their affinity for moisture compared with wood residues.

The results of this study suggest that based on the thermal properties observed in the DSC tests, Acrodur® 950L resin cannot compete with UF resin for bonding wood in particleboard production, and neither is it able to sufficiently bond hemp hurd and flax shive residues. Yet based on performance particleboards bonded with Acrodur® 950L resin produced boards with acceptable properties that met ANSI low density LD2 grade requirements. Therefore, in drawing conclusions about the suitability of Acrodur® 950L for the manufacture of particleboard from wood and non-wood residues, caution must be used as it is unclear whether under similar production conditions and with careful regulation of processing parameters such as moisture content, particle size, resin load, board density, board thickness, press temperature and time, Acrodur® 950L resin can produce particleboards with properties comparable to those manufactured from conventional resins with the added benefit of no formaldehyde emissions. This therefore merits additional investigation.

5. Lignin-based resin for the manufacture of wood and non-wood particleboards

The research presented in this chapter examines the potential of substituting lignin (a cheaper naturally occurring polymer) for a portion of pMDI resin in particleboard production to reduce resin costs. The sections that follow provide background information about substituting percentages of synthetic resins with natural alternatives and the structure of the experimental work carried out in this study. The chapter also contains information on the complete assessment and analysis of short-term properties for the resulting lignin-based particleboards. The concluding section outlines the major outcomes of the study and proposes possible future uses of pMDI-lignin resins.

5.1 Background

Traditionally most of the resins used in the wood products sector are petroleum-based and quite expensive. Several approaches have been researched over the years to identify means of reducing the cost of these resins by substituting part of the resin with less expensive polymers, mostly naturally abundant polymers. One of the methods that has been extensively researched is the use of lignin—a cheaper bio-based thermoset that is abundant in nature—to substitute phenol in phenol formaldehyde (PF) production (Vazquez et al. 1997; Park et al. 2008; Wang et al. 2009; Gothwal et al. 2010; Bertaud et al. 2012). Lignin is a large 3-dimensional polymer found in the cell walls of plants. It is a phenolic material with a complex structure and is commonly available as a by-product of the chemical pulping process. Depending on the plant species from which they are obtained and the extraction procedure used lignin has a variety of functional groups (Calvo-Flores and Dobado 2010).

Lignin has been investigated as a promising alternative because of its structure as a polyphenol with a highly cross-linked network like that of PF resins (Cetin and Ozmen 2002a; Pizzi 2003). However, it is not as reactive an adhesive compared to PF. This is because it has a lower number of reactive sites on its complex chemical structure which has yet to be fully understood. There is

also the issue of chemical variation in the raw material supply depending on the origin and extraction method.

Different procedures have been developed over the years to utilize pure lignin as an adhesive for particleboard production. These include the Pedersen procedure, Shen system and the Nimz system (Pizzi 2003). The Pedersen procedure involved mixing wood chips with technical spent sulfite liquor (pH ~ 3) and pressing boards for 30 minutes at a high temperature (~ 185 °C). The boards were then further heat treated in an autoclave for almost an hour and half at 195 °C, resulting in charred dark coloured high density boards. The Shen system on the other hand involved crosslinking spent sulfite liquor with sulfuric acid under high temperatures, thereby reducing the time required for pressing. But the higher press temperatures and acid concentrations above 0.9% resulted in charring of the boards. To overcome the long press times and charring/decomposition observed in the Pedersen and Shen procedures, the Nimz system was suggested where the spent sulfite liquor was oxidized with hydrogen peroxide using sulfur dioxide as a catalyst. This reaction results in radical coupling of the of the lignin molecules. Unfortunately, none of these processes has been successfully adapted for particleboard production on an industrial scale because they either required post-heat treatments and long press times for resin cure or produced corrosive gases which damaged equipment.

Numerous studies have been conducted and published on the use of lignin to substitute parts of PF resins for the manufacture of particleboard, fiberboard, oriented strand board and plywood (Kazayawoko et al. 1992; Vazquez et al. 1995; Danielson and Simonson 1998; Cetin and Ozmen 2002b; Cavdar et al. 2008; El Mansouri et al. 2007; Sukhbaatar et al. 2009; Kouisni et al. 2011; Hemmila et al. 2013; Zhang et al. 2013; Qiao et al. 2015; Stücker et al. 2016; Zhao et al. 2016). Yet the adoption of this resin technology industrially for the manufacture of particleboard has been hindered by the low reactivity of lignin towards formaldehyde necessitating very long press cycles at high temperatures which are not economically sustainable for industrial applications (Pizzi 2006). In most cases modification or pre-treatment of the lignin such as methylation, periodate oxidation and alkaline hydrolysis is required to improve its low reactivity towards formaldehyde (Kuo et al. 1991; Senyo et al. 1996; Cetin and Ozmen 2002a; Gosselink et al. 2011; Hemmila et al. 2013; Laurichesse and Avérous 2014). It has been reported that lignin can replace as much as 30% of the PF adhesive (Vazquez et al. 1997; Cetin and Ozmen 2003). In recent times a

combination of pre-methylolated lignin, PF and polymeric diphenyl methane diisocyanate resin (pMDI) has been successfully formulated and used on laboratory scale; this adhesive formulation reportedly produced industrially acceptable fast curing rates (Stephanou and Pizzi 1993a, 1993b). The pMDI resin acts as a fortifier and accelerates the cure reaction of the PF and lignin.

This study therefore explored further the concept of using lignin to substitute portions of pMDI resin to make pMDI a more cost-effective resin without compromising its strength properties. The current cost of pMDI resin is approximately \$1.8-\$2.20/kg while that of Kraft lignin is approximately \$0.4-\$0.5/kg. As well most of the lignin-based resin formulations that were mentioned in previous paragraphs have been used purely on wood particleboards and so far, to the best of our knowledge there is little published data on the use of lignin-based resins for the manufacture of particleboards from non-wood residues. Therefore, considering the growing interest in using agricultural crop and plant residues for particleboard production, this study also evaluated the efficacy of consolidating flax shive and hemp hurd residues with a pMDI-lignin based resin to understand the effects (if any) of these residues on the curing behavior of the resin. In line with the idea to reduce quantities of pMDI used in particleboard production and based on results from previous work (Chapter 3), this study used a low resin load of 2.5% pMDI (furnish oven dry weight) for particleboard manufacture. The total quantity of pMDI resin used was further reduced by substituting 5 and 20 weight % of the resin with Kraft lignin.

5.2 Experimental design

Isocyanate resin (pMDI) is commonly used as an accelerator and fortifier for UF and PF resins because of its fast cure rates and durability. Therefore, in this study direct substitution of pMDI with unmodified softwood Kraft lignin Type A was considered. The Kraft lignin was supplied by FPInnovations Forintek West.

Preliminary trials revealed it was possible to obtain a uniform dispersion of the Kraft lignin powder into the pMDI resin by direct mixing at room temperature. This was achieved by using a mechanical stirrer (Lightning Labmaster Model TS-2010) initially set at 350 rpm and slowly adding the Kraft lignin powder; once the required mass of Kraft lignin was added, stirring was

continued for another 15 minutes at 800 rpm to ensure maximum dispersion of the lignin in the resin (Figure 5.1). An increase in the substitution levels of lignin above 20 weight % resulted in a thick adhesive formulation that was difficult to atomize with the spraying system in the particleboard blender.



Figure 5.1: Mechanical stirring of Kraft lignin and pMDI resin. Source: Sam-Brew 2016.

Subsequently the adhesive formulations with 5 and 20 weight % Kraft lignin substitution for pMDI at a 2.5% resin load was used to manufacture 3-layered particleboards from wood, hemp hurd and flax shive residues per the design of experiment summarized in Table 5.1 and the manufacturing procedure described in Section 2.2.3. An average MC of 10% was logged for the wood, flax shive and hemp hurd residues prior to particleboard manufacture. Because of the low reactivity of lignin which customarily requires longer press times, the holding period of the press cycle given in section 2.2.3 was adjusted from 415 s to 515 s to ensure resin cure. Five board replicates were

manufactured for each combination of residue and lignin substitution level for a total of 30 particleboards.

Table 5.1: Design of experiment for particleboards manufactured with pMDI-lignin resin.

	Factors	Levels	Response	Total specimens
Variables	Residue type	wood	MOR/MOR	12
		hemp hurd	IB	32
		flax shive	LE	16
	Lignin % (w/w)	5	TS	12
		20		
Constants	Density (kg/m ³)	620		
	Thickness (mm)	12.7		
	Resin type	pMDI		
	Resin load (%)	2.5		
	Replicates	5		

5.3 Results and discussion

5.3.1 Moisture content, average board density and vertical density profile

The average board density and moisture content (MC) for the lignin-based particleboards is presented in Table 5.2. No significant differences were observed between the particleboards in terms of MC (Appendix D), but the flax shive particleboards particularly those with 5 weight % lignin substitution had a slightly higher MC compared with the other particleboard types. The board MC's were similar to those reported for particleboards manufactured with pMDI in Chapter 3. Analysis of the average board density revealed significant differences ($p < 0.0001$) between the particleboards based on residue type and not lignin substitution levels (Appendix D); the wood particleboards (5 and 20 weight % lignin substitution) were significantly higher in density compared to the hemp hurd and flax shive particleboards. The hemp hurd and flax shive board densities in comparison were not significantly different from each other. The trend observed with

the average board density is similar to that observed in wood, hemp hurd and flax shive particleboards manufactured with 2.5% pMDI resin in Chapter 3 (section 3.3.3).

Table 5.2: Average board density and moisture content of pMDI-lignin particleboards. n=12 for each mean. Values in parenthesis are standard deviations.

Lignin	wood		hemp hurd		flax shive	
	5%	20%	5%	20%	5%	20%
Density (kg/m ³)	552 (19.36)	556 (20.85)	522 (25.94)	521 (22.16)	546 (12.96)	543 (19.63)
MC (%)	10.53 (0.07)	10.53 (0.09)	10.84 (0.10)	10.84 (0.09)	11.19 (0.12)	10.92 (1.42)

As with the average board densities, a look at the density profile through the thickness of the boards also revealed no significant differences between particleboards manufactured from the same residue at either 5 or 20 weight % lignin substitution levels (Figure 5.2). Significantly lower ($p < 0.0001$) face densities were observed in the wood boards (5 and 20 weight % lignin) at 585 kg/m³ with the hemp hurd boards recording the highest face densities at 711 kg/m³. The core densities ranging from 453-497 kg/m³ were significantly higher ($p = 0.002$) in the flax shive boards in comparison to only the wood boards. The difference between the face and core densities as seen in Figure 5.2 explains the low average board density observed in Table 5.2 for the hemp hurd boards and also accounts for the relatively higher board density values observed in the wood boards.

The vertical density gradient observed here is consistent with those previously observed in Chapter 3 (Section 3.3.3) for wood, hemp hurd and flax shive particleboards made with 2.5% pMDI resin load at the same initial moisture content. Indicating the addition of lignin to board manufacture did not adversely affect the board density gradient in anyway.

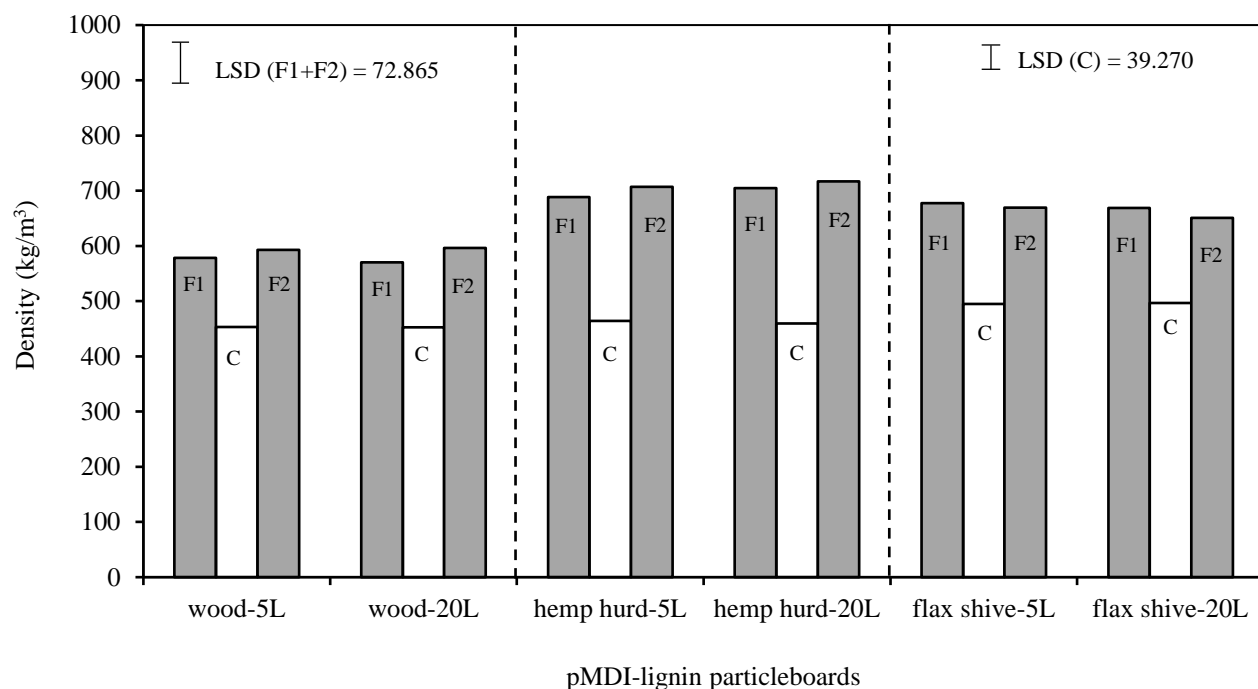


Figure 5.2: Face and core density profiles of pMDI-lignin particleboards; peak face (F1, F2) and core densities (C), 5% lignin (5L), 20% lignin (20L). $n=6$ for each mean. Error bars represent least significant difference (LSD) between means.

5.3.2 DSC analysis

5.3.2.1 pMDI and Kraft lignin

The DSC scans for the neat pMDI resin in combination with Kraft lignin type A with no added water (Figure 5.3) shows two distinct exothermic peaks indicating a two-phase reaction sequence. For the 5 weight % Kraft lignin substitution in pMDI resin (that is pMDI+5lignin), the first broad and less defined exothermic reaction occurs at a lower temperature range 85 °C to 160 °C and is thought to be the initial reaction of the pMDI with the lignin in the absence of moisture to form urethane or covalent bonds. The second narrow more defined exothermic peak occurs at higher temperatures between 170 °C to 200 °C and is attributed to further cure of the pMDI resin molecules and possible decomposition of the Kraft lignin due to impurities in contained in it (which resulted in degradation at lower temperatures). Similar observations were made at 10 weight % lignin substitution levels (that is pMDI+10lignin) with the second exothermic peak within the same temperature range having a relatively larger area in contrast. The opposite was

observed for samples with the 20 weight % Kraft lignin substitution in pMDI resin (that is pMDI+20lignin) where the first exothermic peak was large and well defined over a larger temperature range of 70 °C to 183 °C and a smaller exothermic peak occurring between 185 °C to 220 °C.

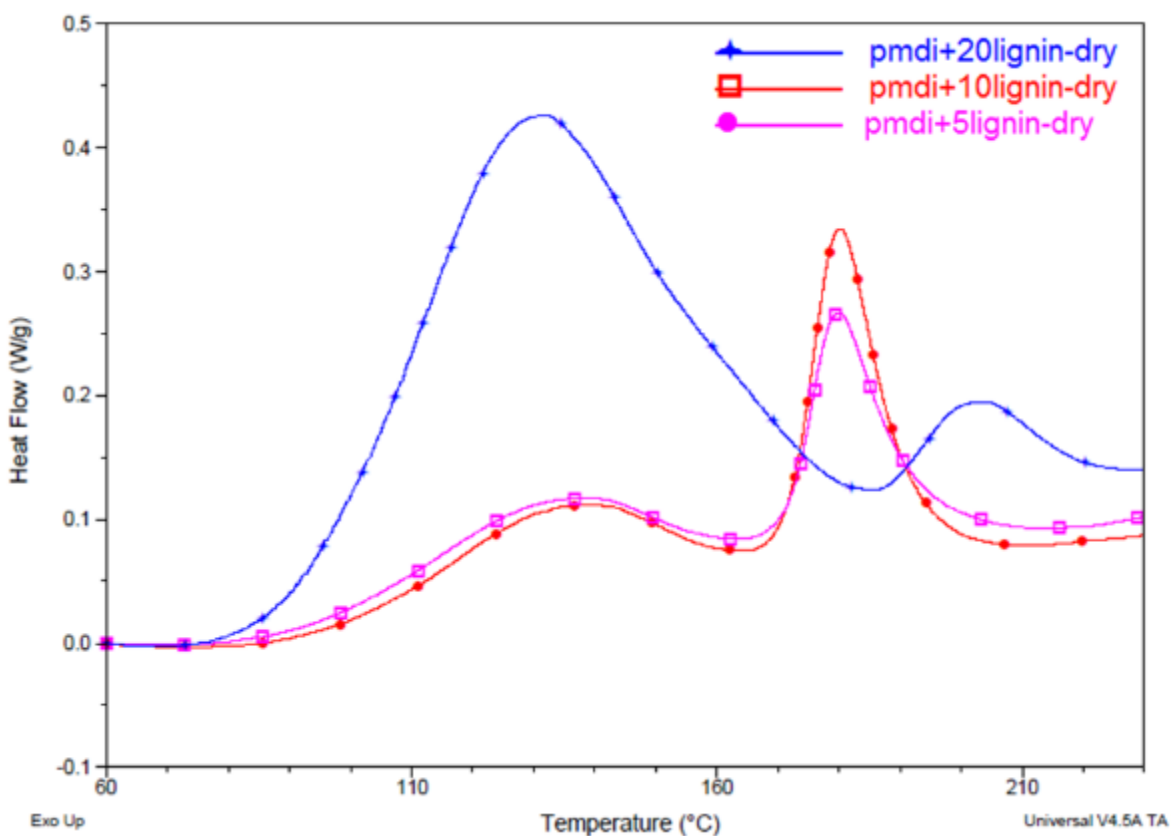


Figure 5.3: DSC graph of pMDI and Kraft lignin at different mass substitutions.

With the addition of distilled water, only a single exothermic reaction was observed for all levels of Kraft lignin substitution in pMDI resin as seen in Figure 5.4. The extracted thermal properties revealed no significant differences in onset temperatures between the 3 lignin substitution levels (Table 5.3 and Appendix D). But there were significant differences ($p=0.0055$) in peak temperature where the pMDI+5lignin resin yielded a higher temperature implying a slower reaction rate compared to the 10 and 20 weight % lignin substitutions which contained more lignin (Appendix D). Significant differences ($p=0.0033$) were also observed in the reaction heat between all the

lignin substitution levels (Appendix D); the pMDI+20lignin attained the lowest reaction heat values (bond formation between the resin molecules) consistent with the low amount of pMDI resin in the mixture when compared to the 5 and 10 weight % lignin substitutions.

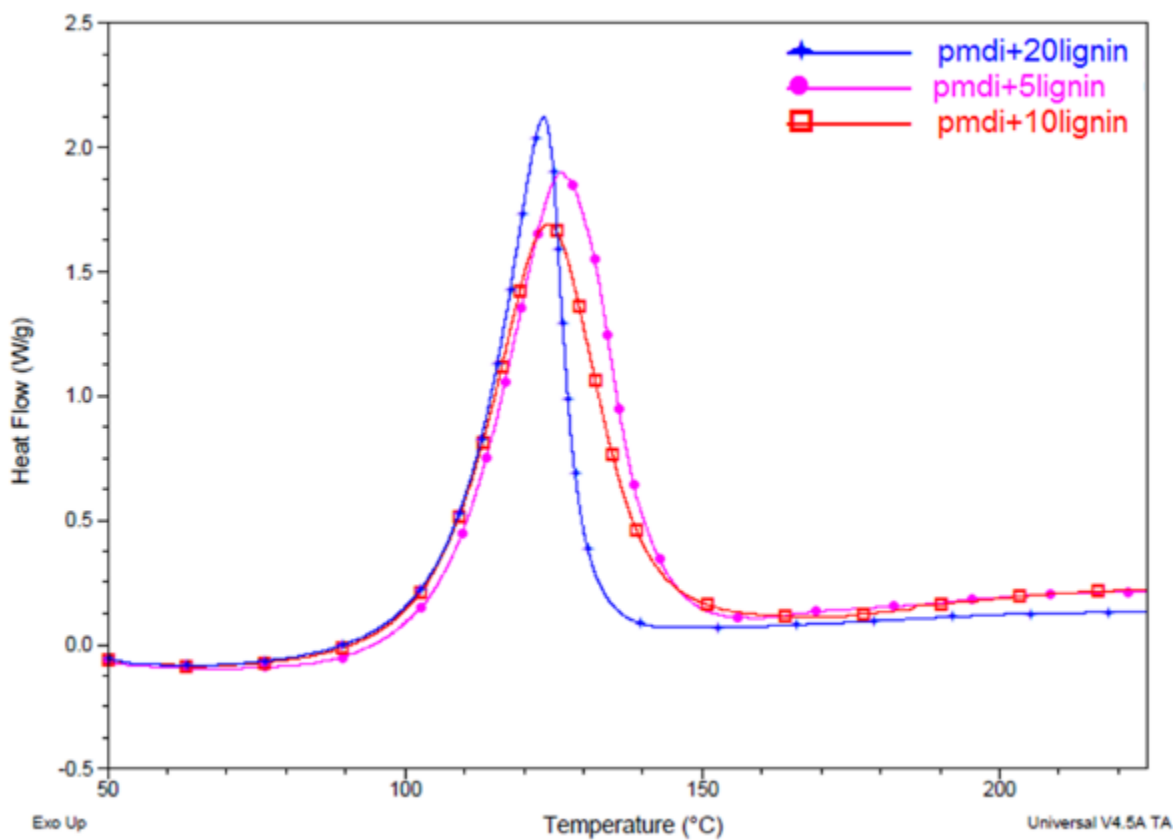


Figure 5.4: DSC graph of a mixture of distilled water, pMDI and Kraft lignin at different mass substitutions.

Table 5.3: Thermal properties of the curing reaction between pMDI and Kraft lignin.

Sample	Onset Temp. (°C)	Peak Temp. (°C)	Reaction heat (J/g)
pMDI+5L	107.1	126.4	252.9
pMDI+10L	107.0	124.4	231.4
pMDI+20L	108.7	123.5	199.3

5.3.2.2 pMDI and 5 weight % lignin substitution

The DSC data for the pMDI resin with 5 weight % Kraft lignin substitution (pMDI+5lignin) in combination with wood, hemp hurd and flax shive residues are shown in Figures 5.5 and 5.6. A single distinct exothermic reaction was observed in the DSC graphs. Analysis of the graphs showed significant differences ($p=0.0009$) in the onset temperature of resin cure between the 3 residue types (Appendix D); the wood samples yielded the lowest onset temperatures (105 °C) suggesting the curing reaction of the pMDI+5lignin resin proceeded easily in the presence of the wood residues, followed by the hemp hurd (110 °C) and flax shive residues (111 °C). The pMDI+5lignin resin and wood mixture was the only sample where onset temperatures were lower than those attained by the neat pMDI+5lignin resin (107 °C).

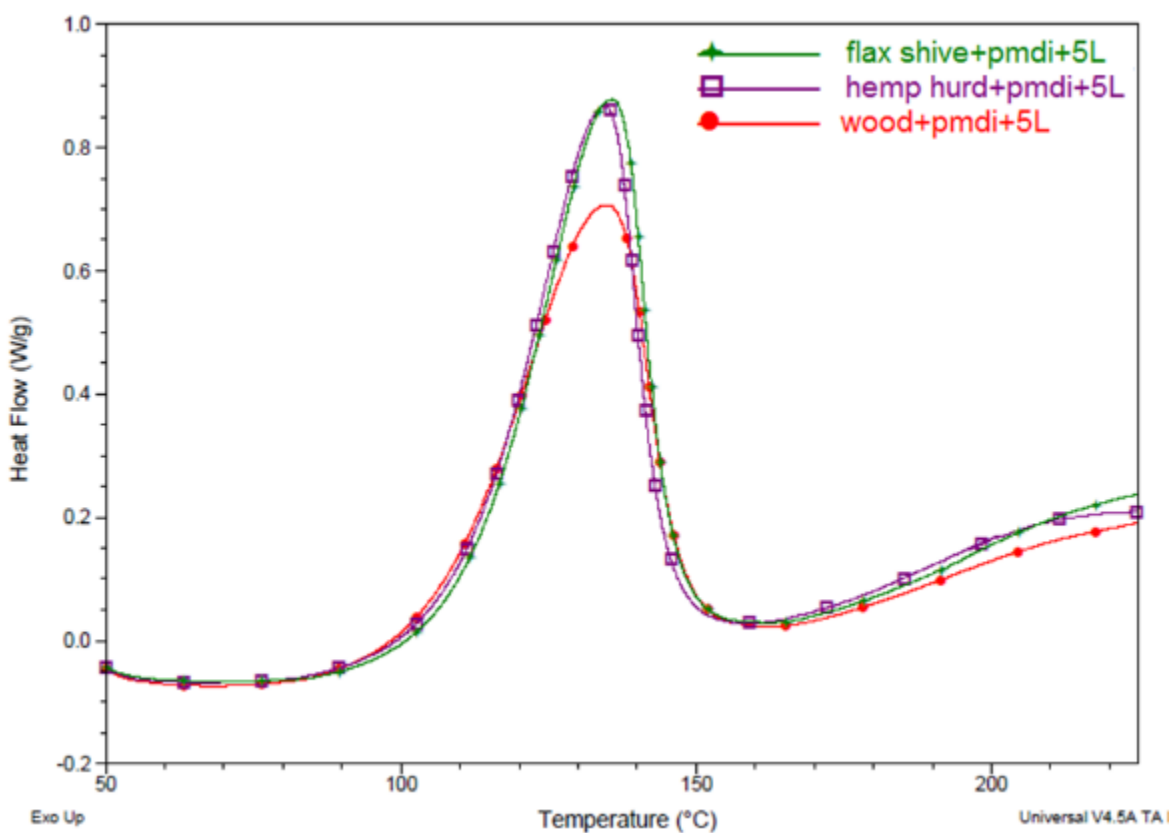


Figure 5.5: DSC graphs of different residues combined with pMDI and 5 weight % Kraft lignin substitution.

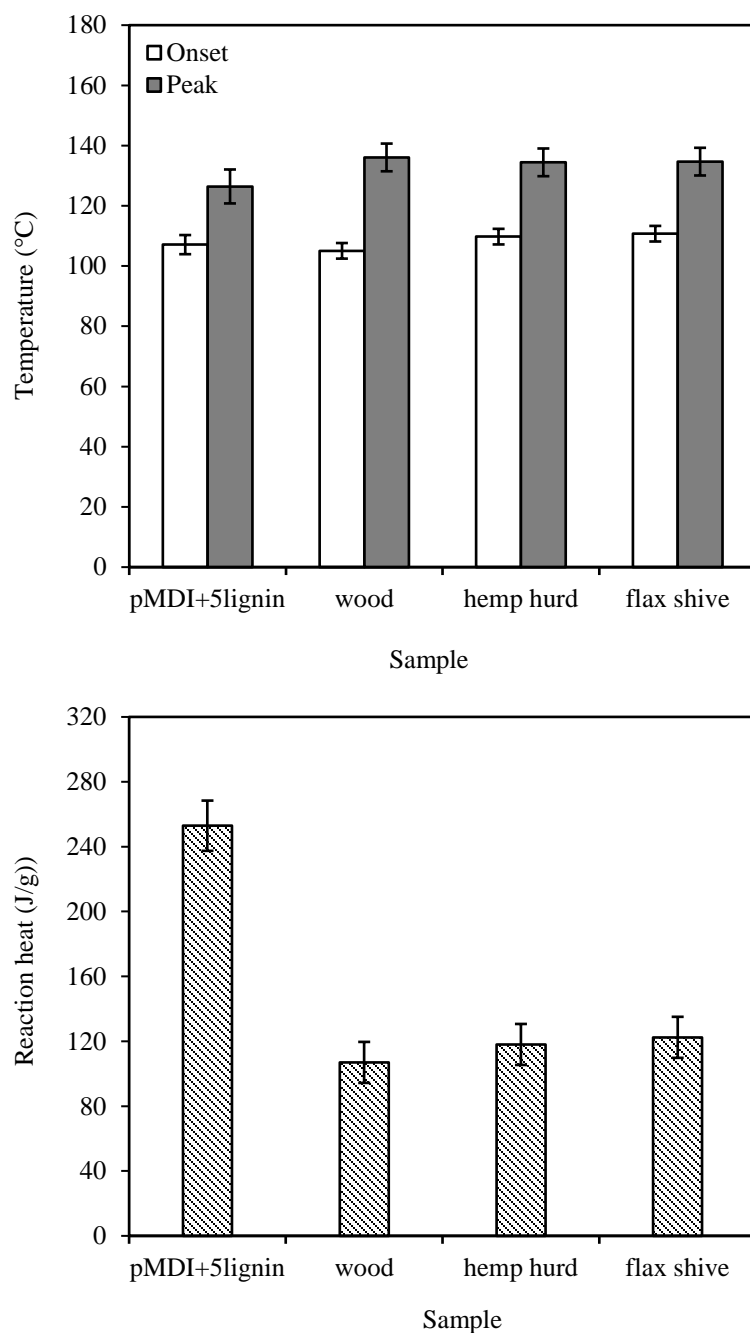


Figure 5.6: Onset temperature, peak temperature and reaction heat comparison between different residues combined with 5 weight % lignin in pMDI resin. $n=3$ for each residue mean and $n=2$ for pMDI-lignin resin. Error bars represent 95% confidence intervals.

No significant differences were observed in peak temperatures for all 3 residue types in combination with the pMDI+5lignin resin (Appendix D) signifying a similar rate of cure for the resin in all cases. The peak temperatures for the pMDI+5lignin resin and residue mixtures were significantly higher ($p=0.0020$) than that observed in the neat pMDI+5lignin resin, an indication that the reaction cure of the resin was slowed down by the addition of the residues (Appendix D). For the reaction heat the neat pMDI+5lignin resin yielded significantly ($p<.0001$) higher heat of reaction at 253 J/g, approximately 50% higher compared to the pMDI+5lignin resin and residue mixtures (Appendix D). A similar trend was observed in section 3.3.4 for the DSC results of neat pMDI resin versus samples of pMDI resin combined with residues. Of the pMDI+5lignin resin and residue mixtures, the flax shive samples yielded the highest reaction heat (122 J/g) and hence the highest bond formation between the resin molecules, followed by the hemp hurd samples (118 J/g) and the wood samples having the lowest reaction heat (107 J/g).

5.3.2.3 pMDI and 10 weight % lignin substitution

Figures 5.7 and 5.8 represent the DSC results for the pMDI resin with 10 weight % Kraft lignin substitution (pMDI+10lignin) in combination with wood, hemp hurd and flax shive residues. As with the 5 weight % lignin substitution there were significant differences ($p=0.0002$) in onset temperatures and none in peak temperature for the pMDI+10lignin and residue mixtures (Appendix D). In a trend similar to that observed in the 5 weight % lignin substitution, the lowest onset temperature of resin cure was observed in the wood samples at 105 °C, and after that the hemp hurd (109 °C) and flax shive (110 °C) samples at relatively higher temperatures. As with the 5 weight % lignin substitution, the onset temperature of pMDI+10lignin resin and wood mixture were lower than that for the neat pMDI+10lignin (107 °C)

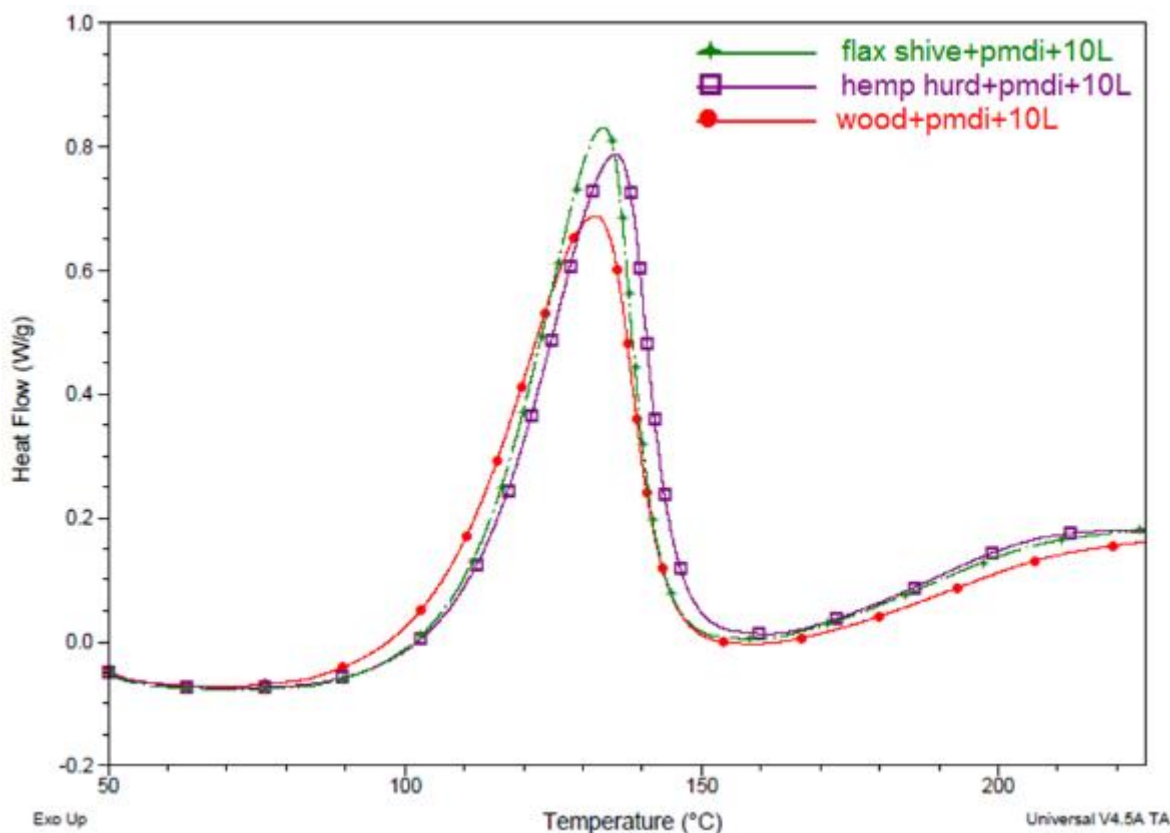


Figure 5.7: DSC graphs of different residues combined with pMDI and 10 weight % Kraft lignin substitution.

The neat pMDI+10lignin resin yielded a significantly ($p=0.0003$) faster curing rate (peak temperature) compared to the same resin in combination with residues (Appendix D). There was no significant difference in reaction heat between the pMDI+10lignin resin and residue mixtures (Appendix D) though as always the resin in the presence of the flax shive residues attained a higher reaction heat and greater degree of resin cure. A comparison of the reaction heat of the pMDI+10lignin resin and residue mixtures with the neat pMDI+10lignin resin also revealed approximately 50% less bonds between the resin molecules in the former.

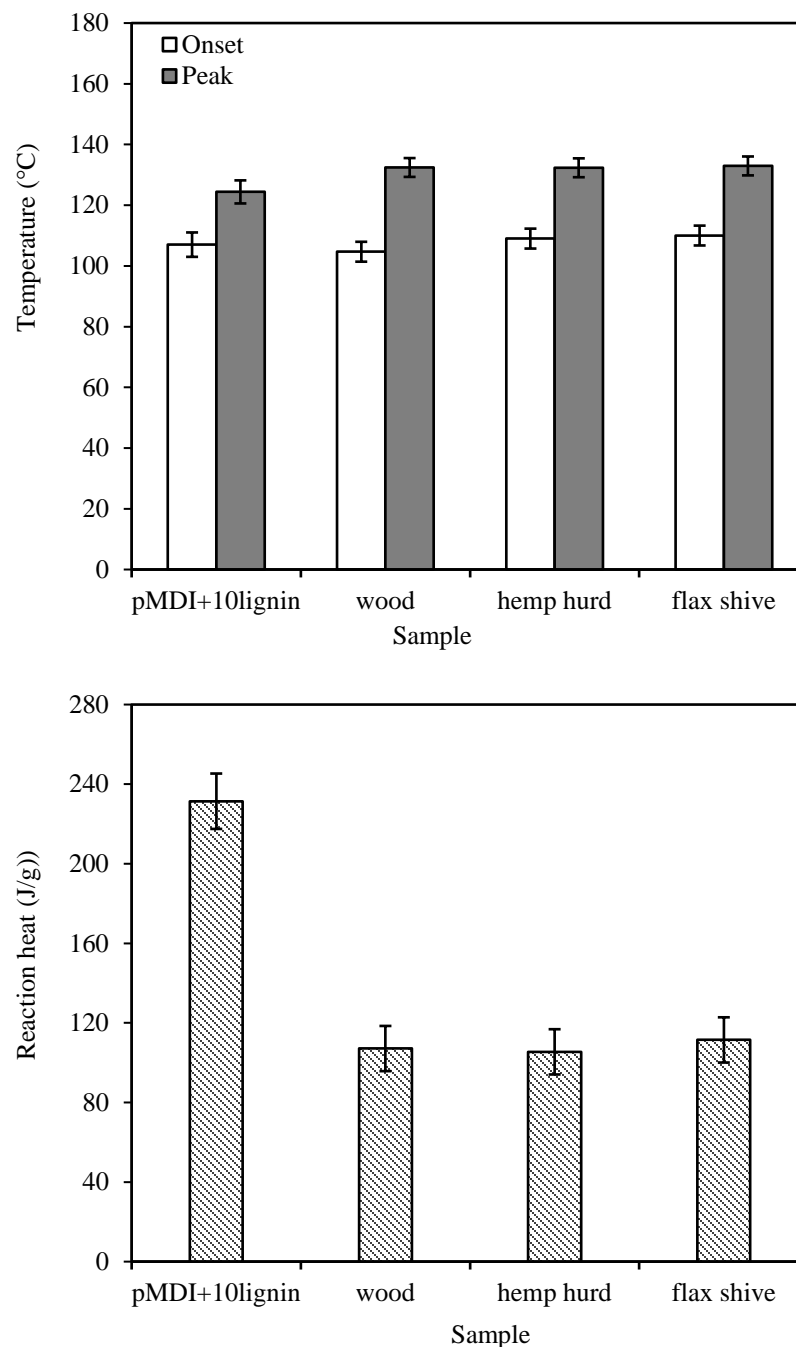


Figure 5.8: Onset temperature, peak temperature and reaction heat comparison between different residues combined with 10 weight % lignin in pMDI resin. $n=3$ for each residue mean and $n=2$ for pMDI-lignin resin. Error bars represent 95% confidence intervals.

5.3.2.4 pMDI and 20 weight % lignin substitution

Figures 5.9 and 5.10 provide the results for the DSC scans on mixtures of pMDI resin containing 20 weight % Kraft lignin substitution (pMDI+20lignin) and wood, hemp hurd and flax shive residues. Significant differences ($p=0.0011$) were observed in onset temperature of resin cure between the 3 residue types with the pMDI+20lignin resin and wood mixture having an early start to its curing reaction (Appendix D); confirming once again the greater initial reactivity of the pMDI-lignin resin in the presence of wood residues.

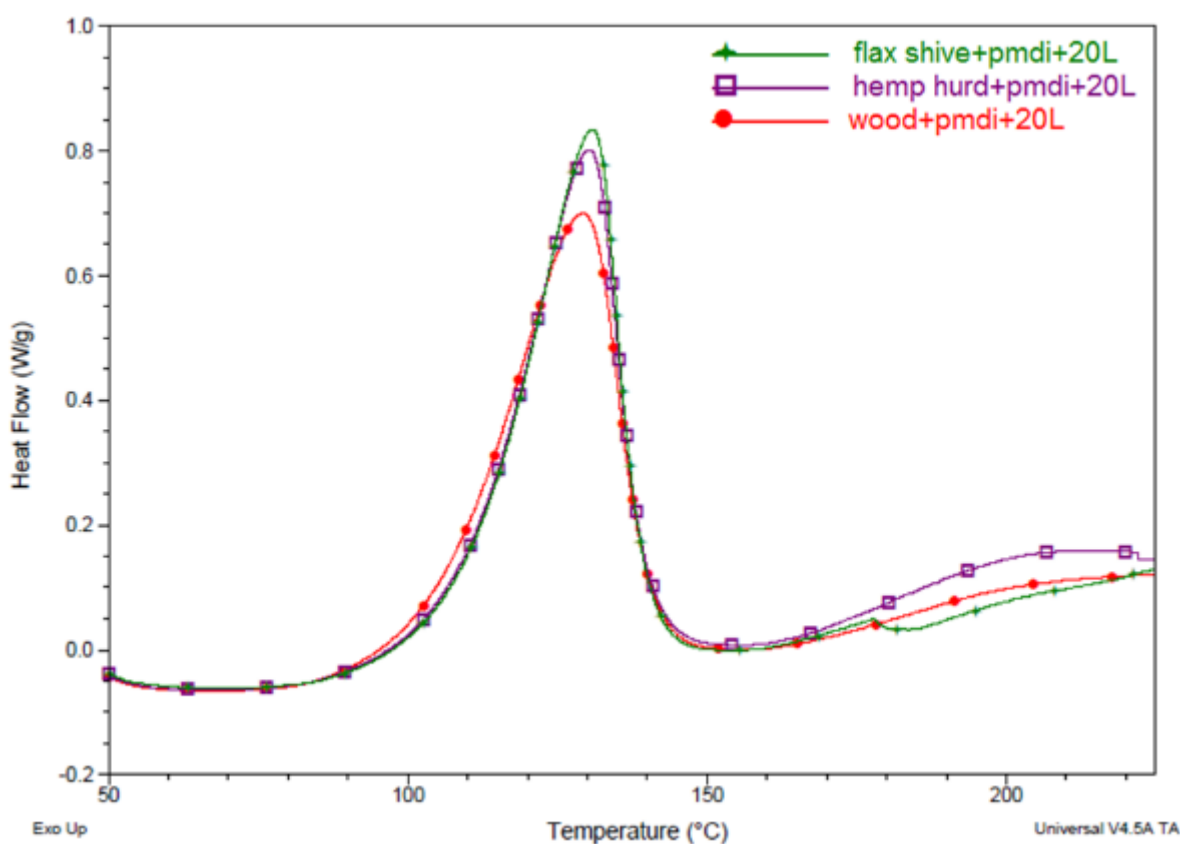


Figure 5.9: DSC graphs of different residues combined with pMDI and 20 weight % Kraft lignin substitution.

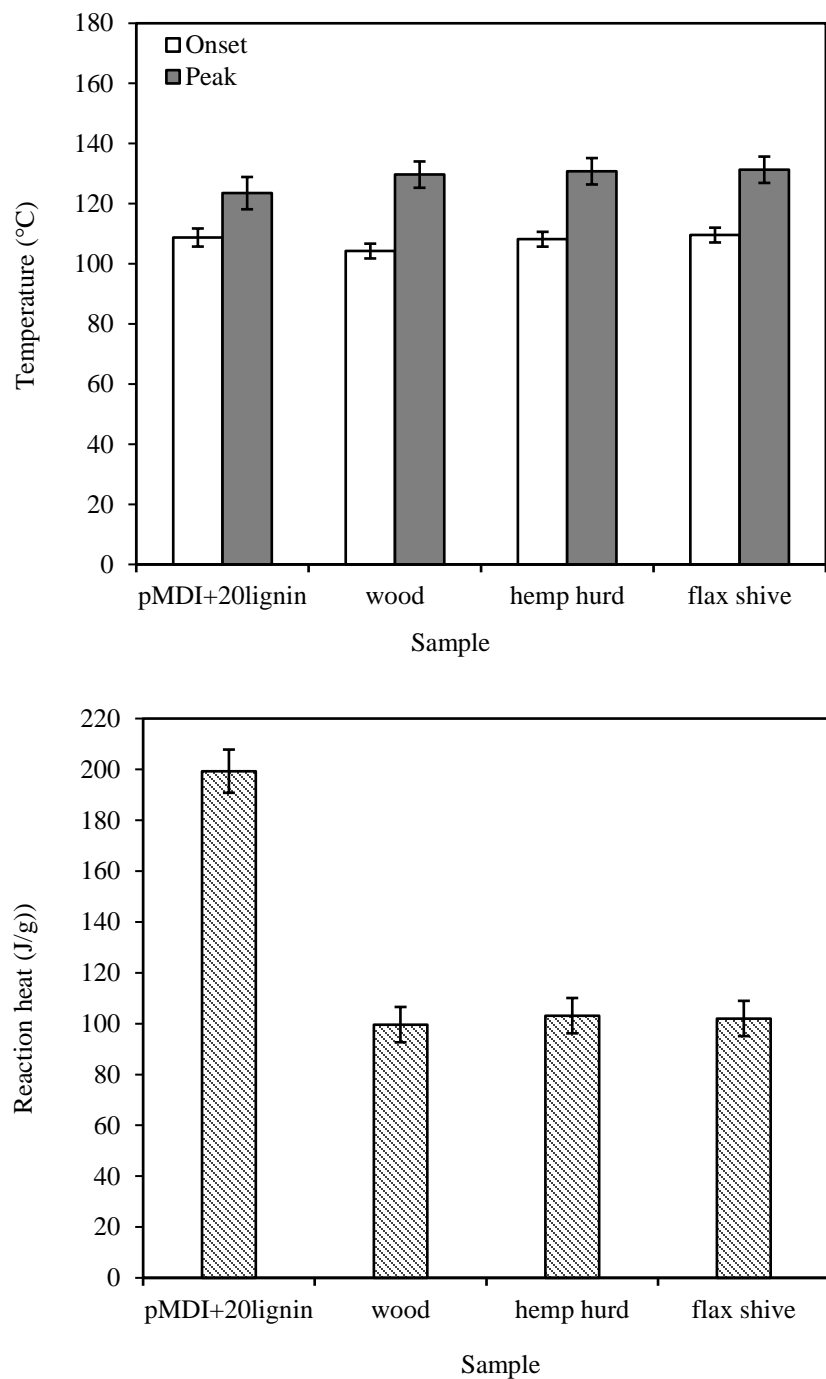


Figure 5.10: Onset temperature, peak temperature and reaction heat comparison between different residues combined with 20 weight % lignin in pMDI resin. $n=3$ for each residue mean and $n=2$ for pMDI-lignin resin. Error bars represent 95% confidence intervals.

No significant differences in resin cure were observed between residue types for the peak temperatures and reaction heat (Appendix D). At the end of the curing process, the resin cure was highest in the hemp hurd residues at 103 J/g and the lowest in the wood residues at 100 J/g. In line with the 5 and 10 weight % substitution levels, a comparison of the neat pMDI+20lignin resin sample to the pMDI+20lignin and residue mixtures revealed significant differences in both peak temperature ($p=0.0047$) and reaction heat ($p<0.0001$) due to the neat pMDI+20lignin resin sample attaining relatively lower values for the peak temperature and approximately 48% higher values for the reaction heat in comparison to the pMDI+20lignin and residue mixtures (Appendix D).

5.3.2.5 Effect of residue type on curing behavior of pMDI-lignin resins

The results of the thermal analysis presented above consistently indicates a single exothermic peak for all residues (wood, hemp hurd and flax shive) in combination with the pMDI-lignin resins. Onset of resin cure for all the pMDI-lignin resin and residue mixtures was observed within the temperature range 104 °C to 111 °C and showed significant differences between the wood samples in comparison to the hemp hurd and flax shive samples (which were not significantly different from each other). Onset of resin cure consistently began at lower temperatures for the pMDI-lignin resin and wood mixture and at higher temperatures for the pMDI-lignin resin and flax shive mixture. The waxy surface of the flax shive and hemp hurd residues may have retarded the initial reaction between the resin molecules. It can therefore be inferred that in the presence of the 3 residues, onset of curing of the pMDI-lignin resin is fastest in the wood samples.

With exception of the pMDI+5lignin resin (pMDI with 5 weight % lignin substitution), the peak temperatures and hence the temperature at which the cure reaction reaches a maximum were consistently lower in the wood samples and highest in the flax shive samples, confirming the quick reaction rates observed in the onset temperatures.

In contrast to the observations made from the onset and peak temperatures, the lowest reaction heats and lowest degree of resin cure was usually observed in the pMDI-lignin resin and wood mixtures with exception of the 10 weight % lignin substitution in pMDI. Rather the pMDI-lignin resin and flax shive mixtures attained the highest reaction heat and degree of resin cure, with

exception of the 20 weight % lignin substitution where it was 1 °C lower than the hemp hurd samples which usually attained the second highest reaction heat.

The results observed in this study for the effect of residue types on the onset temperature of pMDI-lignin resin cure are in line with previous observations made in Chapter 3 (section 3.3.4) for similar residues in combination with neat pMDI resin. Also in the case of the reaction heat, results of both studies are in agreement that the highest degree of resin cure is observed when the flax shive residues is combined with the neat pMDI and pMDI-lignin resins. But regarding the peak temperature there are slight variations in the data obtained. The peak temperatures for the residues in combination with neat pMDI resin indicates a faster cure reaction for the resin in the flax shive samples with the wood residues second in line. This contrasts with the current study where no significant differences were observed in peak temperatures between all 3 residues, though generally a relatively faster curing reaction was attained for both the 5 and 10 weight % lignin substitution in pMDI in combination with the hemp hurd residues.

The results above once again bring into question the differences in the curing behaviour of the resin in the presence of the residues. As with the case of the pMDI resin in section 3.3.4 it is believed that the observations made are the effect of a combination of the residue chemical constituents and anatomy, and their subsequent effect on the cure between the resin molecules — a subject area that requires further work to identify and understand the main factors prohibiting resin cure.

5.3.2.6 Effect of lignin substitution levels on the curing behavior of residues.

Figures 5.11, 5.12 and 5.13 represent a comparison of the onset temperatures, peak temperatures and reaction heats based on residue type (wood, hemp hurd, flax shive) and lignin substitution levels in pMDI resin.

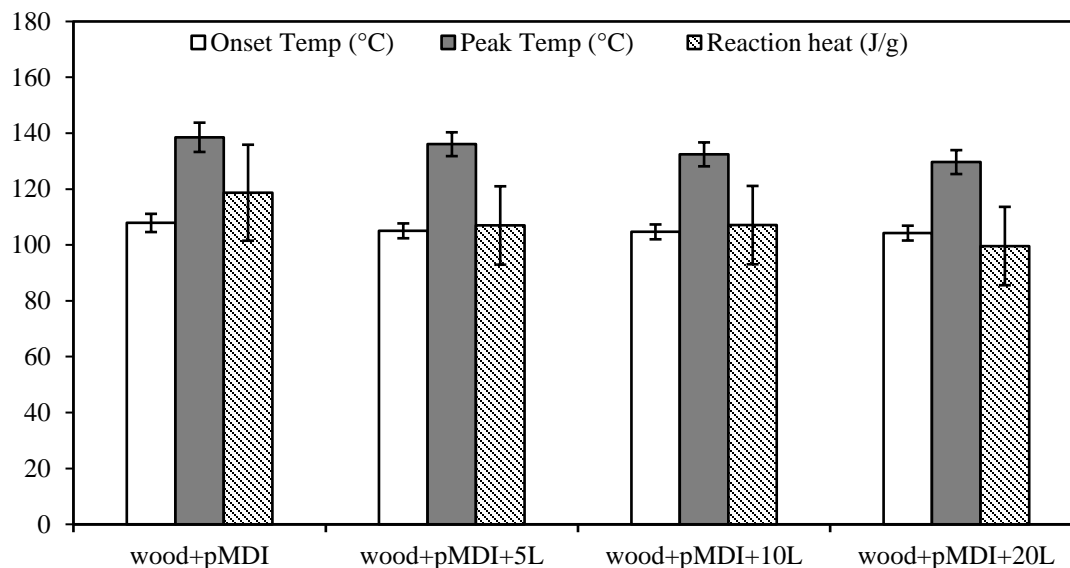


Figure 5.11: Thermal properties of mixtures of wood residue and pMDI resin based on mass lignin substitution levels. $n=3$ for each mean. Error bars represent 95% confidence intervals.

In the presence of each type of residue either wood, hemp hurd or flax shive samples, the onset and peak temperatures, and the reaction heat values decreased with increasing lignin substitution in pMDI resin from 5 to 20 weight %. For each residue, this indicates an initial faster reaction rate of the resin with the addition of more lignin, but at the same time a lower degree of cure between the resin molecules. The reduction in reaction heat and onset temperatures observed for all the residue types, was mostly due to differences between samples with no or low lignin content (that is samples combined with the neat pMDI and the lower 5 weight % lignin substitution) and samples with higher lignin substitution levels (10 and 20 weight % lignin substitutions) as seen from the Figures 5.11, 5.12 and 5.13. This reduction in reaction heat values (i.e. the amount of chemical bond formation between the resin molecules) is ultimately a reflection of the quantity of pMDI resin molecules available in the system. These results suggest that the Kraft lignin does not react with the pMDI as was expected in the presence of these residues. This effect of lignin substitution levels observed is likely to negatively influence particleboard mechanical and physical strength properties.

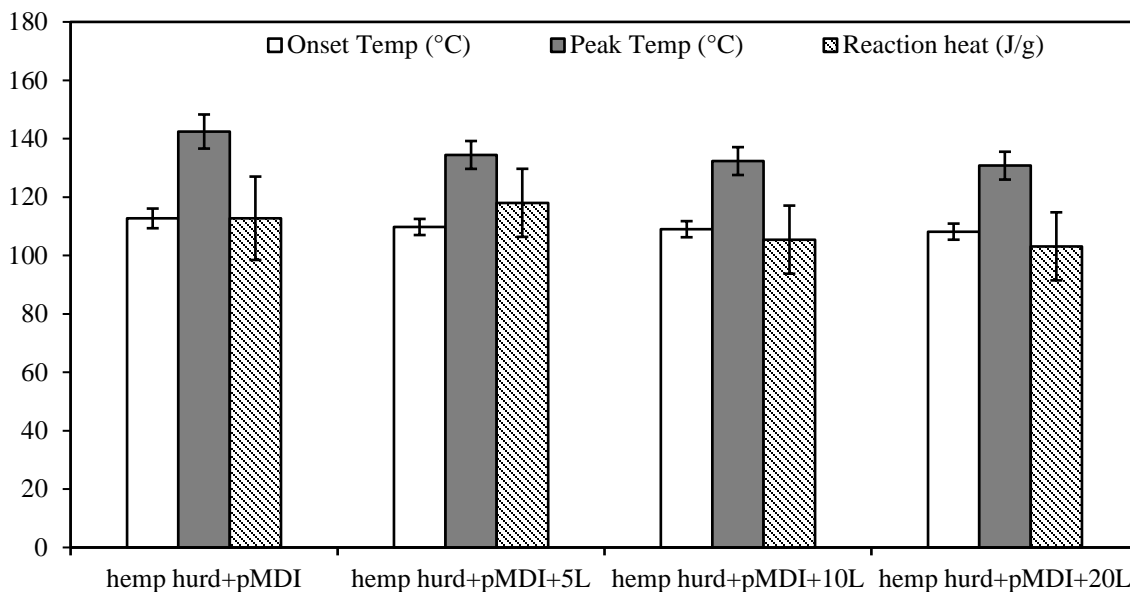


Figure 5.12: Thermal properties of mixtures of hemp hurd residue and pMDI resin based on mass lignin substitution levels. $n=3$ for each mean. Error bars represent 95% confidence intervals.

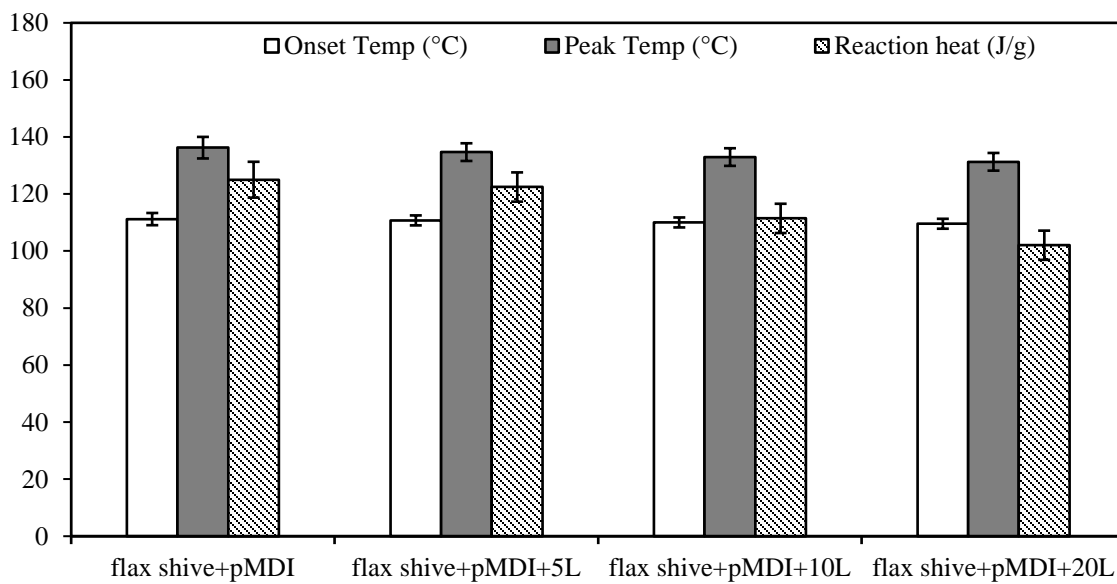


Figure 5.13: Thermal properties of mixtures of flax shive residue and pMDI resin based on mass lignin substitution levels. $n=3$ for each mean. Error bars represent 95% confidence intervals.

For all 3 lignin substitutions (5%, 10%, 20%) in pMDI resin, the reaction heat was relatively higher in combination with flax shive residues and lowest with the wood residues, indicating higher degree of resin cure in the presence of the flax shive residues compared to the wood residues.

5.3.3 Mechanical properties

5.3.3.1 IB

Table 5.4 lists the results of the IB strength properties of particleboards manufactured from pMDI with 5 and 20 weight % Kraft lignin substitution. For comparison, wood (576 kg/m³), hemp hurd (540 kg/m³) and flax shive (541 kg/m³) particleboards manufactured without lignin at a 2.5% pMDI resin load from previous studies presented in Chapter 3 are included in the table as control particleboards. With exception of lignin substitution, the control particleboards were manufactured with the same processing parameters (resin load, target density, thickness, temperature). The IB results for the pMDI-lignin particleboards are also graphically presented in Figure 5.14.

*Table 5.4: Internal bond properties of particleboards made with 2.5% pMDI-lignin resin load. Panel means based on ANSI A208.1-1999. For each mean n=32. Values in parenthesis are standard deviations. * indicates control samples.*

Board Type	IB (MPa)	
	5L	20L
wood	0.60 (0.09)	0.50 (0.11)
hemp hurd	0.80 (0.08)	0.62 (0.04)
flax shive	0.59 (0.07)	0.50 (0.05)
wood*	0.74 (0.15)	
hemp hurd*	0.87 (0.10)	
flax shive*	0.61 (0.08)	
ANSI LD2	0.15	
ANSI M2	0.45	

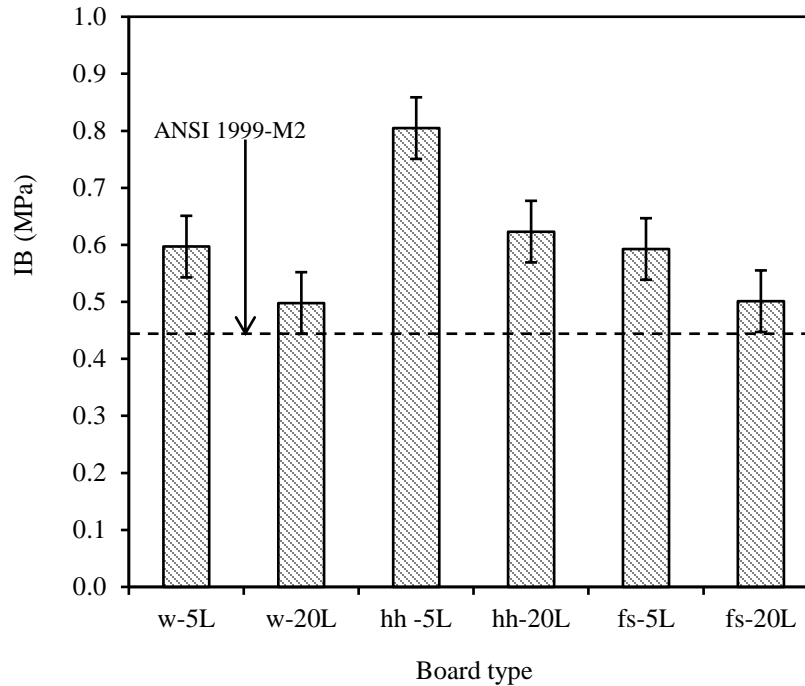


Figure 5.14: Internal bond strength of particleboards manufactured from pMDI-lignin resin; 5% lignin (5L), 20% lignin (20L), w (wood), hh (hemp hurd), fs (flax shive). Horizontal line indicates minimum value stipulated by ANSI A208.1-1999 for M2 grade particleboards $n=32$ for each mean. Error bars represent 95% confidence intervals.

Significant differences ($p < 0.0001$) were observed in bond strength between all the particleboards based on residue type and lignin substitution level (5 and 20 weight %) (Appendix D). Higher IB values were observed in the hemp hurd particleboards particularly boards containing 5 weight % lignin substitution. No significant differences were observed in bond strength between the wood and flax shive particleboards at the same lignin substitution level. The trend observed here for IB results are similar to those observed in the control wood, flax and hemp hurd particleboards (manufactured from 2.5% pMDI resin load) where the hemp hurd particleboards attained the highest bond strength. All particleboards met the minimum ANSI A208.1-1999 requirements for low density LD2 particleboard and exceeded that of medium density M2 grade particleboard.

The internal bond values obtained for the particleboards with lignin substitution were lower compared to the control boards without lignin, with an increase in lignin content leading to a further decrease in internal bond strength (Figure 5.15); a decrease of 3–18% for particleboards

with 5 weight % lignin substitution and 17–32% for particleboards with 20 weight % lignin substitution. Similar results were reported by Cetin and Ozmen (2002b, 2003) for particleboards bonded with unmodified, phenolated and methylolated organosolv lignin-PF resin formulations.

This decrease in bond strength with lignin content was most evident in the wood and hemp hurd particleboards while the flax shive boards were the least affected as shown in Figure 5.15. These IB results are in line with the DSC results where increasing lignin content was linked with decreasing reaction heat and degree of cure between the resin molecules as seen in Figure 5.11, 5.12 and 5.13. The result suggests that though the addition of the Kraft lignin initially accelerates the curing reaction of the resin it does not necessarily result in the formation of effective bonds between the resin molecules, and this is more evident as the Kraft lignin content increases and the pMDI content decreases.

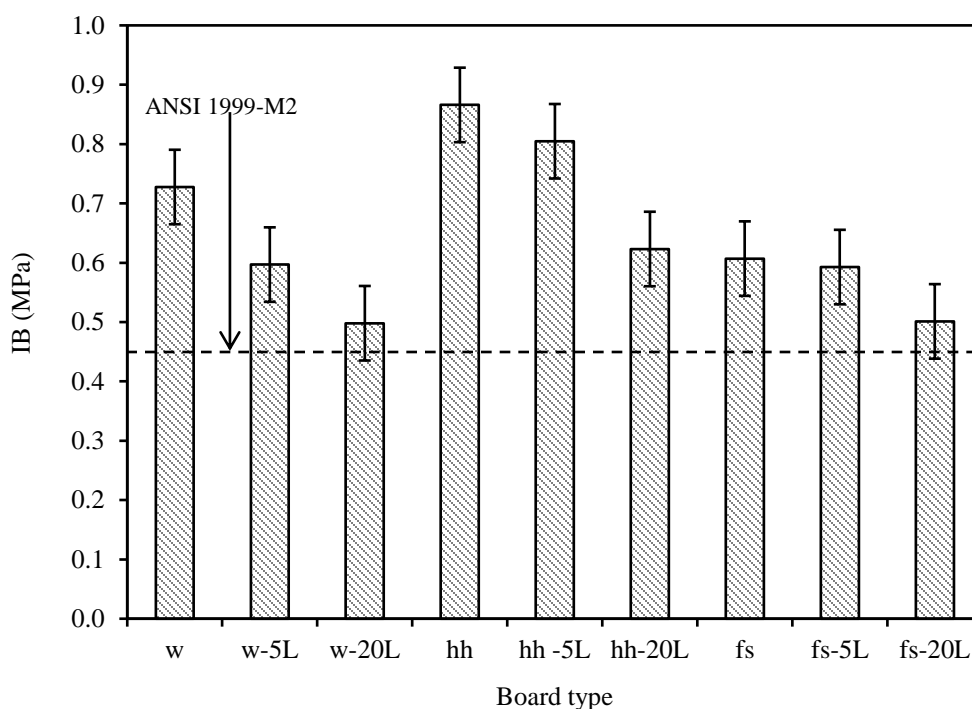


Figure 5.15: Comparison of internal bond strength of particleboards manufactured with pMDI and pMDI-lignin resin; 5% lignin (5L), 20% lignin (20L), w (wood), hh (hemp hurd), fs (flax shive). Horizontal line indicates minimum value stipulated by ANSI A208.1-1999 for M2 grade particleboard. n=32 for each mean. Error bars represent 95% confidence intervals.

In 2007, El Mansouri et al. (2007) worked on single layered beech and spruce particleboards bonded with methylolated and glyoxalated lignin-pMDI resin at 60:40 component ratios. The lignin was calcium lignosulfonate based. The 14 mm particleboards were manufactured with 10% resin content, at 195-200 °C in 3.5 to 7.5 minutes to a density of approximately 700 kg/m³. IB strength results ranged between 0.76–0.82 MPa for the methylolated lignin-pMDI boards and 0.67-0.81 MPa for the glyoxalated lignin-pMDI. Though these particleboards had a higher percentage of lignin and attained relatively higher IB values compared to those from the current study, it is important to note that the lignin was pretreated (methylolated/glyxolated) increasing its reactivity, the resin formulation was also applied at higher loads, and the particleboards pressed at a higher temperature to a higher density. In contrast the Kraft lignin utilized in this study were used directly and unmodified, applied at a lower resin load of 2.5% and pressed at 140 °C to a low-density range of approximately 521-556 kg/m³.

5.3.3.2 MOR and MOE

In terms of static bending properties (Table 5.5 and Figures 5.16 and 5.17), there were significant differences ($p < 0.0001$) in both MOR and MOE between all the particleboards based on the type of residue used (Appendix D). For both lignin substitution levels (5 and 20 weight %), the flax shive and hemp hurd particleboards attained higher MOR and MOE values approximately 73% and 60% greater compared to the wood particleboards. Within each board type the only significance between lignin substitution levels (5 and 20 weight %) was observed in MOR for the flax shive particleboards (Appendix D). The advantage of the flax shive and hemp hurd particle geometry described in Chapter 3 (section 3.3.1) was observed once again as particleboards made from these residues at all lignin substitution levels exceeded the minimum bending strength and stiffness properties stipulated by ANSI A208.1-1999 for low density LD2 particleboard. The flax shive particleboard with 5 weight % lignin substitution was the only board to attain MOE properties that exceeded the ANSI requirements for medium density M2 grade particleboard.

Table 5.5: Mechanical strength properties of pMDI-lignin particleboards. Panel means presented according to ANSI A208.1-1999. For each mean $n=32$ for IB and $n=12$ for MOR/MOE. Values in parenthesis are standard deviations. * indicates control panels.

Board Type	MOR (MPa)		MOE (GPa)	
	5L	20L	5L	20L
Wood	2.38 (0.75)	2.57 (0.95)	0.80 (0.18)	0.81 (0.22)
Hurd	10.72 (1.55)	10.21 (1.54)	1.92 (0.48)	1.74 (0.20)
Shive	11.70 (1.82)	9.57 (1.73)	2.36 (0.27)	2.00 (0.26)
Wood*	4.07 (1.54)		1.06 (0.29)	
Hurd*	12.40 (1.83)		2.09 (0.28)	
Shive*	10.01 (2.32)		2.14 (0.32)	
ANSI LD2	5.00		1.03	
ANSI M2	14.50		2.25	

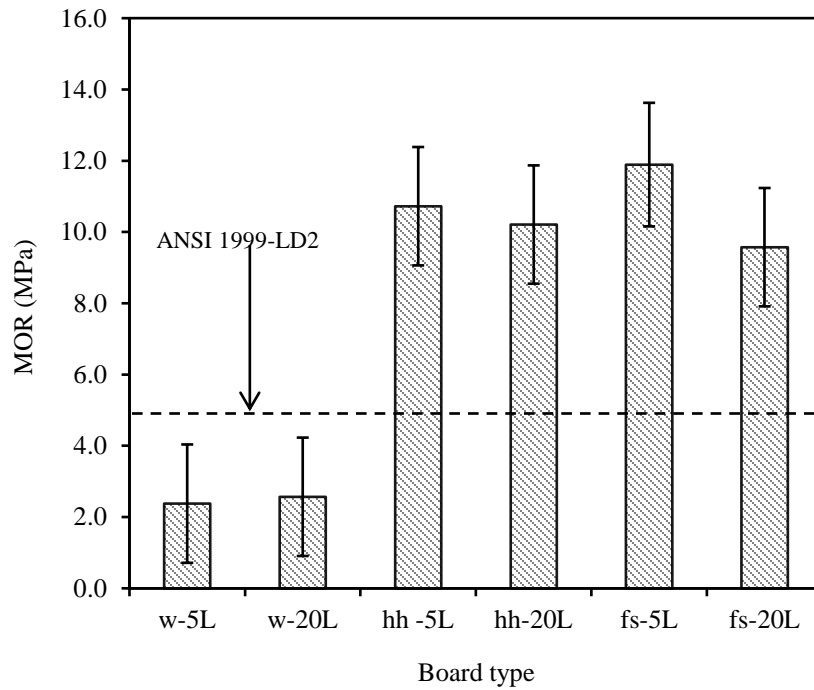


Figure 5.16: Bending strength properties of pMDI-lignin particleboards; 5% lignin (5L), 20% lignin (20L), w (wood), hh (hemp hurd), fs (flax shive). Horizontal line indicates minimum value stipulated by ANSI A208.1-1999 for LD2 grade particleboard. $n=12$ for each mean. Error bars represent 95% confidence intervals.

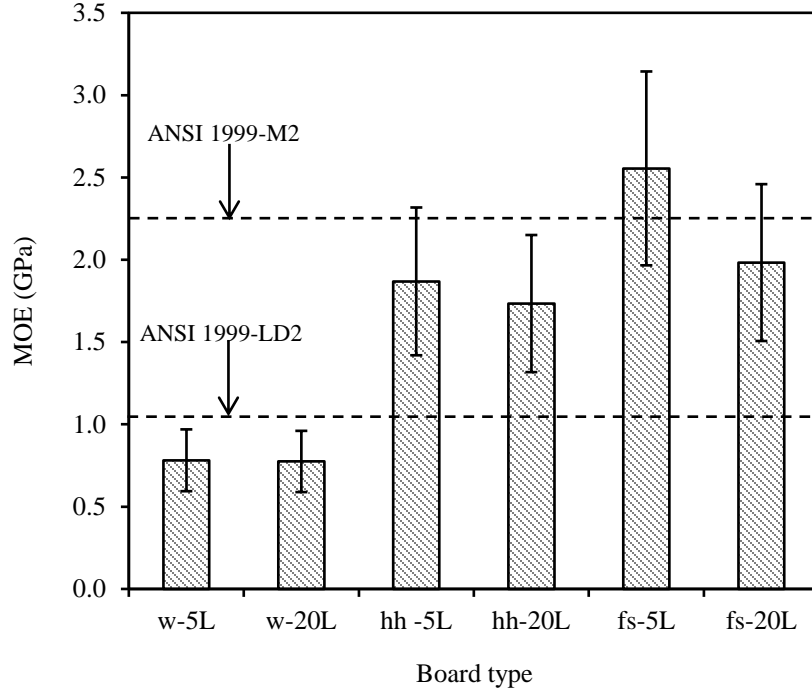


Figure 5.17: Bending stiffness properties of pMDI-lignin particleboards; 5% lignin (5L), 20% lignin (20L), w (wood), hh (hemp hurd), fs (flax shive). Horizontal line indicates minimum value stipulated by ANSI A208.1-1999 for LD2 and M2 grade particleboards. $n=12$ for each mean. Error bars represent 95% confidence intervals.

A comparison of static bending properties between particleboards with and without lignin (Figure 5.18) indicates that the control particleboards without lignin achieved higher values than those with lignin incorporated; the flax shive particleboards with 5 weight % lignin substitution being an exception. A general trend is observed in keeping with the DSC reaction heat results where particleboards manufactured with the greater 20 weight % lignin substitution attained the lowest MOR and MOE values, with exception of the wood particleboards where the 20 weight % lignin substitution yielded a slight non-significant increase in MOR compared to the 5 weight % lignin substitution (Appendix D).

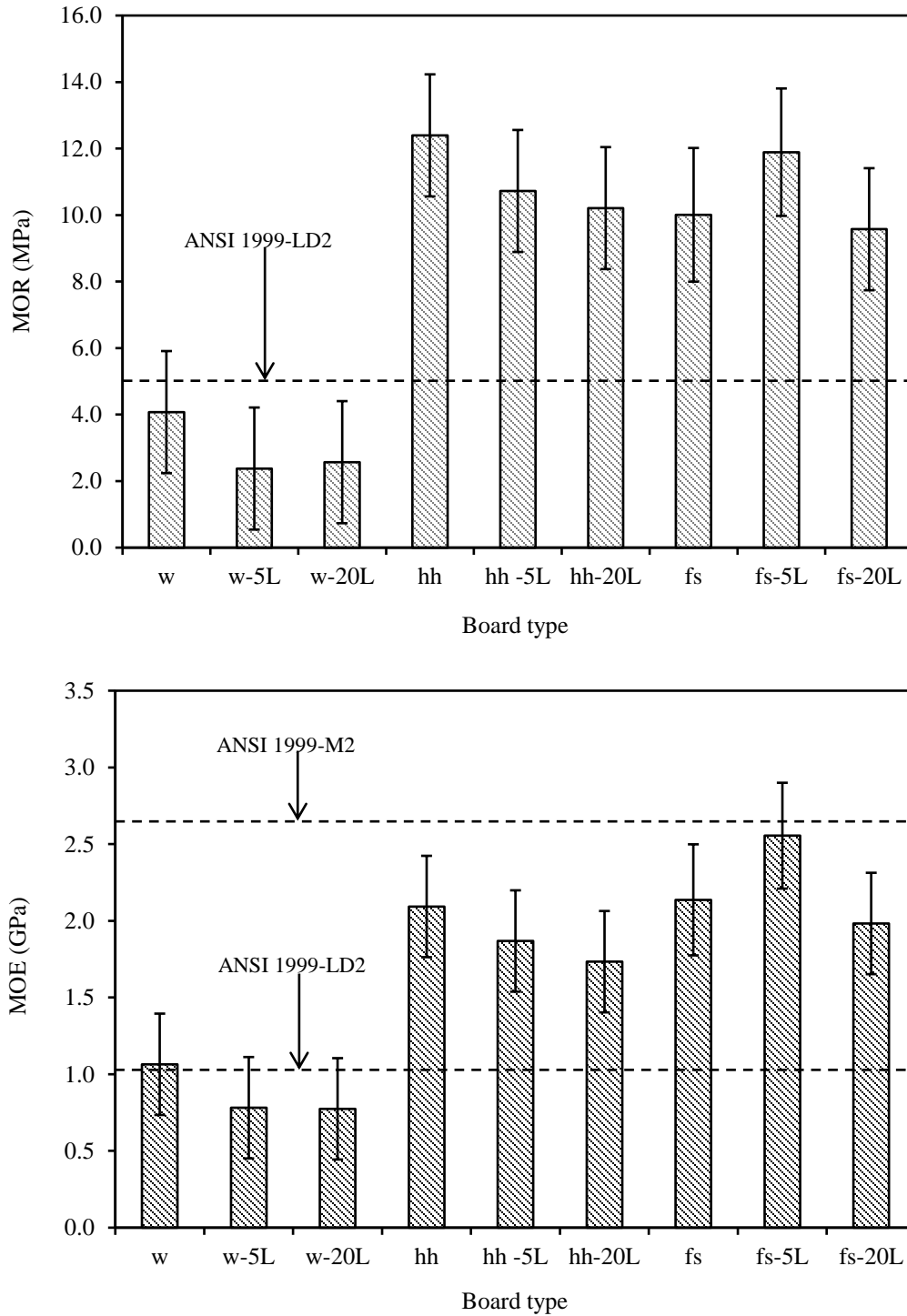


Figure 5.18: Comparison of the bending strength and stiffness properties of pMDI and pMDI-lignin particleboards; 5% lignin (5L), 20% lignin (20L), w (wood), hh (hemp hurd), fs (flax shive). Horizontal line indicates minimum value stipulated by ANSI A208.1-1999 for LD2 and M2 grade particleboard. $n=12$ for each mean. Error bars represent 95% confidence intervals.

The findings of the current study agree with those of Senyo et al. (1999) and Cetin and Ozmen (2002b). Senyo et al. (1999) studied the formaldehyde emissions from particleboards manufactured with a combination of 20% low molecular weight organosolv lignin (unmodified and methylolated) and 80% PF resin. Results of their study revealed a decrease in MOR values for the 19 mm thick particleboards ($772\text{--}800\text{ kg/m}^3$) compared to the pure PF control panels. A similar trend was observed by Cetin and Ozmen (2002b) for 12 mm thick particleboards (650 kg/m^3) bonded with PF resin which exhibited higher MOR values when compared to boards bonded with 20% and 40% unmodified organosolv lignin-PF resins; in contrast the MOE values were not affected by the addition of lignin.

5.3.4 Physical properties

5.3.4.1 LE

Figure 5.19 presents the linear expansion values between 50% and 90% relative humidity at $20 \pm 3\text{ }^\circ\text{C}$ for particleboards bonded with pMDI-lignin resin. For each particleboard type no significant difference was observed between samples obtained parallel and perpendicular to the forming direction of the board, thus the results presented are representative of the pooled data from both directions. Significant differences ($p < 0.0001$) were however observed between the particleboards based on residue type and lignin substitution (Appendix D). The wood particleboards yielded the highest LE values and the flax shive particleboards the lowest. Boards with 20 weight % lignin substitution consistently exhibited higher linear expansion than those with 5 weight % lignin; this difference was only significant in the flax shive and hemp hurd boards.

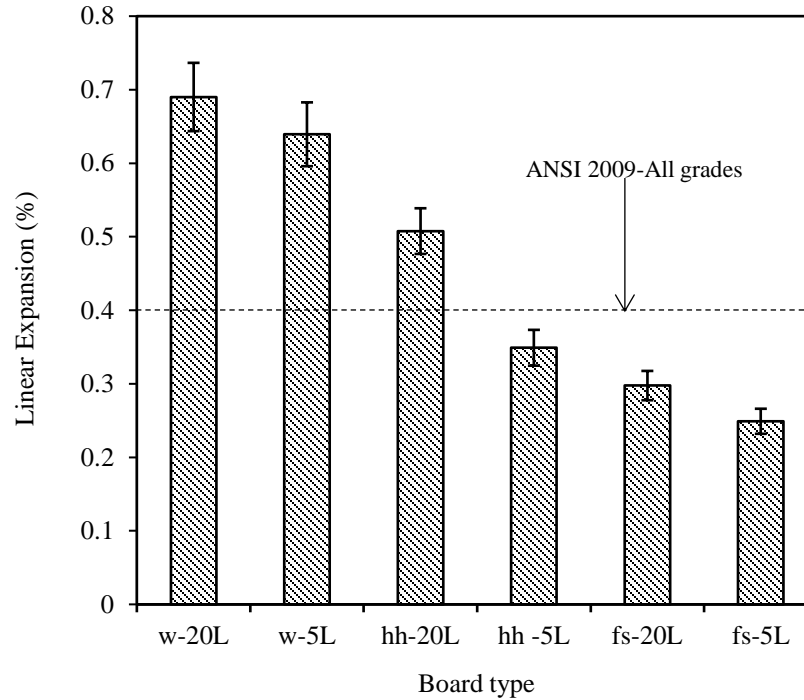


Figure 5.19: Linear expansion of samples subjected to increasing relative humidity from 50% to 90%; 5% lignin (5L), 20% lignin (20L), w (wood), hh (hemp hurd), fs (flax shive). Horizontal line indicates maximum value stipulated by ANSI A208.1-2009. $n=16$ for each mean. Error bars represent 95% confidence intervals.

As was observed in the control particleboards bonded with 2.5% pMDI resin load (section 3.3.6), the lowest LE values were attained by the flax shive particleboards, followed by the hemp hurd particleboards and finally the highest LE observed in the wood particleboards. The flax shive boards manufactured with both 5 and 20 weight % lignin substitution and the hemp hurd board with 5 weight % lignin substitution fell below the maximum LE value stipulated in ANSI A208.1-2009 for all particleboard grades. These LE results observed here are not likely the consequence of board density as it would have meant the hemp hurd boards with the lowest average board density (section 5.3.1) would have attained the lowest linear expansion. Instead the results are more dependent on the differences in particle geometry and possible arrangement during particleboard manufacture. Residues with longer lengths and likely greater longitudinal sections resulted in less changes in length with increases in moisture from the 50% to 90% relative humidity.

A comparison of the LE values for particleboards manufactured with the 2.5% pMDI resin (control particleboards) and the pMDI-lignin particleboards is provided in Figure 5.20. From the figure, it is evident that increasing the lignin content results in a simultaneous increase in linear expansion. The only exceptions being the 5 weight % lignin substitutions in the flax shive and hemp hurd boards where LE values were less than the control boards, though not significantly so and remains unexplained. The problem of increased linear expansion in the presence of increasing lignin content just like the IB results is linked to the decreasing bond formation between the resin molecules. The function of the resin in the composite is to intimately connect the residues and help transfer stresses that are applied among the residues. Once the curing of resin is compromised the adhesive bond between the resin and the residue is not effective. This allows moisture to easily infiltrate the particleboards and further weaken the adhesive bonds. This seems to be the case with the greater changes in length being observed within the residue types as lignin content increases.

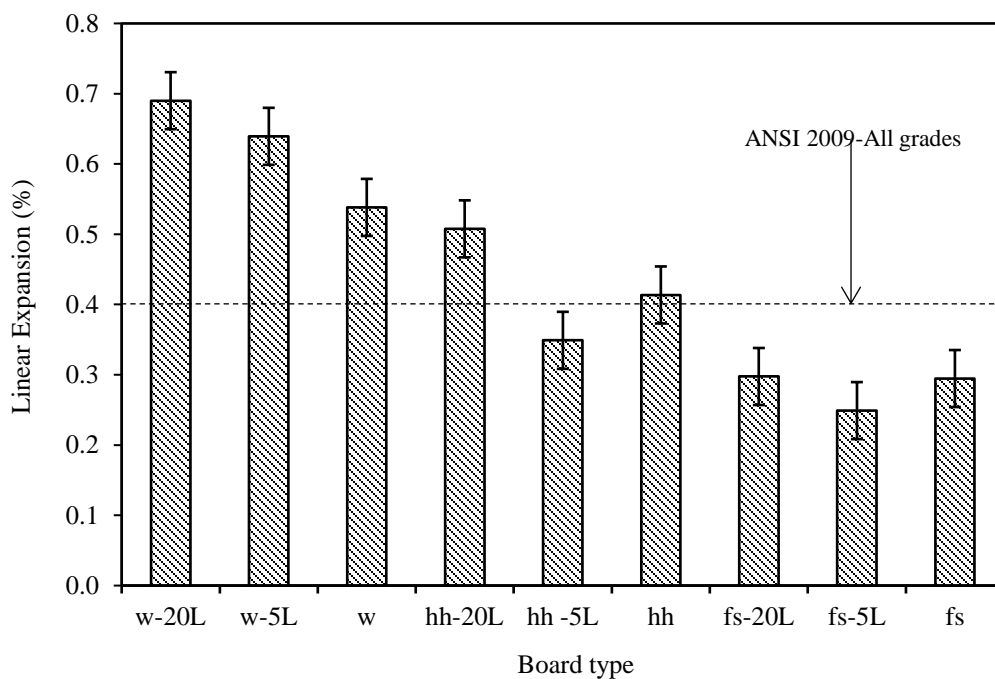


Figure 5.20: Comparison of the linear expansion values of pMDI and pMDI-lignin particleboards; 5% lignin (5L), 20% lignin (20L), w (wood), hh (hemp hurd), fs (flax shive). Horizontal line indicates maximum value stipulated by ANSI A208.1-2009. n=16 for each mean. Error bars represent 95% confidence intervals.

5.3.4.2 TS and WA

The 2 and 24 hours thickness swell (TS) and water absorption (WA) characteristics of the pMDI-lignin particleboards (Figure 5.21) were significantly different ($p < 0.0001$) from each other based on both residue type and lignin substitution levels. The highest swell in thickness and corresponding water absorption for both the 2 and 24 hours submersion was observed in the hemp hurd boards. In terms of lignin content, particleboards manufactured with the higher 20 weight % lignin substitution in pMDI resin yielded significantly higher TS and WA values; the wood particleboards were the only exception where the difference between 5 and 20 weight % lignin substitution was not significant.

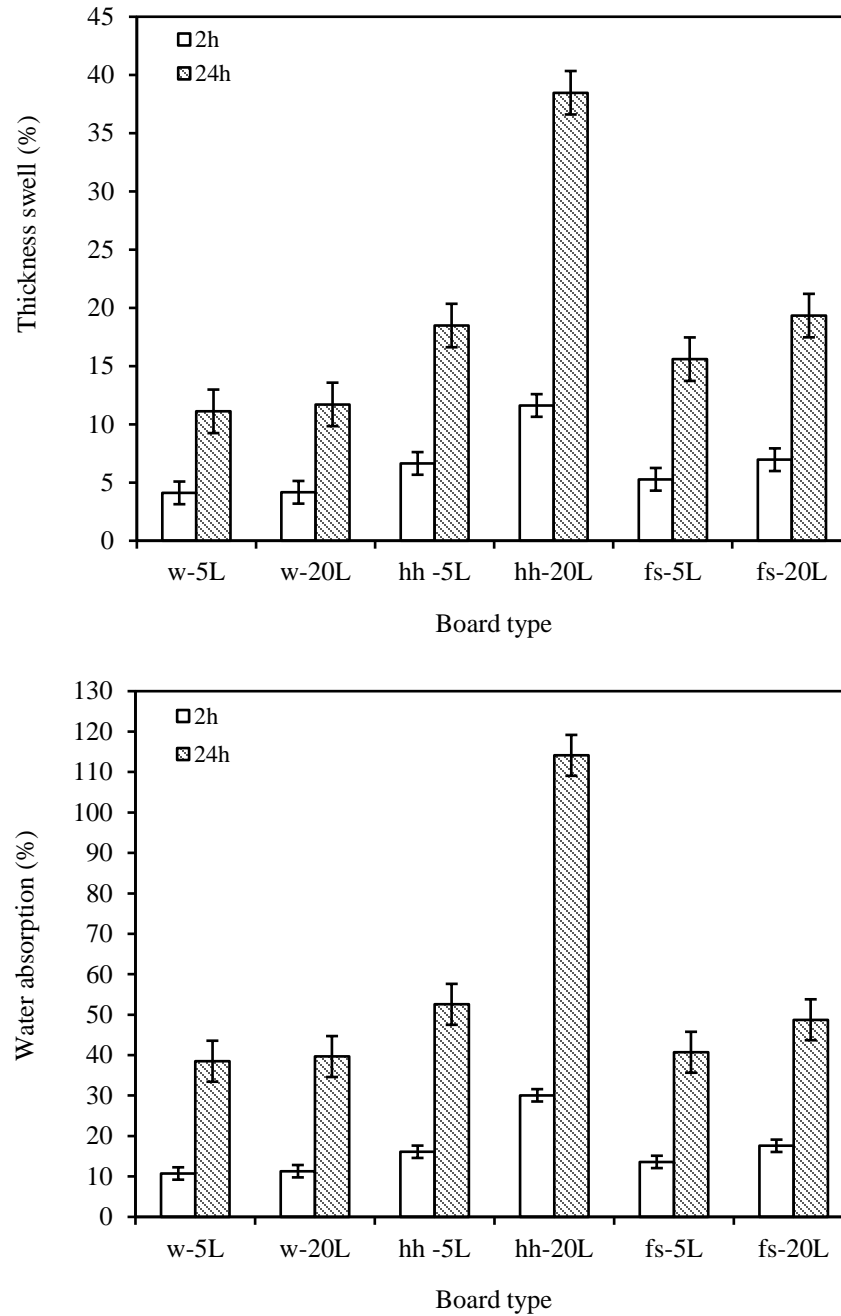


Figure 5.21: Thickness swell and water absorption properties of particleboards manufactured with pMDI-lignin resin; 5% lignin (5L), 20% lignin (20L), w (wood), hh (hemp hurd), fs (flax shive). $n=12$ for each mean. Error bars represent 95% confidence intervals.

Comparing the results of the particleboards containing lignin with the control particleboards manufactured from 2.5% pMDI resin load (Figure 5.22), it is evident that increases in the lignin content resulted in an equivalent increase in thickness swell and water absorbed with exception of the flax shive boards bonded with 5 weight % lignin substitution where a non-significant reduction was observed. The trend observed in the TS results with lignin content are consistent with reports by Cetin and Ozmen (2002b) from their study on particleboard fabricated with both unmodified lignin (20% and 40% substitution) and phenolated lignin (20%, 25% and 30% substitutions) in combination with phenol formaldehyde resin. The high water absorption capacity of the hemp hurd and flax shive residues which has previously been discussed is also evident as this factor too contributed to the negative impact that increasing lignin content had on these particleboards in contrast to the wood particleboards.

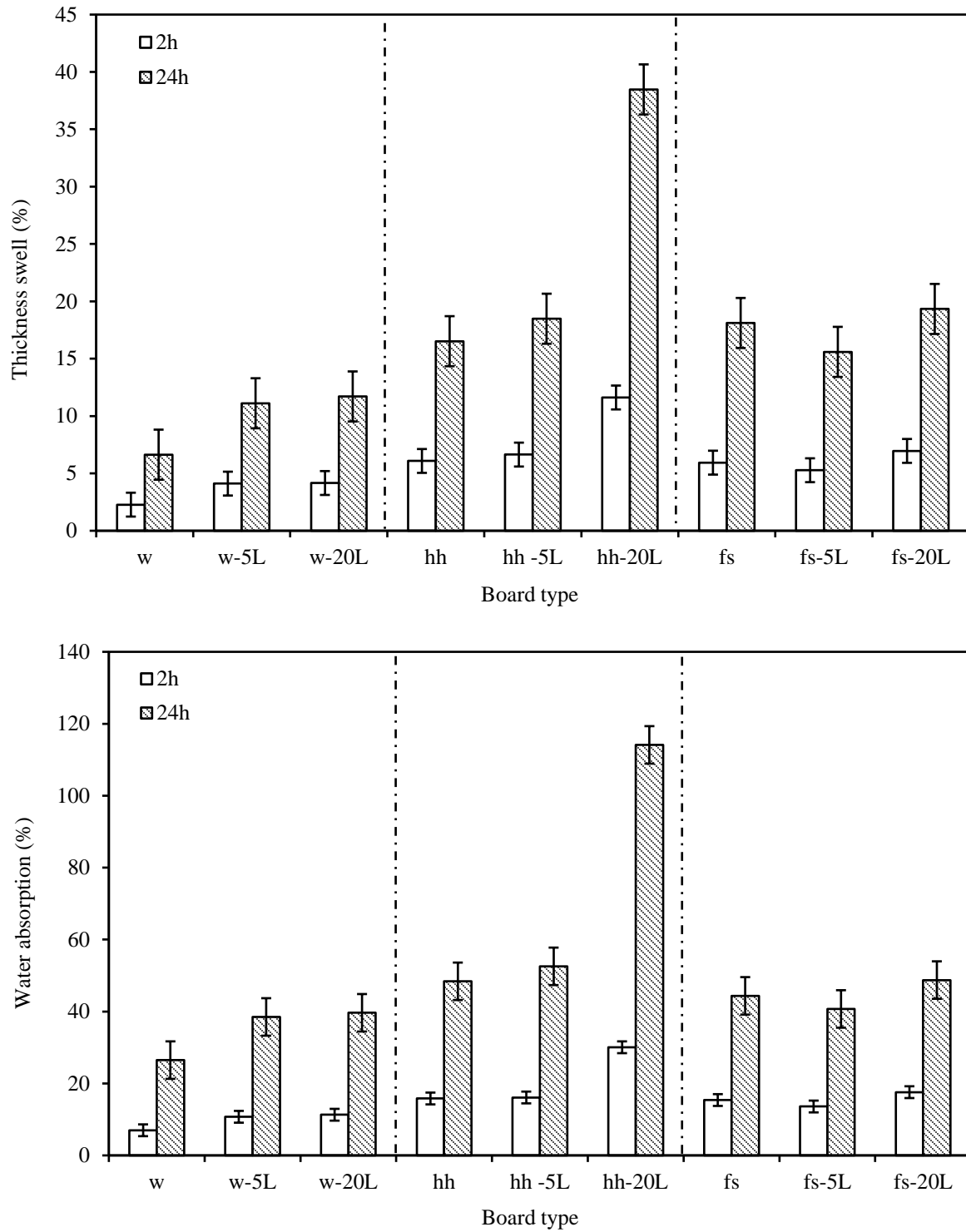


Figure 5.22: Comparison of the thickness swell and water absorption characteristics of pMDI and pMDI-lignin based particleboards; 5% lignin (5L), 20% lignin (20L), w (wood), hh (hemp hurd), fs (flax shive). $n=12$ for each mean. Error bars represent 95% confidence intervals.

5.4 Conclusions

Lignin, a naturally abundant phenolic binder in plants, has been researched for decades as a polymer that can be used to replace mass fractions of more expensive synthetic-based thermosetting adhesives such as urea and phenol formaldehyde. In this study 5 and 20 weight % of fast curing pMDI resin was substituted with unmodified Kraft lignin, and the adhesive formulation subsequently used for the manufacture of particleboard from wood, hemp hurd and flax shive residues. A low resin load of 2.5% pMDI was used in keeping with the objective of reducing resin cost.

Results of the dynamic scanning calorimetry of pMDI-lignin resins at the different lignin substitution levels (5%, 10% and 20 weight %) in combination with wood, hemp hurd and flax shive residues revealed that in terms of the type of residue, the onset temperatures of resin curing were consistently lower in the wood samples and highest in the flax shive samples, indicating a greater reactivity of the pMDI-lignin resin in the presence of the wood samples. The opposite was observed with the reaction heat values where the highest degree of resin cure was detected in the pMDI-lignin resins in combination with the flax shive residues. With increasing lignin content from 5 to 20 weight % a general decrease in onset and peak temperatures was observed, signifying an early start of curing but ultimately the reaction heat values were lowered suggesting lower degrees of resin cure.

The internal bond strength of the low density 521-556 kg/m³ particleboards manufactured with pMDI-lignin resins exceeded the minimum ANSI A208.1-1999 requirements for LD2 grade particleboard and medium density M2 grade particleboard. But in comparison to boards manufactured with only 2.5% pMDI resin, particleboards with lignin displayed significantly lower bond strength with increasing lignin content. This decrease in board properties with increasing lignin substitution from 5 to 20 weight % was also observed in the bending strength and stiffness, linear expansion, water absorption and thickness swell properties, and was in accordance with the reaction heat results observed in the DSC tests.

The hemp hurd and flax shive particleboards at both 5 and 20 weight % lignin substitution levels yielded significantly higher MOR and MOE values which exceeded the ANSI A208.1-1999 LD2 mechanical strength requirements; an approximate increase of 73% in MOR and 60% in MOE

above that of the wood particleboards. With exception of the flax shive particleboards manufactured with 5 weight % lignin substitution, no significant differences were observed in static bending properties between the 5 and 20 weight % lignin substitution levels for the wood and hemp hurd particleboards.

The linear expansion values for both 5% and 20 weight % lignin substitution in the flax shive boards as well as the 5 weight % lignin substitution in the hemp hurd boards were the only ones within the acceptable limits specified in ANSI A208.1-2009. The thickness swell and water absorption properties were negatively affected by the addition of lignin and were significantly higher in the hemp hurd particleboards.

Overall, the findings of this study show that the addition of lignin at 5 and 20 weight % substitution in 2.5% pMDI resin results in lower physical and mechanical board properties. The results point to the fact that the Kraft lignin does not react with the pMDI resin as was expected to create boards with better performance. Notwithstanding the resulting low density flax shive and hemp hurd particleboards produced passed the minimum ANSI low density LD2 particleboard requirements for internal bond, static bending and linear expansion- all of which are basic properties of importance when considering the use of particleboard for furniture production. Most interesting is the fact that despite the general lower board properties observed, the adhesive bond strength of the pMDI-lignin particleboards exceeded the minimum requirement for medium density M2 grade particleboard though they were manufactured to a low density ($<620 \text{ kg/m}^3$). Suggesting that particleboards can be manufactured using low pMDI quantities (as low as 2.5% resin load) with as much as 20 weight % Kraft lignin substitution to obtain particleboards of comparable performance to those manufactured with 2.5% pMDI resin load only.

6. Flax and hemp fiber-reinforced particleboard¹

The study presented in this chapter is based on efforts to improve the mechanical strength properties of particleboards using natural bast fibers as reinforcement material. The chapter covers an introduction to the use of non-wood residues and other materials to enhance particleboard strength properties and contains a detailed design of experiment explaining the factors and effects of interest that were studied. Also included is a discussion section on the test results obtained from short-term testing of the fiber-reinforced particleboards and evaluation of the properties against ANSI particleboards standards. The chapter ends with an overview of the major outcomes of the study and its implications for particleboard manufacturers.

6.1 Background

The use of non-wood residues as supplements or as direct substitutes for wood in wood composites (particleboard, medium density fiber boards, hardboards, OSB) has been and continues to be a subject for research and development. Several studies have been conducted over the years to ascertain the viability of using a wide variety of agricultural crop and plant residues for the manufacture of particleboard (Youngquist et al. 1994). Some of these include the use of maize husk and cob (Sampathrajan et al. 1992), reed (Han et al. 1998), wheat straw (Mo et al. 2003), bamboo (Papadopoulos et al. 2004), cotton carpels (Alma et al. 2005), kenaf (Kalaycioglu and Nemli 2006), hazelnut husk (Cöprü et al. 2007), eggplant stalks (Guntekin and Karaku, 2008), bagasse (Xu et al. 2009), oil palm fronds, leaves and trunks (Hashim et al. 2011), walnut shell (Pirayesh et al. 2012), rice straw and coir fiber (Zhang and Hu 2014). In most cases the harvested portions (stalks, leaves, fruits or seeds) were dried, chopped and milled, sieved into face and core fractions, mixed with urea or phenol formaldehyde resin and formed into mats which were then hot pressed into panels. The resulting board properties were acceptable in some studies and in others were reported to almost meet the minimum required standards. In other studies, admixtures of the waste residues and industrial wood particles were used and panels were reported to exhibit

¹ A version of this chapter has been published. Sam-Brew, S., and Smith, G.D. (2015). Flax and Hemp fiber-reinforced particleboard. *Ind. Crop. Prod.* 77, 940-948. <http://dx.doi.org/10.1016/j.indcrop.2015.09.079>

improved strength compared to boards made from 100% agricultural crop and plant residues (Kuo et al. 1998; Grigoriou, 2000; Ntalos and Grigoriou 2002; Nemli et al. 2003; Bektas et al. 2005; Guler et al. 2008 and Kibria 2012).

Using a wide range of reinforcing materials, attempts have been made over the years to enhance the strength properties of wood composite products. Fitzgerald et al. (1992) considered a symmetrical lattice of wooden strips in particleboard core and Mura and Mura (2001) proposed synthetic fabric sheets (glass fiber, carbon fiber, kevlar) inserted at 1/3 of the board thickness. Reinforcements have also been considered for plywood, oriented strand board and laminated veneer lumber using cords of metal, plastic and rubber (Dimakis et al. 2006). Glass fiber, carbon fiber and metal/woven synthetic nets embedded at 1/4 of the board thickness have also been considered in medium density fiberboards (Malcom 1992, Cai 2006 and Mohebbi et al. 2011). The reinforcements have been reported to improve the load carrying capacity and impact strength properties of the wood products. But the use of expensive synthetic fibers or materials increases the production cost for panel manufacturers and presents disposal problems.

This study attempts to use natural bast fibers (flax and hemp fibers) with high tensile properties as reinforcements in particleboard panels. Flax (*Linum usitatissimum*) and hemp (*Cannabis sativa* L.) are commercially important fibers that have been cultivated for centuries and used in the production of clothing, nets, industrial and marine ropes (Batra 2007). To obtain the long fiber portions, the harvested flax or hemp stalks are degraded and taken through a series of mechanical breaking processes to loosen the fibers from the inner woody core. This waste woody portion commonly referred to as the flax shive or hemp hurd are typically used for animal beddings; some studies have also considered these by-products for particleboard production (Hague, 1998; Theis and Grohe, 2002; Papadopoulos and Hague, 2003; Balducci et al., 2008; Osman et al., 2009; Nikvash et al., 2010 and Lühr et al., 2013).

The mechanical strength properties of the flax and hemp fiber (Table 6.1) show that they have high strength-to-weight ratios and their stiffness values (though a wide range) match or exceed those of wood, wood products and E type glass fiber (Wumba et al. 2003; Sapuan et al. 2006; Ashori 2008).

Table 6.1: Mechanical properties of some fiber materials.

Fiber	Density (g/cm³)	Tensile strength (MPa)	Youngs modulus/E (Gpa)	Specific strength (kNm/kg)	Specific stiffness (MNm/kg)
Pine (Lodgepole)	0.45	76	10.9	169	24.3
Softwood (kraft)	1.5	1000	40	667	26.7
Flax	1.5	800-1500	60-80	533-1000	40-53
Hemp	1.47	550-900	70	374-612	47.6
E-glass	2.55	2400-2500	73	941-1333	28.6

Source: Bismarck et al. 2006; Anandjiwala and Blouw 2007; Ghosh et al 2007; Green et al. 2007

Anatomically the flax and hemp fiber bundles consist of a complex assembly of technical fibers and elementary fibers bonded together by a strong pectin and lignin interface (Baley 2002; Bos et al. 2002). The fibers are polygonal in cross-section with a lumen surrounded by a primary and secondary cell wall (Batra 2007). The thin primary cell wall is composed of a random network of crystalline cellulose microfibrils and the larger secondary cell wall which consists of 3 separate layers is made up of spirally arranged crystalline cellulose microfibrils; the cellulose microfibrils are embedded in an amorphous matrix of hemicellulose and lignin (Bos et al. 2002; Zimmermann et al. 2004; Thomas et al. 2011). The cellulose microfibrils are made up of long chains of cellulose molecules which contribute to the mechanical strength of the fiber (refer to Figure 1.22) (Mwaikambo and Ansell 2002).

Generally, fiber strength and stiffness is greatest along the fiber axis or longitudinal direction where lays the highly oriented crystalline cellulose structure (Bos et al., 2002). The work presented here involves the production of a 3-layered particleboard reinforced with aligned flax and hemp fiber mats in the upper and lower panel surfaces as close as possible to the points corresponding to the maximum compressive and tensile stresses respectively (Figure 6.1). The factor of interest is the effect of the fiber type (flax or hemp) as reinforcement on particleboard strength properties. The study also makes use of the woody core waste materials – flax shive and hemp hurd - that are obtained as by-products from the flax and hemp fiber extraction process for particleboard manufacture.

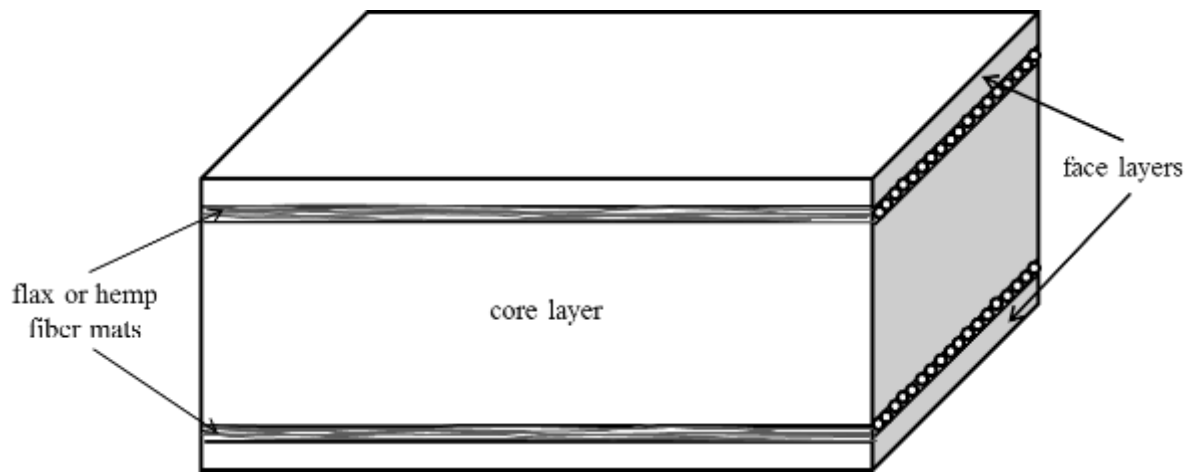


Figure 6.1: Particleboard reinforced with continuous layers of flax or hemp fiber. Source: Sam-Brew 2016.

6.2 Experimental design

The main factor of interest for this study was the effect of the fiber reinforcement (flax or hemp fiber) on particleboard strength properties. As stated in section 2.1.3, the current cost of flax and hemp fiber is approximately \$1.20/kg far more expensive than wood residues which cost \$50/ bone dry ton (2000 lbs wood at 0% moisture content). This study is not aimed at incorporating the flax and hemp fiber to substitute the wood residue in particleboard production. Instead the objective is to evaluate the feasibility of including the flax and hemp fiber to serve as reinforcements and to help address the creep deformation often observed in particleboard products in service. Therefore, the flax and hemp fibers made up only 15 weight % of the whole particleboard.

Accordingly, 3-layered particleboards comprising 50% core furnish, 35% face furnish and 15% flax or hemp fiber mats were manufactured from wood, hemp hurd and flax shive residues according to the experimental design outlined in Table 6.2. The wood particleboard was used in conjunction with both flax and hemp fiber while the flax shive and hemp hurd were combined with the fibers from which they were derived, i.e., flax and hemp respectively. For each particleboard type 4 replicates were manufactured for a total of 16 particleboards. In accordance with ASTM D1348-94, the moisture content of the raw materials prior to board manufacture was determined

to be 11% for the flax fiber, 9.5% for the hemp fiber, 8% for the wood residue, and 9.8% for the hemp hurd and flax shive residues.

Table 6.2: Design of experiment for 3-layered fiber-reinforced particleboard.

	Factors	Levels	Response	Total specimens
Variables	Residue-fiber type	wood-flax	MOR/MOE	14
		wood-hemp	IB	48
		hemp hurd-hemp	TS	11
		flax shive-flax		
Constants	Density (kg/m ³)	620		
	Thickness (mm)	12.7		
	Resin type	pMDI		
	Resin load (%)	5-6%		
	Replicates	4		

Consistent with the manufacturing sequence described in section 2.2.3, the face and core furnish were blended with 5% pMDI resin (based on furnish oven dry weight), and both surfaces of the aligned flax and hemp fiber mats were hand sprayed with 5–6% pMDI resin load. To allow smooth resin flow and penetration into the fiber mats the viscosity of the pMDI resin was lowered by mixing with acetone at a ratio of 1:1. After spraying the fiber mats were left for 15–20 minutes to allow evaporation of the acetone. In line with particleboard manufacturing technique, mats were formed through a layering process—face furnish—fiber layer—core furnish—fiber layer—face furnish as shown in Figure 6.2. The lower face furnish was first uniformly spread in the forming box and prepressed. The sprayed fiber mats were then evenly laid unto the face with 1mm overlap along fiber edges. Next the resinated core particles were spread on the aligned fibers and prepressed. Afterwards the upper fiber layers were carefully placed unto the core furnish and evenly covered with the upper face furnish. The formed mat was prepressed and subsequently pressed according to section 2.2.3.



Figure 6.2: Three-layered particleboard mat comprising of flax shive residues and flax fiber.
Source: Sam-Brew 2015.

Preliminary boards were first manufactured to help identify the minimum amount of face furnish that would provide a smooth surface for the particleboard and simultaneously ensure fiber mats were located as practical as possible at the points of maximum stresses. Trial boards manufactured with less than 35% face furnish had dry and flaky face layers. Failure in these boards during internal bond tests were predominantly in the upper and lower residue face layers. Sanding to remove approximately 1 mm of material from both faces resulted in boards with higher bond strength that failed in the core or fiber layer. Thus, material mass for the face furnish was doubled (to ensure greater compaction between particles), the press cycle maintained for a target density of 620 kg/m^3 and afterwards approximately a total of 2 mm material sanded off both face layers.

6.3 Results and discussion

6.3.1 Moisture content, average board density and vertical density profile

Table 6.3 below lists the average board density and moisture content (MC) of the different fiber-reinforced particleboard types. No significant differences were observed between the particleboard types in terms of density or MC (Appendix E).

Table 6.3: Average board density and moisture content of fiber reinforced particleboards. n=11 for each mean. Values in parenthesis are standard deviations.

	wood-flax	wood-hemp	hemp hurd-hemp	flax shive-flax
Density (kg/m ³)	775.31 (25.35)	764.05 (39.02)	765.86 (25.52)	794.17 (39.20)
MC (%)	9.56 (0.09)	9.76 (0.11)	9.70 (0.32)	9.58 (2.63)

The vertical density profile (VDP) through the thickness of the boards indicated peak density areas corresponding to the densely compacted face layers (F1, F2) and sections where the flax and hemp fibers were inserted during board production (Figure 6.3). Relatively higher peaks were observed for particleboards with the rigid hemp fiber than boards with the softer more flexible flax fibers. The higher density peaks for the fiber section is attributed to the fact that the natural density of the flax and hemp fibers (approximately 1.5 g/cm³) are much higher than that of the wood, hemp hurd and flax shive particles, as such more compaction is needed to compress the fibers above their natural density. Since the particles in the face are in direct contact with the heated press platen, they are readily plasticized and quickly densified. With time through heat transfer the core layer is also gradually compressed and soon the board reaches its target thickness and density before the flax and hemp fibers can be compressed above their natural density.

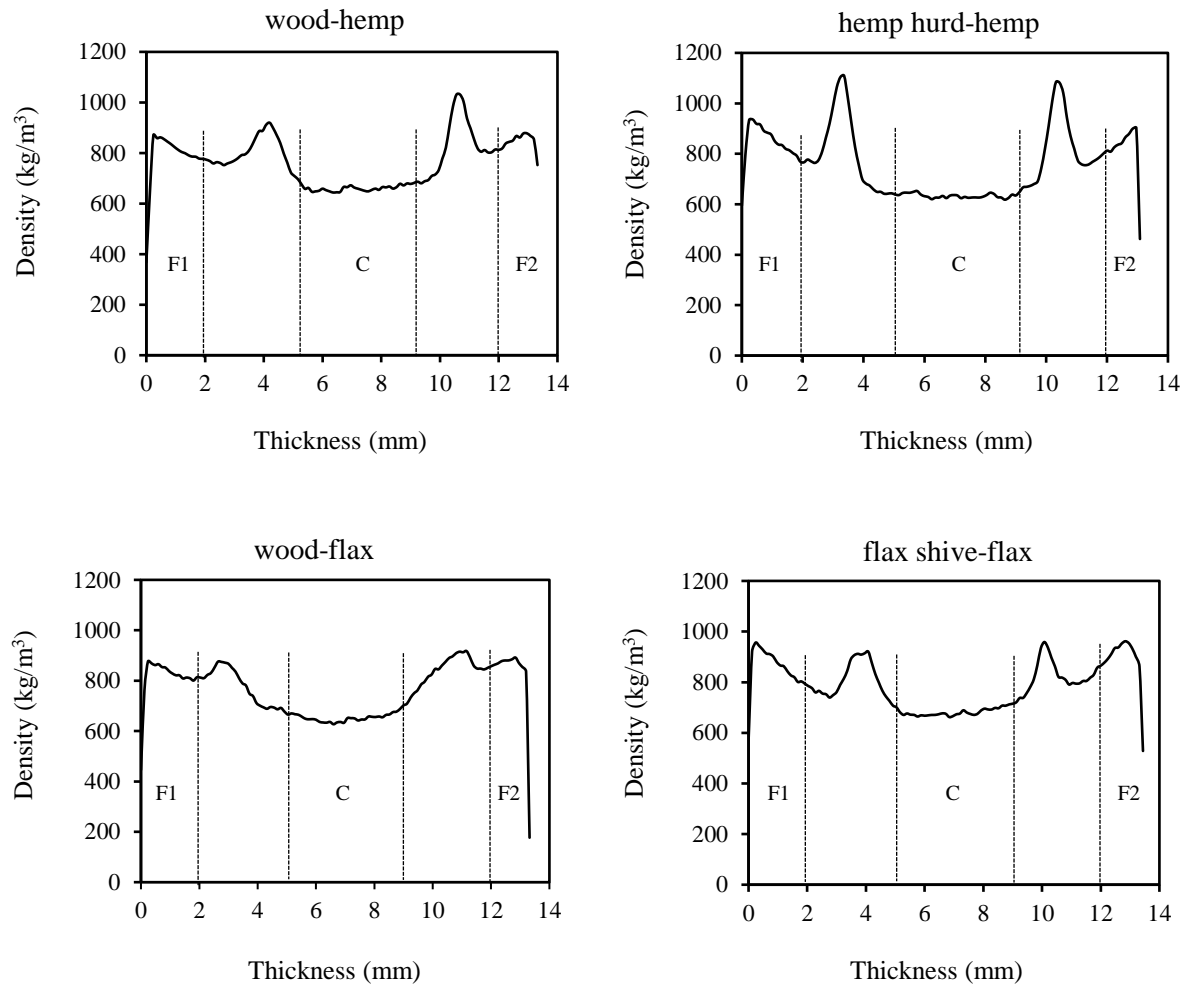


Figure 6.3: Comparison of vertical density profile through fiber-reinforced particleboard. Vertical lines indicate fiber-reinforcement zones).

The core densities (C) ranged from 648-684 kg/m³ and were not statistically significant from each other (Figure 6.4). Within each particleboard type no differences were observed between peak face densities, i.e., F1 and F2, though uneven face densities were observed in the wood-flax and hemp hurd-hemp particleboards due to unequal sanding of the faces. When the average peak face densities were compared significant differences ($p=0.0061$) were noted based on the type of residue used in board manufacture; the flax shive-flax and hemp hurd-hemp particleboard face densities were significantly higher compared to the wood-flax and wood-hemp particleboards. This difference in peak face densities can be attributed to the differences in initial MC of the

residues prior to board manufacture where the hemp hurd and flax shive residues had approximately 9.8% moisture and the wood residues 8% moisture.

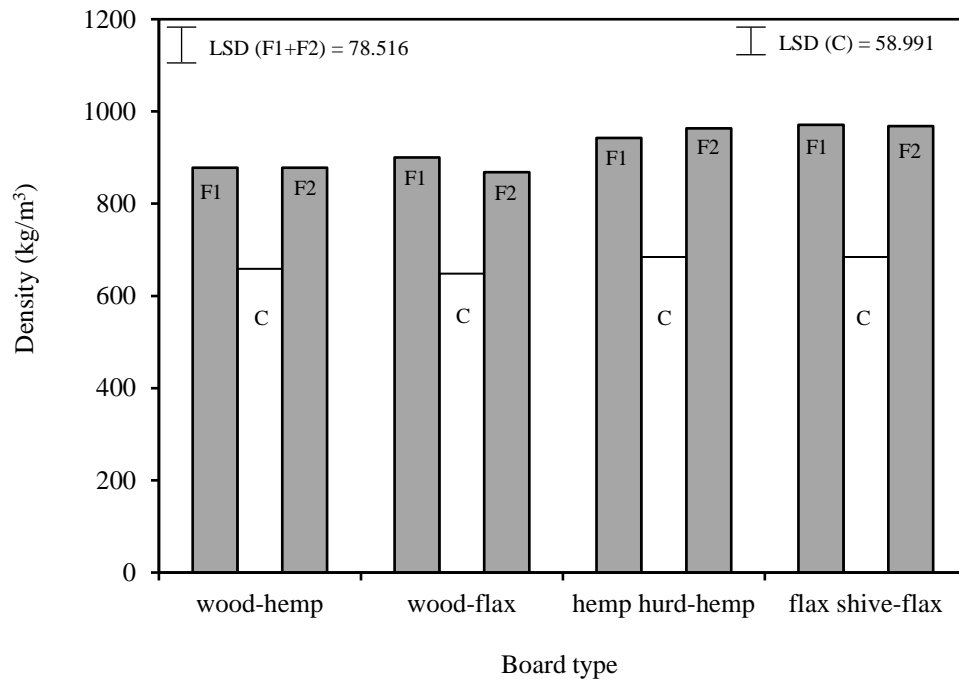


Figure 6.4: Face and core density profile of fiber-reinforced particleboards expressed as peak face (F1, F2) and core (C) densities. $n=6$ for each particleboard mean. Error bars represent least significant difference between means.

6.3.2 Mechanical properties

Results of the mechanical strength properties are presented in Table 6.4. For comparison purposes results from Chapter 3 on particleboards manufactured from 100% wood, hemp hurd and flax shive residues using 5% pMDI resin load to a target density of 620 kg/m^3 are included in the table. It is evident from the table that the densities of the fiber-reinforced particleboards are significantly higher than those of the 100% wood, hemp hurd and flax shive particleboards. To enable an accurate comparison between the mechanical strength properties of these particleboard types, an analysis of covariance (ANCOVA) was carried out to eliminate the effect of variability due to density differences. Where applicable logarithm transformations were applied to ensure that all

analysis met the assumptions regarding the error term (i.e., independent observations, normal distribution and equal variance).

Table 6.4: Mechanical strength properties of 3-layered hemp and flax fiber-reinforced particleboard. Data presented is based on ANSI A208.1-2009 lower 5th percentile panel averages.

Board Type	IB (MPa)	MOR (MPa)	MOE (GPa)	Density (kg/m³)
wood-flax	0.43 (0.38)	13.62 (4.17)	2.72 (0.45)	775.31 (25.35)
wood-hemp	0.54 (0.43)	7.19 (8.39)	2.06 (0.79)	764.05 (39.02)
hemp hurd-hemp	0.39 (0.38)	19.59 (8.48)	2.64 (1.26)	765.86 (25.52)
flax shive-flax	0.68 (0.36)	18.32 (3.89)	4.02 (0.33)	794.17 (39.20)
100% wood	1.07 (0.21)	4.29 (1.56)	1.05 (0.26)	656.11 (30.08)
100% hemp hurd	0.98 (0.10)	13.88 (2.27)	2.12 (0.34)	641.56 (20.86)
100% flax shive	0.88 (0.12)	15.00 (1.83)	2.88 (0.24)	662.26 (30.07)

6.3.2.1 IB

There was a wide variability in the IB test data and some data points were discarded because of specimen failure in the fiber layer immediately load was applied to the sample. To permit an accurate analysis of variance between particleboard types, 32 data points were randomly selected from the acceptable IB data and analysed. A significant difference was observed in IB strength ($p=0.0136$) among the fiber-reinforced particleboards (Appendix E); the bond strength being significantly higher in the flax shive–flax and wood–hemp particleboards, the second owing to several samples which had comparatively higher densities (Figure 6.5). The IB strength of all the board types met the 0.45 MPa ANSI A208.1-1999 requirement for medium density M2 grade particleboard.

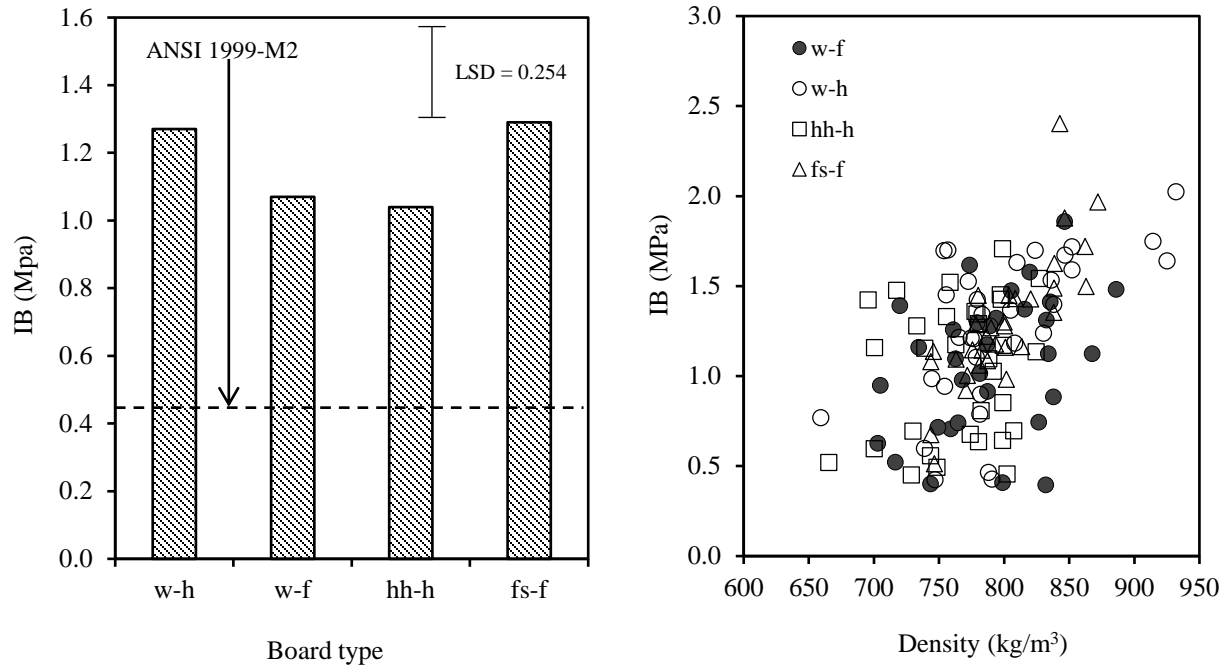


Figure 6.5: Internal bond strength of fiber-reinforced particleboards: wood-hemp (w-h), wood-flax (w-f), hemp hurd-hemp (hh-h) and flax shive-flax (fs-f). Horizontal line indicates minimum value stipulated by ANSI A208.1-1999 for M2 boards. $n=32$ for each mean. Error bars represent least significant difference between means.

In comparison to particleboards made from 100% wood, hemp hurd and flax shive residues the IB strength of the fiber-reinforced particleboards were significantly lower, approximately 30% and 41% for the wood-hemp and wood-flax particleboards ($p<0.0001$), 29% for the hemp hurd-hemp particleboards ($p=0.0087$) and 46% for the flax shive-flax particleboards ($p<0.0001$) (Figure 6.6 and Appendix E). The failure mode of the IB samples was predominantly delamination in the fiber layers. This is clearly seen in Figure 6.7 where the lower flax fiber layer has delaminated, likely a result of poor resin distribution resulting in very weak resin free zones between adjacent fibers.

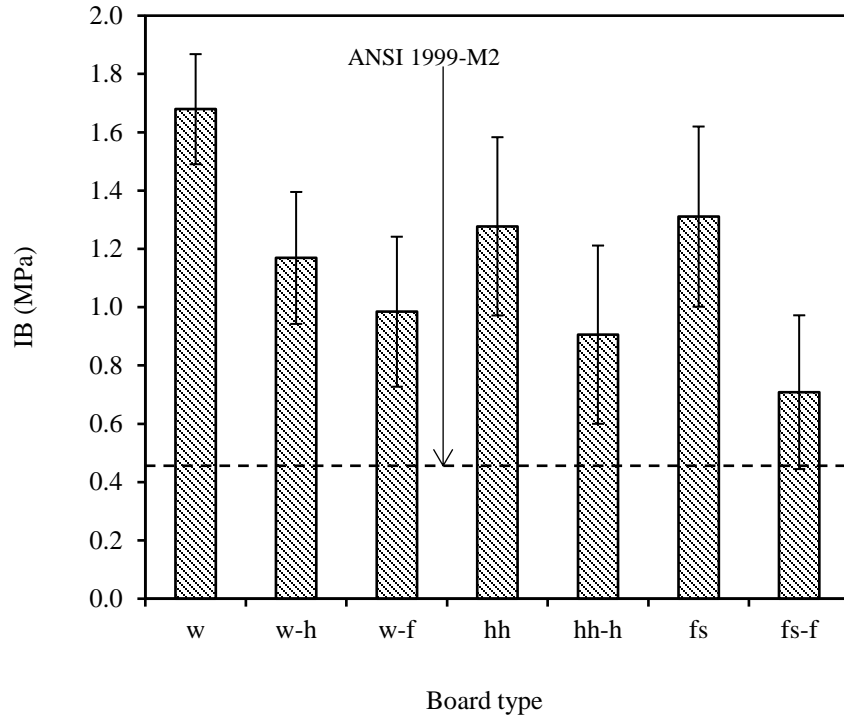


Figure 6.6: Comparison of 100% wood, hemp hurd and flax shive particleboards with flax and hemp fiber-reinforced particleboards: wood (w), wood-hemp (w-h), wood-flax (w-f), hemp hurd (hh), hemp hurd-hemp (hh-h), flax shive (fs) and flax shive-flax (fs-f). $n=32$ for each mean. Error bars represent 95 % confidence intervals.

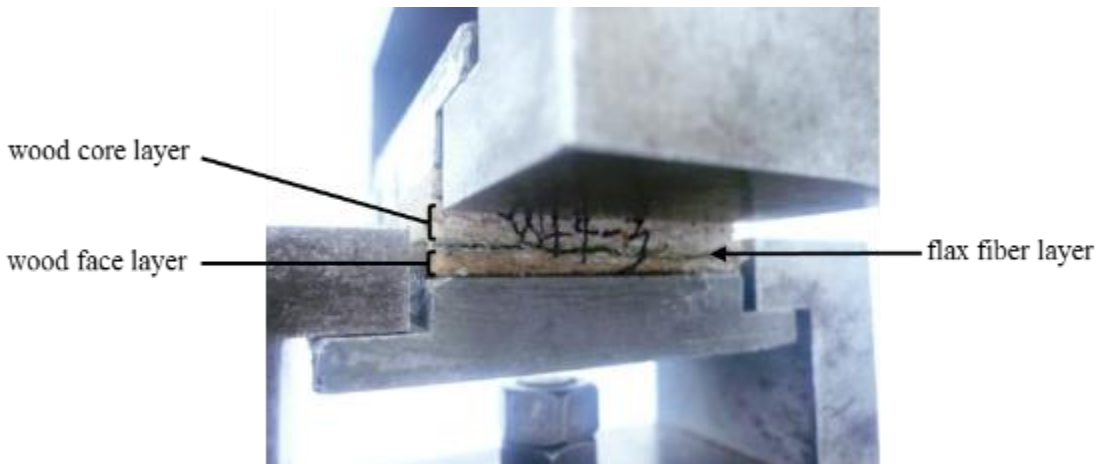


Figure 6.7: Photograph of typical delamination during internal bond testing in the lower flax fiber layer for particleboard composed of wood residues and flax fibers. Source: Sam-Brew 2015.

To confirm this pMDI resin was stained with Toluidine Blue O stain at 0.5 weight % and used for board manufacture in order to visually characterize the resin in the fiber-fiber interface. IB tests were conducted and majority of the failure observed for specimens during testing was delamination in the fiber layers. Using light microscopy the failure sites were examined under a Wild-Leitz M420 Macroscope equipped with a Diagnostic Instruments SPOT-RT digital camera and a Micro Lite FL3000 fiber-optic illuminator. Figure 6.8 shows the fracture surface in the flax and hemp fiber layer of a wood-flax and wood-hemp particleboard after IB testing. It is evident from the figure that there are some fiber areas that do not have resin. Also interesting is the fact that there was a break in a hemp hurd particle contained in the hemp fiber at the fracture site, the particle pair is outlined with black ovals. This break in the hemp hurd particle indicates that in some areas where resin was well distributed the bond strength between the fibers and or particles exceeded that within the particles, hence the fracture observed within the particle.

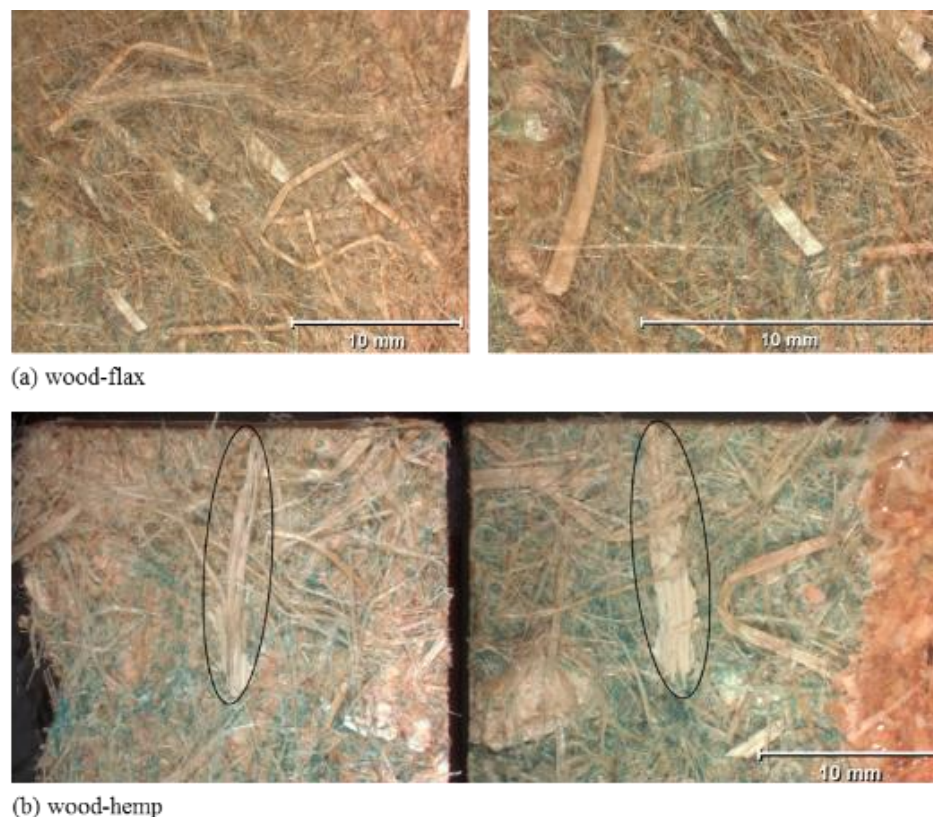


Figure 6.8: Fracture surface of (a) flax fiber layer in a wood-flax particleboard and (b) hemp fiber layer in a wood-hemp particleboard. Source: Sam-Brew 2016.

6.3.2.2 MOR and MOE

Significantly higher bending strength and stiffness properties ($p < 0.0001$) were observed for the hemp hurd–hemp and flax shive–flax particleboards (Appendix E), a trend similar to that observed in the 100% hemp hurd and flax shive particleboards from Chapter 3 (section 3.3.5) and attributed to their higher particle length to thickness ratios which enhanced bending strength and stiffness (Figure 6.9). Comparing these results to the MOR and MOE specifications outlined in ANSI A208.1-1999, all the particleboard types exceeded the minimum values for both low density LD2 and medium density M2 grade particleboard

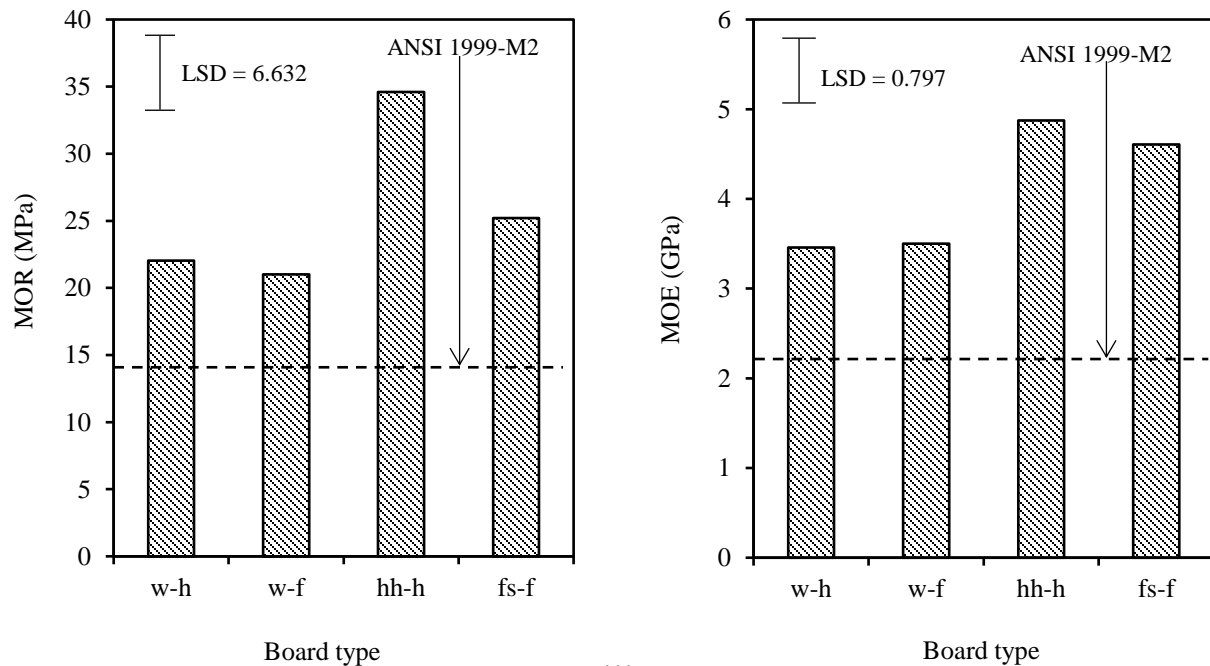


Figure 6.9: Bending strength properties of fiber-reinforced particleboard: wood-hemp (w-h), wood-flax (w-f), hemp hurd-hemp (hh-h) and flax shive-flax (fs-f). Horizontal line indicates minimum value stipulated by ANSI A208.1-1999 for M2 boards. $n=14$ for each mean. Error bars represent least significant difference between means.

After adjusting the MOR and MOE values of the fiber-reinforced particleboards for the effect of density variation, significant differences ($p=0.0158$, $p=0.0024$) were observed when 100% wood particleboard was compared to wood–flax and wood–hemp (Figure 6.10 and Appendix E). A

percent increase in MOR and MOE of 42% and 28% was observed in the wood–flax particleboards, and 53% and 32% for the wood–hemp particleboards respectively. The higher bending strength and stiffness properties observed for the fiber-reinforced particleboards is largely due to the tensile strength and modulus contribution of the fibers (Table 6.1) which have been reported to be in the range of 800–1500 MPa and 60–80 GPa for flax fiber and 550–900 MPa and 70 GPa for hemp fiber respectively (Anandjiwala and Blouw, 2007). A comparison between the 100% hemp hurd particleboard and the hemp hurd–hemp fiber-reinforced board yielded significant differences ($p<0.0001$), with the hemp hurd–hemp boards 60% stronger and 46% stiffer than the 100 % hemp hurd particleboards (Figure 6.10 and Appendix E). Particleboards containing the flax shive residue, that is the 100% flax shive and flax shive–flax boards, were only significantly different ($p=0.0005$) in terms of their bending stiffness values (27% increase for the flax shive–flax boards) but not their bending strength (Figure 6.10 and Appendix E).

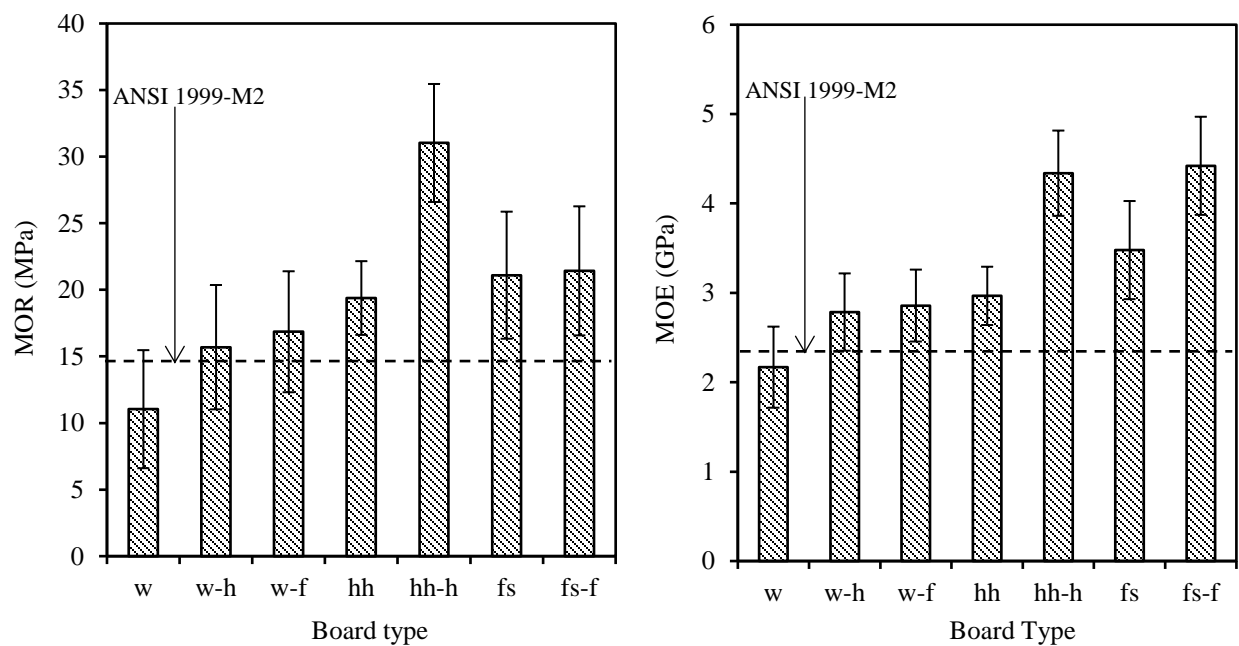


Figure 6.10: Comparison of the bending strength and stiffness properties of 100% wood, hemp hurd and flax shive particleboards with flax and hemp fiber-reinforced particleboards: wood (w), wood-hemp (w-h), wood-flax (w-f), hemp hurd (hh), hemp hurd-hemp (hh-h), flax shive (fs) and flax shive-flax (fs-f). $n=14$ for each mean. Error bars represent 95% confidence interval.

The improvement in strength characteristics observed in this study are similar to those reported by Troger et al. (1998) who used flax fiber and flax straw as reinforcements in Spruce and Beech particleboards. Using 3–6% flax fiber and flax straw, approximately 20–60% increase in bending properties was observed in particleboards 20 mm and 38 mm thick bonded with 5.5% pMDI resin load to 715 kg/m³ and 750 kg/m³ density respectively. This increase particularly for the flax fiber-reinforced particleboards was ascribed by the researchers to the fiber properties and not necessarily the proportion of fiber used.

6.3.3 Physical properties

6.3.3.1 TS and WA

The thickness swell (TS) and water absorption (WA) properties of the fiber-reinforced particleboards are shown in Figure 6.11. Significant differences ($p=0.0028$ and $p<0.0001$) were observed between board types for both short-term (2 hours) and long term (24 hours) thickness swell and water absorption properties (Appendix E). The greatest swell in thickness (10%) corresponding to the highest water absorption (21%) was consistently observed for the hemp hurd–hemp particleboards, closely followed by the flax shive–flax (9.8% and 17.6%) and the wood–hemp boards (8% and 15%) respectively. The wood–flax boards proved to be most dimensionally stable with a maximum thickness swell of 6% and 11.7% water absorption. The ANSI standards have no stipulated maximum values for thickness swell or water absorption regarding medium density M2 particleboard, as such there was no benchmark for evaluation of the fiber-reinforced particleboards. Generally, the thickness swell and water absorption were highest in particleboards manufactured with the hemp hurd and flax shive residues.

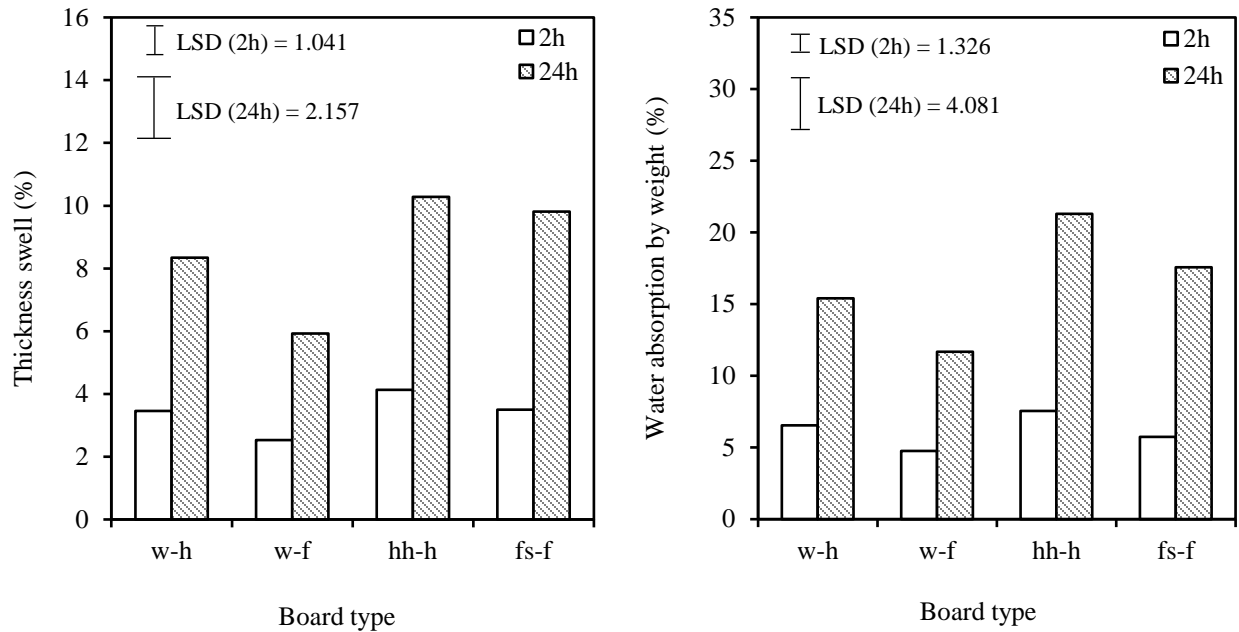


Figure 6.11: Short and long-term thickness swell and water absorption of flax and hemp fiber-reinforced particleboards: wood-hemp (w-h), wood-flax (w-f), hemp hurd-hemp (hh-h) and flax shive-flax (fs-f). $n=11$ for each mean. Error bars represent least significant difference between means.

As was done with the mechanical properties, the physical properties were normalized for variations in density. A general decrease of 23–84% was observed in thickness swell and water absorption between the particleboards manufactured from a 100% wood, hemp hurd and flax shive residues and the fiber-reinforced boards with exception of the hemp hurd-based particleboards. A comparison between the wood-based particleboards revealed no significant differences in both TS and WA characteristics for the first 2 hours (Figure 6.12 and Appendix E). After 24 hours of submersion slightly significant differences ($p=0.0046$ for TS and $p=0.0496$ for WA) were observed: the wood–flax particleboards swelled the least with the lowest water uptake corresponding to approximately 45% and 70% decrease respectively (Appendix E).

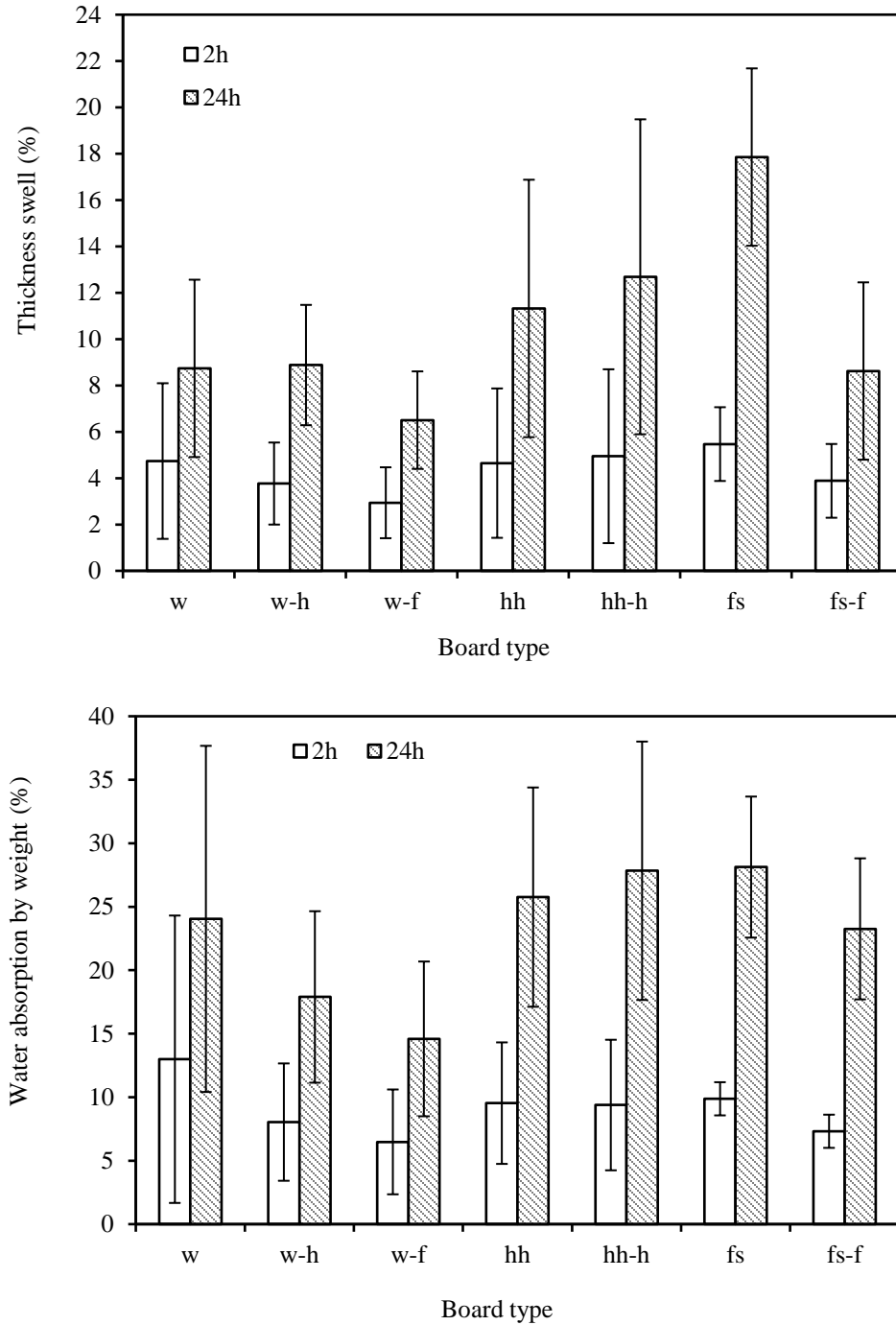


Figure 6.12: Comparison of the 2 and 24 hours thickness swell and water absorption characteristics of 100% wood, hemp hurd and flax shive particleboards with fiber-reinforced particleboards: wood (w), wood-hemp (w-h), wood-flax (w-f), hemp hurd (hh), hemp hurd-hemp (hh-h), flax shive (fs) and flax shive-flax (fs-f). $n=11$ for each mean. Error bars represent 95% confidence interval.

No differences were observed in TS and WA between the 100% hemp hurd boards and hemp hurd–hemp particleboards. The 100% flax shive particleboards were significantly greater from the flax shive–flax boards in their 2 and 24 hours TS values ($p=0.0337$ and $p<0.0001$), and significantly different for only WA in the first 2 hours ($p=0.0003$) (Appendix E). A total decrease of 42% and 48% was observed for 24 hours TS and WA in the flax shive–flax boards.

The lower WA and TS of particleboards with the flax fiber specifically in the wood–flax and flax shive–flax compared with those containing the hemp fiber is credited to the fact that the flax fiber mats were relatively softer and allowed better compaction between the fibers compared to the rigid hemp fiber mats. To evaluate the compaction characteristics of the aligned flax and hemp fiber mats were sprayed with 5% pMDI resin and compressed in the hot press at 140 °C and 2 MPa. Test samples were embedded in Spurr's resin (hard formulation) and sections approximately 0.5 μm thick were cut using a Leica UC 7 microtome. A light microscope, Zeiss Axioplan II equipped with a QImaging camera was used for image analysis and capturing. As is evident from Figure 6.13, sections of the compressed samples compared with the uncompressed samples reveal highly compacted flax fibers which made them less permeable resulting in a lower rate of water diffusion into the particleboard.

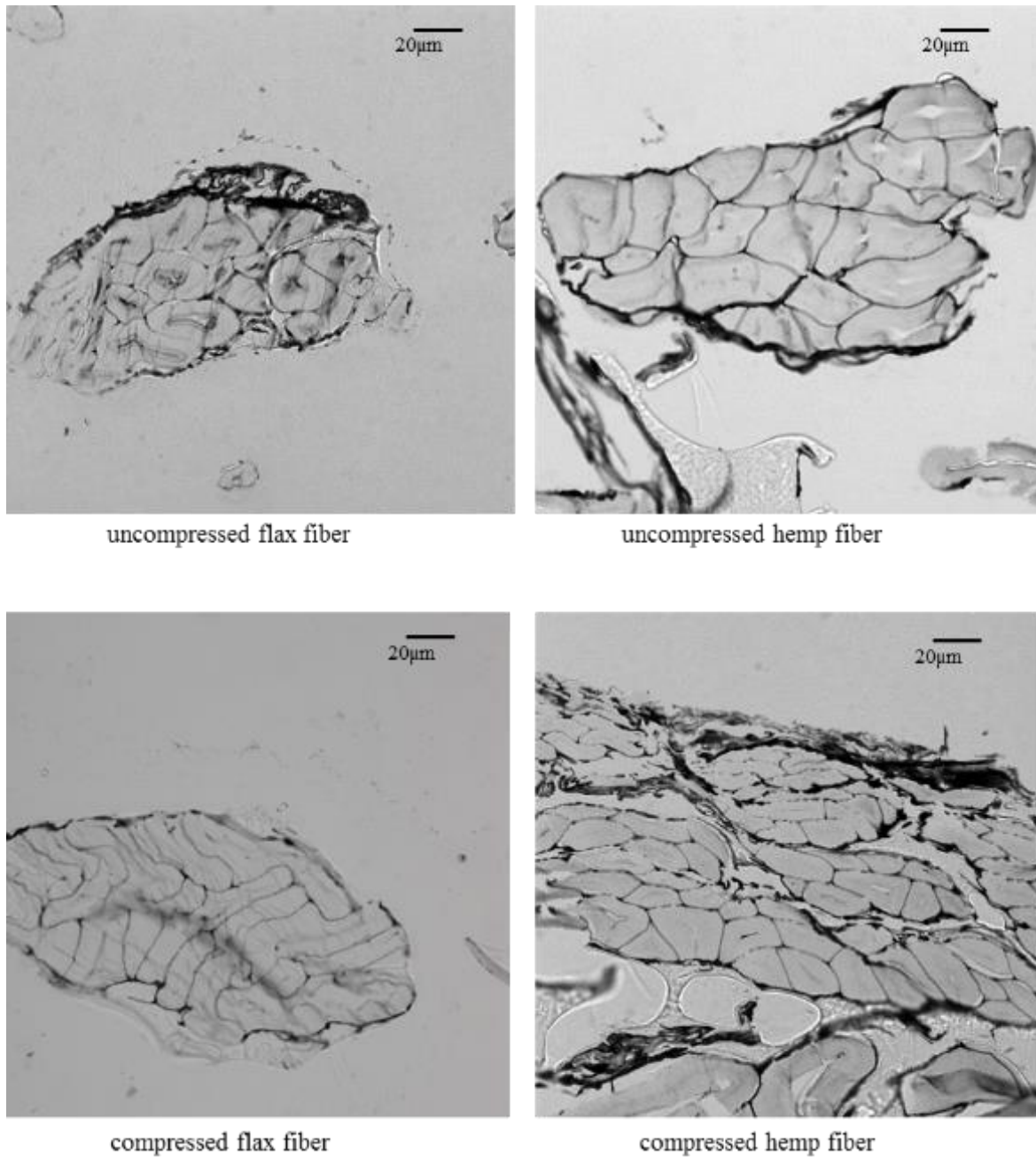


Figure 6.13: Microscopic images of the cross-section of uncompressed and compressed flax and hemp fiber mats. Source: Sam-Brew 2016.

6.4 Conclusions

Several methods have been employed over the years to improve the strength properties of particleboard panels using both natural and synthetic materials. The findings presented in this

study is based on the use of flax and hemp fibers as reinforcement in the upper and lower faces of particleboards where the greatest compressive and tensile forces are observed in service. A resin load of 5% pMDI (oven dry weight) was used in combination with the flax and hemp fiber, and wood, flax shive and hemp hurd residues for particleboard manufacture.

A wide variability was observed in internal bond data due to the failure mode which frequently occurred in the flax and hemp fiber layers. These sections were identified as weak resin free zones in most of the particleboards. Spraying of resin onto the fiber mats and subsequent laying of the mats during board manufacture was identified as the major source of variation within the boards causing poor resin distribution. Even so the internal bond strength of all the particleboards exceeded the ANSI A208.1-1999 requirement for medium density M2 grade particleboard.

The bending strength and stiffness properties were significantly improved in comparison to similar particleboards manufactured without fiber reinforcement. A percent increase of above 40% in MOR and 25% in MOE was observed for all the particleboards with the inclusion of flax and hemp fiber mats; the result of the high tensile properties of the flax and hemp fibers. These flexure properties were most prominent in particleboards manufactured from a combination of hemp hurd residues and hemp fiber.

Though thickness swell and water absorption properties were generally higher in the hemp hurd and flax shive particleboards without flax or hemp fiber reinforcement, the addition of flax fiber to the flax shive particleboards significantly reduced the thickness swell values by approximately 42% and the water absorption by 48%. The same was true of the wood particleboards, where with the addition of flax fiber mats, a 45% and 70% reduction was observed in the thickness swell and water absorption properties respectively. This was attributed to the nature of the softer flax fiber which permitted greater compaction between fibers making it less permeable to water.

The results of this study indicate enhanced performance in mechanical strength properties when flax and hemp fibers are incorporated as reinforcements in particleboards. Based on the findings it is recommended that fiber reinforcement with aligned flax and hemp fiber mats be considered for particleboard panels, especially for medium to high density particleboards where thinner yet stronger boards (example 9.5 or 11 mm boards equivalent to 3/8 and 7/16 inch) can be

manufactured using this approach. Such thin medium density particleboards will be of interest to furniture manufacturers who produce flat-pack furniture.

7. Research summary and conclusions

7.1 Summary

Particleboard is a non-structural panel used for furniture, kitchen cabinets, floor underlayment, door cores, shelving and counter top applications. Limited wood residue supply and the cost of transporting the residue over long distances to particleboard mills continues to be a matter of great concern. In this research, alternate residue resource in the form of flax shive and hemp hurd residues were investigated as substitutes to wood fiber for particleboard production in conjunction with isocyanate, acrylic and lignin-based resins. Flax and hemp fibers were also identified as natural materials that can be used as reinforcements to improve particleboard strength properties. This concluding chapter gives a summary of the significant findings obtained from the experimental work conducted on the performance of particleboards manufactured from flax shive residue, hemp hurd residue, flax and hemp fibers.

The first part of the study focused on characterization of the flax shive and hemp hurd residues and the feasibility of manufacturing 3-layered particleboards from these residues using 2.5% and 5% pMDI resin (based on oven dry weight of furnish). The low density particleboards were evaluated according to ASTM and ANSI standards for their short term (2 hours) and long term (24 hours) water absorption and thickness swell characteristics, internal bond, modulus of rupture and elasticity properties. Lower bulk densities were observed in the slender and rectangular flax shive and hemp hurd residues in comparison to wood residue. The test results indicated higher bending properties for the flax shive and hemp hurd boards compared with the wood particleboards owing to their greater length to thickness ratios. Thermal analysis of the pMDI resin and residues indicated more chemical bond formations between the pMDI resin molecules in the presence of the flax shive residue, and lower cure properties with the hemp hurd residue. Flax shive and hemp hurd particleboards manufactured with the lower 2.5% resin load surpassed the mechanical strength (internal bond and static bending) requirements for low density LD2 grade particleboard indicating that lower rates of isocyanate resin could still be used for particleboard manufacture. The same boards manufactured with 5% pMDI resin load to a low density exceeded the bending strength and stiffness properties mandated for medium density M2 grade particleboard compared to wood particleboards. The absorbent nature of the flax shive and hemp hurd residues resulted in

consistently higher thickness swell properties than those of the wood residues. Despite this porous feature, in contrast to the wood particleboards the linear expansion in the hemp hurd and flax shive residues with increased relative humidity from 50% to 90% (20 ± 3 °C) were within ANSI's stipulated minimum values due to their particle geometry and arrangement during board manufacture. Therefore, based on the residue characteristics and strength performance obtained for the hemp hurd and flax shive particleboards, these residues can be used in combination with low pMDI resin quantities (2.5% resin load) to substitute wood residue for niche particleboard products which demand higher mechanical strength properties.

A wide variety of adhesives are used in the manufacture of wood panel products, most of which are formulated based on condensation reactions of formaldehyde with urea, phenol, melamine or a combination of these. Formaldehyde emissions from these adhesives has been a matter of concern. Stringent emission regulations have led to research and development of ultra-low emitting and no-added formaldehyde resin technologies. In the second part of this study low density wood, flax shive and hemp hurd particleboards were fabricated with an acrylic based formaldehyde-free resin — Acrodur® 950L. The thermal properties of Acrodur® 950L resin in combination with wood, hemp hurd and flax shive residues evaluated against that of UF resin in combination with the same residues revealed a lower degree of crosslinking between the Acrodur® 95L resin molecules. The degree of cure observed between the Acrodur® 950L resin molecules in the presence of the non-wood residues was approximately 64% less than that in the UF resin. Signifying that Acrodur® 950L resin may not be suited for bonding flax shive and hemp hurd residues. The internal bond and static bending properties evaluated against ANSI A208.1 standards revealed that Acrodur® 950L resin at a low application rate of 7% (oven dry weight) produced hemp hurd and flax shive particleboards that exceed the requirements for low density LD2 grade particleboard. Linear expansion values for the flax shive and hemp hurd particleboards were within the ANSI stipulations and were comparable to those manufactured from 2.5% pMDI resin. Consistent with their high affinity for water, the thickness swell and water absorption characteristics of the hemp hurd and flax shive boards were once again significantly higher than those of the wood boards. Based on the DSC results it appears that Acrodur® 950L cannot efficiently bond non-wood residues despite the performance of their mechanical board properties. But to validate this finding further work will be required where processing parameters such as

residue size, resin load, moisture content, press temperature and time are carefully controlled for particleboards manufactured from both UF resin and Acrodur® 950L under the same processing conditions.

Polymeric diphenyl methane diisocyanate (pMDI) is an expensive resin that costs twice the price of urea formaldehyde resin. But its superior bonding properties makes it most ideal for bonding non-wood residues which are not easily bonded by the amino and phenolic-based resins. To reduce the cost of pMDI resin used in particleboard production 5 and 20 weight % of the resin was substituted with softwood Kraft lignin in the third part of the research. Using 2.5% pMDI resin load (oven dry weight of furnish) with 5 and 20 weight % lignin substitutions, low density wood, hemp hurd and flax shive particleboards were manufactured. Based on the test results an increase in lignin substitution resulted in lower internal bond strength, static bending properties and an increase in linear expansion, thickness swell and water absorption properties of the particleboards compared to the control boards without lignin. This observation was in line with the reaction heat values observed in the DSC tests conducted on combinations of the wood, hemp hurd and flax shive residues with the pMDI-lignin resins which indicated a lower degree of cure between resin molecules with increasing lignin content. Yet the internal bond strength of all the particleboards manufactured with pMDI-lignin resins irrespective of residue type exceeded the minimum ANSI requirements for medium density M2 grade particleboard and the bending strength properties of only the hemp hurd and flax shive particleboards surpassed the requirements for low density LD2 grade particleboard. In terms of physical properties linear expansion values for the flax shive particleboards and hemp hurd with 5 weight % lignin substitution complied with the ANSI requirements. The results present an interesting trend in that even with a low pMDI resin load which had mass fractions replaced with lignin, the properties of the flax shive and hemp hurd boards surpassed the minimum ANSI requirements stipulated for low density grade particleboard, and were comparable in performance to particleboards produced with 2.5% pMDI resin only. Implying that even with the limited number of bonds between the resin molecules with the addition of Kraft lignin, the possibility exists for bonding non-wood residues with pMDI-lignin resins.

The final part of the study was aimed at reinforcing particleboard products with natural bast fibers. Three-layered particleboards were manufactured from wood, hemp hurd and flax shive residues and reinforced in the upper and lower face layers with aligned flax and hemp fiber mats. For

comparison purposes results of wood, hemp hurd and flax shive particleboards manufactured with 5% pMDI resin load from the first part of the study were used as control samples. The internal bond strength data displayed a great deal of variability within the fiber-reinforced particleboard types. The results revealed low interfacial bond strength within the flax and hemp fiber layers, thus these regions were the major points of failure during testing. Compared with the control samples the bending strength properties of the fiber-reinforced particleboards were significantly ($p=0.0158$, $p<0.0001$, $p=0.0005$) improved, an increase in MOR and MOE of 42% and 28% for the wood residue and flax fiber boards, 53% and 32% for wood residue and hemp fiber boards, 60% and 46% for hemp hurd and hemp fiber boards and 27% only in MOE for the flax shive and flax fiber boards. The internal bond and bending strength properties of the fiber-reinforced particleboards exceeded the ANSI requirements for medium density M2 grade particleboard. The thickness swell and water absorption properties were also significantly reduced for the fiber-reinforced boards especially in the wood-flax particleboards by 45% and 70% respectively. This was attributed to the ease with which the flax fiber mats could be tightly compacted making it less permeable to moisture. The improved strength properties observed for the fiber-reinforced particleboards suggest that natural bast fibers can be used in place of expensive synthetic materials to strengthen particleboard products, by so doing improve its period in service and permit its use for applications with higher strength requirements.

7.2 Significance of the study to the particleboard industry

The original motivation for this study was to identify alternate residue resources available in Canada that can substitute wood for particleboard production to serve as a solution to wood residue shortage. Flax shive and hemp hurd residues were identified and evaluated through a series of experimental work.

The high cost of pMDI resin (approximately \$1.8-\$2.2/kg) as well as the protection requirements that comes with it has commonly deterred its use for panel production. Despite this pMDI was selected over the traditional urea and phenol formaldehyde resins because of its high reactivity, low viscosity, non-formaldehyde emitting properties and ability to penetrate the waxy outer layer of most agricultural residues. Typically for particleboard manufacture pMDI is applied at 3% to

6% resin load. The results of this study indicate that it is entirely possible to manufacture low density particleboards (500-620 kg/m³) from flax shive and hemp hurd residues to conform to ANSI standard specifications using isocyanate resin quantities as low as 2.5% (furnish oven dry weight). The mechanical performance of the low density particleboards produced from the flax shive and hemp hurd residues were significantly higher than those obtained from wood particleboards. And with further detailed studies in which production parameters are adequately controlled higher quality particleboards can be produced. The downstream impacts of such low density flax shive and hemp hurd particleboards to panel manufacturers include easy handling and processing which correlates to faster production rates, less energy requirements to compact the material, savings on transportation and shipping costs on the final product.

Also, the results show that further savings can be achieved by replacing up to 20 weight % pMDI resin with Kraft lignin at low resin application rates of 2.5% pMDI resin load to produce boards that satisfy low density grade standards. Currently Kraft lignin costs approximately \$0.4-\$0.5/kg. Therefore, for a 762 mm by 762 mm by 12.7 mm particleboard bonded with pMDI at a 2.5% resin load (oven dry weight), a total of 0.112 kg pMDI resin will be used for board manufacture at a resin cost of \$0.25 per board (assuming the higher resin price point per kg). At a Kraft lignin substitution level of 20 weight %, the amount of pMDI resin used will be 0.09 kg at a cost of \$0.20, while the lignin used will cost \$0.01. Bringing the total cost of resin for the same 762 mm by 762 mm by 12.7 mm particleboard bonded with pMDI-lignin resin to \$0.21 per board. A total resin savings of \$0.04 per board. Ultimately these findings confirm that cost savings in terms of pMDI resin can be attained by decreasing the quantity of resin used for board manufacture and or substituting part of the resin with Kraft lignin.

However, the current cost of the flax shive and hemp hurd residues and their transportation costs over significant distances does not make them cost competitive and commercially viable for particleboard production compared to wood residues.

Progress in no-added formaldehyde resin technologies have been made in response to regulatory and market drivers such as the Airborne Toxic Control Measure, Green building interests and market needs. The performance of one such resin—Acrodur[®] 950L and its ability to bond both wood and non-wood residues in the form of flax shive and hemp hurd residues was evaluated

through the manufacture of low density particleboards. Thermal analysis of the Acrodur® 950L resin in combination with wood, hemp hurd and flax shive residues revealed lower bonding properties than those obtained for UF resin in combination with the same residues. For the particleboard industry, these dynamic scanning calorimetry results in conjunction with the cost of the resin suggests that Acrodur® 950L might not be a suitable resin for particleboard manufacture from flax shive and hemp hurd residues.

The study also demonstrates that the approach to reinforce particleboard panels using bast fibers is viable. The experimental data obtained indicates that 15% (weight basis) aligned flax and hemp fiber mats placed at points of maximum tensile and compressive stresses can be efficiently used to reinforce particleboard products. For particleboard manufacturers, the higher strength properties obtained for the fiber-reinforced particleboards suggests much thinner boards such as 9.5 or 11 mm boards of equivalent strength to a 12.7 mm board can be produced using this technique. To a furniture manufacturer on the other hand such thin medium density particleboards will permit design freedom and the use of the particleboard in products with higher load capacity requirements.

7.3 Limitations of the study and future research directions

In the second part of this study, the focus was on evaluating mechanical and physical properties of particleboards manufactured from an acrylic-based resin, Acrodur® 950L, a non-formaldehyde emitting resin comparable to urea and phenol formaldehyde resins. A limitation in the study design was identified in the form of the absence of experimental work to equally produce wood, hemp hurd and flax shive particleboards bonded with urea formaldehyde resin as was done in the DSC thermal analysis. This error occurred because based on a review of literature, urea formaldehyde resins had been reported to be unable to efficiently bond non-wood residues thus there was no need to repeat experiments along those lines. Consequently, the current results obtained for particleboards (wood, hemp hurd and flax shive) manufactured with Acrodur® 950L resin lacks a common basis for comparison with particleboards manufactured from similar residues bonded with urea formaldehyde resins (a common resin for particleboard production). But based on the DSC results which indicates lower bonding properties between the Acrodur® 950L resin and the wood, hemp hurd and flax shive residues compared with urea formaldehyde resin, and based on

the particleboard properties observed, the study is worth repeating to include particleboards manufactured from urea formaldehyde resin to strongly substantiate the potential of Acrodur® 950L resin in bonding wood and non-wood residues in contrast to urea formaldehyde resins.

The study on the substitution of portions of pMDI resin with Kraft lignin produced results that indicated that an increase in lignin substitution from 5 weight % directly to 20 weight % resulted in a decrease in particleboard mechanical and physical properties. This study is limited by the fact that incremental lignin substitution levels between 5% and 20 weight % were not considered in particleboard manufacture. The results obtained are indicative of a trend but this cannot be necessarily generalized to include lignin substitution levels at 10 or 15 weight %. All the same the results obtained from DSC and particleboard properties tests are still applicable as they provide insight into the curing behavior of pMDI-lignin based resins in combination with non-wood residues. To re-test this phenomenon, it is recommended that the board experiments be repeated with levels 5, 10, 15 and 20 weight % lignin substitution in pMDI resin. This will provide more evidence to corroborate the observation that an increase in the lignin substitution in pMDI resin is consistently accompanied by a simultaneous decrease in particleboard strength properties. It is also suggested that further research be carried out to identify means of incorporating more than 20 weight % lignin directly in pMDI resin in such a way that allows uniform dispersion of the resin by a blender atomizer.

The technique of reinforcing particleboard panels with aligned bast fiber mats is a novel approach which proved to produce boards of higher strength properties. Yet a major challenge with this approach was ensuring even distribution of resin on and within the flax and hemp fiber mats during resin application, which in turn affected the internal bond properties of the particleboards. Application methods such as paint rollers and dipping were considered but these were unsuccessful; the fibers stuck to the rollers in the first case or absorbed and used too much resin in the latter. Based on these results the recommended next steps will be to identify a highly automated resin application system that guarantees a high level of consistent and accurate distribution of resin within the fibers mats. This will ensure greater bond strength between the fibers and effective stress transfer from one fiber to the next, ultimately providing a compelling evidence base for this method of strengthening particleboard panels. Furthermore, due to lack of equipment, the creep properties of the fiber-reinforced particleboards were not evaluated. It is recommended that future

research be conducted to evaluate this property and provide information on the time-dependent deformation characteristics of the reinforced boards. This will also help determine its performance in service.

Thermal analysis via differential scanning calorimetry indicated lower cure properties when pMDI and Acrodur[®] 950L resin were combined with hemp hurd residues. On the other hand, the highest reaction heat values were consistently observed in pMDI and pMDI-lignin resin in combination with the flax shive residues. These results could be caused by certain chemical constituents and/or anatomical features of the residue which inhibits or enhances bond formation between the resin molecules. These results remain unexplained and more work is required to better understand the effect of the residues on the curing reaction of the resins.

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Appendices

Appendix A: wood-based panels production and consumption statistics

Total wood-based panel production quantities in Canada 2014-2015

Year	Production quantities (m ³)					
	Veneer sheets	Plywood	Particleboard	Hardboard	MDF	OSB
2014	520000	1810000	8587000	82000	849000	6877000
2015	580000	1929000	8796000	84000	970000	7074000

Source: FAO, United Nations Statistics Division 2016

Canadian particleboard statistics. * is predicted data.

	2014	2015	2016*	2017*
Production (m ³)	8,587,000	8,796,000	9,540,000	10,107,000
Apparent consumption (m ³)	4,345,000	4,324,000	4,061,000	4,094,000
Imports (m ³)	1,036,000	1,227,000	1,053,000	999,000
Exports (Total m ³)	5,278,000	5,699,000	6,532,000	7,012,000

Source: UNECE Committee on Forests and the Forest Industry 2016

Appendix B: statistical analysis - chapter 3

Face bulk density statistics

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample	2	19115.330	9557.66	1069.701	<.0001*
Error	6	53.609	8.93		
C. Total	8	19168.939			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd face	3	88.135	1.7258	83.91	92.36
flax shive face	3	140.012	1.7258	135.79	144.23
wood face	3	200.902	1.7258	196.68	205.13

Std Error uses a pooled estimate of error variance

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha
3.06815 0.05

Positive values show pairs of means that are significantly different.

Levels not connected by same letter are significantly different.

Level		Mean
wood face	A	200.90223
flax shive face	B	140.01167
hemp hurd face	C	88.13490

Core bulk density statistics

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample	2	10608.127	5304.06	1158.129	<.0001*
Error	6	27.479	4.58		
C. Total	8	10635.606			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd core	3	99.530	1.2356	96.51	102.55
flax shive core	3	89.875	1.2356	86.85	92.90
wood core	3	167.050	1.2356	164.03	170.07

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
3.06815	0.05

Positive values show pairs of means that are significantly different.

Levels not connected by same letter are significantly different.

Level		Mean
wood core	A	167.05004
hemp hurd core	B	99.53035
flax shive core	C	89.87496

2.5% resin load average board density

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Board type	2	3970.177	1985.09	2.5086	0.0978
Error	31	24530.898	791.32		
C. Total	33	28501.075			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	12	532.432	8.1205	515.87	548.99
flax shive	10	533.131	8.8956	514.99	551.27
wood	12	555.354	8.1205	538.79	571.92

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
2.46119	0.05

Positive values show pairs of means that are significantly different.

Levels not connected by same letter are significantly different.

Level		Mean
wood	A	555.35429
flax shive	A	533.13121
hemp hurd	A	532.43179

2.5% resin load moisture content

Analysis of Variance (Logarithm transformation)

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample	2	0.01671682	0.008358	99.0765	<.0001*
Error	33	0.00278398	0.000084		
C. Total	35	0.01950080			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	12	1.03883	0.00265	1.0334	1.0442
flax shive	12	1.06283	0.00265	1.0574	1.0682
wood	12	1.01012	0.00265	1.0047	1.0155

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.45379 0.05

Positive values show pairs of means that are significantly different.

Levels not connected by same letter are significantly different.

Level	Mean
flax shive A	1.0628286
hemp hurd B	1.0388309
wood C	1.0101150

5% resin load average board density

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Board type	2	5363.498	2681.75	3.4977	0.0401*
Error	39	29901.787	766.71		
C. Total	41	35265.285			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	14	630.530	7.4003	615.56	645.50
flax shive	14	657.417	7.4003	642.45	672.39
wood	14	638.275	7.4003	623.31	653.24

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha
2.43631 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
flax shive	A	657.41733
wood	A B	638.27463
hemp hurd	B	630.53043

5% resin load moisture content

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Board Type	2	2.2765337	1.13827	78.2197	<.0001*
Error	30	0.4365651	0.01455		
C. Total	32	2.7130988			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	11	10.5261	0.03637	10.452	10.600
flax shive	11	10.8618	0.03637	10.788	10.936
wood	11	10.2186	0.03637	10.144	10.293

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha
2.46534 0.05

Levels not connected by same letter are significantly different.

Connecting Letters Report

Level		Mean
flax shive	A	10.861786
hemp hurd	B	10.526120
wood	C	10.218629

DSC analysis

Onset temperature - residues (moist)

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	2	24.354633	12.1773	16.5374	0.0240*
Error	3	2.209050	0.7364		
C. Total	5	26.563683			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	2	112.705	0.60677	110.77	114.64
flax shive	2	111.190	0.60677	109.26	113.12
wood	2	107.880	0.60677	105.95	109.81

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
4.17871	0.05

Levels not connected by same letter are significantly different.

Connecting Letters Report

Level	Mean
hemp hurd	A 112.70500
flax shive	A B 111.19000
wood	B 107.88000

Onset temperature - residues and neat pMDI resin (moist)

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	3	53.973972	17.9913	28.8145	0.0014*
Error	5	3.121917	0.6244		
C. Total	8	57.095889			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	2	112.705	0.55874	111.27	114.14
pMDI	3	106.743	0.45621	105.57	107.92
flax shive	2	111.190	0.55874	109.75	112.63
wood	2	107.880	0.55874	106.44	109.32

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
3.69002	0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
hemp hurd A	112.70500
flax shive A	111.19000
wood B	107.88000
pMDI B	106.74333

Peak Temperature - residues (moist)

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	2	39.698533	19.8493	72.9395	0.0029*
Error	3	0.816400	0.2721		
C. Total	5	40.514933			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Hp	2	142.460	0.36887	141.29	143.63
Sp	2	136.230	0.36887	135.06	137.40
Wp	2	138.530	0.36887	137.36	139.70

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
4.17871	0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Levels not connected by same letter are significantly different.

Level	Mean
hemp hurd A	142.46000
wood B	138.53000
flax shive C	136.23000

Peak Temperature - residues and neat pMDI resin (moist)

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	3	187.04576	62.3486	32.6549	0.0010*
Error	5	9.54660	1.9093		
C. Total	8	196.59236			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	2	142.460	0.97707	139.95	144.97
pMDI	3	130.490	0.79777	128.44	132.54
flax shive	2	136.230	0.97707	133.72	138.74
wood	2	138.530	0.97707	136.02	141.04

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha
3.69002 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
hemp hurd	A 142.46000
wood	A B 138.53000
flax shive	B 136.23000
pMDI	C 130.49000

Reaction heat - residues (moist)

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	2	150.10333	75.0517	2.6228	0.2195
Error	3	85.84500	28.6150		
C. Total	5	235.94833			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	2	112.750	3.7825	100.71	124.79
flax shive	2	125.000	3.7825	112.96	137.04
wood	2	118.700	3.7825	106.66	130.74

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha
4.17871 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
flax shive	A 125.00000
wood	A 118.70000
hemp hurd	A 112.75000

Reaction heat - residues and neat pMDI resin (moist)

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	3	27286.837	9095.61	290.9572	<.0001*
Error	5	156.305	31.26		
C. Total	8	27443.142			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	2	112.750	3.9535	102.59	122.91
pMDI	3	235.300	3.2281	227.00	243.60
flax shive	2	125.000	3.9535	114.84	135.16
wood	2	118.700	3.9535	108.54	128.86

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha
3.69002 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
pMDI	A 235.30000
flax shive	B 125.00000
wood	B 118.70000
hemp hurd	B 112.75000

2.5% resin load internal bond

Analysis of Variance (square root transformation)

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample	2	0.36729681	0.183648	41.4489	<.0001*
Error	93	0.41205714	0.004431		
C. Total	95	0.77935395			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	32	0.930563	0.01177	0.90720	0.95393
flax shive	32	0.779065	0.01177	0.75570	0.80243
wood	32	0.853003	0.01177	0.82964	0.87637

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.38183 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
hemp hurd A	0.93056315
wood B	0.85300324
flax shive C	0.77906503

5% resin load internal bond

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Board Type	2	2.7466590	1.37333	56.5258	<.0001*
Error	141	3.4256839	0.02430		
C. Total	143	6.1723429			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	48	1.15005	0.02250	1.1056	1.1945
flax shive	48	1.08599	0.02250	1.0415	1.1305
wood	48	1.40569	0.02250	1.3612	1.4502

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.36876 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
wood	A	1.4056928
hemp hurd	B	1.1500529
flax shive	B	1.0859893

2.5% resin load modulus of rupture

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample	2	437.54085	218.770	60.9568	<.0001*
Error	31	111.25725	3.589		
C. Total	33	548.79810			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	12	12.3952	0.54688	11.280	13.511
flax shive	10	10.0068	0.59908	8.785	11.229
wood	12	4.0747	0.54688	2.959	5.190

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.46119 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
hemp hurd	A	12.395160
flax shive	B	10.006806
wood	C	4.074708

2.5% resin load modulus of elasticity

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample	2	8.543590	4.27179	48.6514	<.0001*
Error	31	2.721930	0.08780		
C. Total	33	11.265520			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	12	2.09332	0.08554	1.9189	2.2678
flax shive	10	2.13672	0.09370	1.9456	2.3278
wood	12	1.06472	0.08554	0.8903	1.2392

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.46119 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
flax shive	A	2.1367226
hemp hurd	A	2.0933218
wood	B	1.0647233

5% resin load modulus of rupture

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Board Type	2	1132.3712	566.186	154.9460	<.0001*
Error	39	142.5093	3.654		
C. Total	41	1274.8804			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	14	17.9041	0.51089	16.871	18.937
flax shive	14	18.2353	0.51089	17.202	19.269
wood	14	7.0587	0.51089	6.025	8.092

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.43631 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
flax shive	A	18.235321
hemp hurd	A	17.904085
wood	B	7.058654

5% resin load modulus of elasticity

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Board Type	2	23492664	11746332	151.1432	<.0001*
Error	39	3030946	77716.57		
C. Total	41	26523610			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	14	2717.25	74.506	2566.5	2867.9
flax shive	14	3293.73	74.506	3143.0	3444.4
wood	14	1499.56	74.506	1348.9	1650.3

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.43631 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
flax shive	A	3293.7274
hemp hurd	B	2717.2456
wood	C	1499.5573

2.5% resin load linear expansion

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample	2	0.45846575	0.229233	86.6470	<.0001*
Error	45	0.11905181	0.002646		
C. Total	47	0.57751756			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	16	0.305447	0.01286	0.27955	0.33135
flax shive	16	0.218481	0.01286	0.19258	0.24438
wood	16	0.455119	0.01286	0.42922	0.48102

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha
2.42362 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
wood A	0.45511920
hemp hurd B	0.30544730
flax shive C	0.21848119

5% resin load linear expansion

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample	3	0.43690910	0.145636	170.4920	<.0001*
Error	56	0.04783588	0.000854		
C. Total	59	0.48474498			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	20	0.290633	0.00654	0.27754	0.30373
flax shive-parallel	10	0.197494	0.00924	0.17898	0.21601
flax shive-perpendicular	10	0.270437	0.00924	0.25192	0.28895
wood	20	0.430717	0.00654	0.41762	0.44381

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.64794 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
wood	A 0.43071676
hemp hurd	B 0.29063320
flax shive-perpendicular	B 0.27043652
flax shive-parallel	C 0.19749373

2.5% resin load thickness swell-2 hours

Analysis of Variance-Logarithm transformation

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample	2	1.4226771	0.711339	81.2531	<.0001*
Error	33	0.2889017	0.008755		
C. Total	35	1.7115788			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	12	0.784313	0.02701	0.72936	0.83927
flax shive	12	0.773570	0.02701	0.71862	0.82852
wood	12	0.357340	0.02701	0.30239	0.41229

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.45379 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
hemp hurd	A 0.78431348
flax shive	A 0.77356997
wood	B 0.35733994

2.5% resin load thickness swell-24 hours

Analysis of Variance-Logarithm transformation

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample	2	1.3962979	0.698149	213.2953	<.0001*
Error	33	0.1080142	0.003273		
C. Total	35	1.5043121			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	12	1.21817	0.01652	1.1846	1.2518
flax shive	12	1.25802	0.01652	1.2244	1.2916
wood	12	0.82175	0.01652	0.7881	0.8553

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.45379 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
hemp hurd	A 1.2580219
flax shive	A 1.2181704
wood	B 0.8217476

2.5% resin load water absorption-2 hours

Analysis of Variance – Logarithm transformation

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample	2	0.9750538	0.487527	203.4205	<.0001*
Error	33	0.0790893	0.002397		
C. Total	35	1.0541431			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	12	1.19929	0.01413	1.1705	1.2280
flax shive	12	1.18736	0.01413	1.1586	1.2161
wood	12	0.84436	0.01413	0.8156	0.8731

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.45379 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
hemp hurd A	1.1992852
flax shive A	1.1873608
wood B	0.8443601

2.5% resin load water absorption-24 hours

Analysis of Variance – Reciprocal transformation

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample	2	0.00210574	0.001053	84.1919	<.0001*
Error	33	0.00041269	0.000013		
C. Total	35	0.00251843			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	12	0.020660	0.00102	0.01858	0.02274
flax shive	12	0.022544	0.00102	0.02047	0.02462
wood	12	0.037744	0.00102	0.03567	0.03982

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.45379 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
wood A	0.03774412
flax shive B	0.02254448
hemp hurd B	0.02066030

5% resin load thickness swell – 2 hours

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Board Type	2	10.63266	5.31633	1.2485	0.3014
Error	30	127.74939	4.25831		
C. Total	32	138.38204			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	11	5.78368	0.62219	4.5130	7.0544
flax shive	11	5.85771	0.62219	4.5870	7.1284
wood	11	7.02311	0.62219	5.7524	8.2938

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha
2.46534 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
wood	A	7.0231096
flax shive	A	5.8577083
hemp hurd	A	5.7836842

5% resin load thickness swell – 24 hours

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Board Type	2	186.11845	93.0592	22.7207	<.0001*
Error	30	122.87350	4.0958		
C. Total	32	308.99195			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	11	14.1303	0.61020	12.884	15.376
flax shive	11	16.6671	0.61020	15.421	17.913
wood	11	10.8651	0.61020	9.619	12.111

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.46534 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
flax shive	A	16.667084
hemp hurd	B	14.130271
wood	C	10.865111

5% resin load water absorption-2 hours

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Board Type	2	1945.3133	972.657	8.2996	0.0014*
Error	30	3515.7857	117.193		
C. Total	32	5461.0990			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	11	12.0142	3.2640	5.348	18.680
flax shive	11	11.4559	3.2640	4.790	18.122
wood	11	28.0150	3.2640	21.349	34.681

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.46534 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
wood	A	28.015021
hemp hurd	B	12.014240
flax shive	B	11.455937

5% resin load water absorption-24 hours

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Board Type	2	248.9044	124.452	1.0517	0.3619
Error	30	3550.1854	118.340		
C. Total	32	3799.0898			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	11	33.5135	3.2800	26.815	40.212
flax shive	11	33.7979	3.2800	27.099	40.496
wood	11	39.4764	3.2800	32.778	46.175

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha
2.46534 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
wood	A	39.476403
hemp hurd	A	33.797862
flax shive	A	33.513490

Appendix C: statistical analysis - chapter 4

Acrodur particleboards average board density

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Board type	2	8767.993	4384.00	10.1281	0.0002*
Error	57	24672.818	432.86		
C. Total	59	33440.811			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	20	568.672	4.6522	559.36	577.99
flax shive	20	577.003	4.6522	567.69	586.32
wood	20	597.446	4.6522	588.13	606.76

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha
2.40642 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
wood	A	597.44565
hemp hurd	B	577.00308
flax shive	B	568.67240

Acrodur particleboards moisture content

Analysis of Variance – Logarithm transformation

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample	2	0.01671682	0.008358	99.0765	<.0001*
Error	33	0.00278398	0.000084		
C. Total	35	0.01950080			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	12	1.03883	0.00265	1.0334	1.0442
flax shive	12	1.06283	0.00265	1.0574	1.0682
wood	12	1.01012	0.00265	1.0047	1.0155

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.45379 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
flax shive	A	1.0628286
hemp hurd	B	1.0388309
wood	C	1.0101150

DSC analysis

Onset temperature – acrodur (residues)

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	2	94.72557	47.3628	26.1763	0.0050*
Error	4	7.23752	1.8094		
C. Total	6	101.96309			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	2	147.810	0.95115	145.17	150.45
flax shive	2	141.095	0.95115	138.45	143.74
wood	3	149.833	0.77661	147.68	151.99

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

3.56399 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
wood	A	149.83333
hemp hurd	A	147.81000
flax shive	B	141.09500

Onset temperature – UF (residues)

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	2	136.52577	68.2629	193.1914	<.0001*
Error	5	1.76672	0.3533		
C. Total	7	138.29249			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	3	81.2967	0.34319	80.414	82.179
flax shive	3	81.4900	0.34319	80.608	82.372
wood	2	71.8550	0.42032	70.775	72.935

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
3.25386	0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
flax shive	A	81.490000
hemp hurd	A	81.296667
wood	B	71.855000

Onset temperature – UF (neat UF resin and residues)

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	3	702.40583	234.135	783.4250	<.0001*
Error	6	1.79317	0.299		
C. Total	9	704.19900			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	3	81.2967	0.31563	80.524	82.069
flax shive	3	81.4900	0.31563	80.718	82.262
UF	2	97.8150	0.38656	96.869	98.761
wood	2	71.8550	0.38656	70.909	72.801

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
3.46171	0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
UF	A	97.815000
flax shive	B	81.490000
hemp hurd	B	81.296667
wood	C	71.855000

Peak temperature – acrodur (residues)

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	2	1.507776	0.75389	0.3314	0.7359
Error	4	9.099767	2.27494		
C. Total	6	10.607543			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	2	175.465	1.0665	172.50	178.43
flax shive	2	175.795	1.0665	172.83	178.76
wood	3	176.533	0.8708	174.12	178.95

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
3.56399	0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
wood	A	176.53333
hemp hurd	A	175.79500
flax shive	A	175.46500

Levels not connected by same letter are significantly different.

Peak temperature – UF (residues)

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	2	142.25827	71.1291	516.2265	<.0001*
Error	5	0.68893	0.1378		
C. Total	7	142.94720			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	3	118.003	0.21431	117.45	118.55
flax shive	3	117.303	0.21431	116.75	117.85
wood	2	107.940	0.26248	107.27	108.61

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
3.25386	0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
hemp hurd	A	118.00333
flax shive	A	117.30333
wood	B	107.94000

Peak temperature – UF (neat UF resin and residues)

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	3	796.41571	265.472	2161.286	<.0001*
Error	6	0.73698	0.123		
C. Total	9	797.15269			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	3	118.003	0.20235	117.51	118.50
flax shive	3	117.303	0.20235	116.81	117.80
UF	2	135.445	0.24782	134.84	136.05
wood	2	107.940	0.24782	107.33	108.55

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
3.46171	0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
UF	A	135.44500
hemp hurd	B	118.00333
flax shive	B	117.30333
wood	C	107.94000

Reaction heat – acrodur (residues)

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	2	147.23975	73.6199	62.5999	0.0010*
Error	4	4.70415	1.1760		
C. Total	6	151.94390			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	2	8.2800	0.76682	6.151	10.409
flax shive	2	9.9810	0.76682	7.852	12.110
wood	3	18.3067	0.62611	16.568	20.045

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
3.56399	0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
wood	A	18.306667
flax shive	B	9.981000
hemp hurd	B	8.280000

Reaction heat – UF (residues)

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	2	11.189754	5.59488	7.1749	0.0339*
Error	5	3.898933	0.77979		
C. Total	7	15.088688			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	3	29.0733	0.50983	27.763	30.384
flax shive	3	28.0567	0.50983	26.746	29.367
wood	2	26.0300	0.62441	24.425	27.635

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
3.25386	0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
hemp hurd	A 29.073333
flax shive	A B 28.056667
wood	B 26.030000

Levels not connected by same letter are significantly different.

Reaction heat – UF (neat UF resin and residues)

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	3	3359.1748	1119.72	1654.202	<.0001*
Error	6	4.0614	0.68		
C. Total	9	3363.2362			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	3	29.0733	0.47501	27.911	30.236
flax shive	3	28.0567	0.47501	26.894	29.219
UF	2	73.6750	0.58176	72.251	75.099
wood	2	26.0300	0.58176	24.606	27.454

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha
3.46171 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
UF A	73.675000
hemp hurd B	29.073333
flax shive B C	28.056667
wood C	26.030000

Levels not connected by same letter are significantly different.

Internal bond – acrodur particleboards

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample	2	1.2653869	0.632693	69.5989	<.0001*
Error	93	0.8454228	0.009091		
C. Total	95	2.1108096			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	32	0.431000	0.01685	0.39753	0.46447
flax shive	32	0.170957	0.01685	0.13749	0.20443
wood	32	0.393707	0.01685	0.36024	0.42718

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha
2.38183 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
hemp hurd A	0.43099973
wood A	0.39370686
flax shive B	0.17095748

Levels not connected by same letter are significantly different.

Modulus of rupture – acrodur particleboards

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample	2	351.44384	175.722	89.2430	<.0001*
Error	57	112.23451	1.969		
C. Total	59	463.67835			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	20	10.7073	0.31377	10.079	11.336
flax shive	20	10.1898	0.31377	9.562	10.818
wood	20	5.3341	0.31377	4.706	5.962

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.40642 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
hemp hurd	A 10.707294
flax shive	A 10.189818
wood	B 5.334120

Levels not connected by same letter are significantly different.

Modulus of elasticity – acrodur particleboards

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample	2	13.670072	6.83504	104.5243	<.0001*
Error	57	3.727334	0.06539		
C. Total	59	17.397406			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	20	2.06441	0.05718	1.9499	2.1789
flax shive	20	2.26035	0.05718	2.1459	2.3749
wood	20	1.16415	0.05718	1.0497	1.2787

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.40642 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
flax shive A	2.2603543
hemp hurd B	2.0644093
wood C	1.1641536

Levels not connected by same letter are significantly different.

Linear expansion – acrodur particleboards

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	2	0.56290673	0.281453	294.4226	<.0001*
Error	39	0.03728206	0.000956		
C. Total	41	0.60018879			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	14	0.320241	0.00826	0.30353	0.33695
flax shive	14	0.253162	0.00826	0.23645	0.26988
wood	14	0.525316	0.00826	0.50860	0.54203

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.43631 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
wood A	0.52531591
hemp hurd B	0.32024074
flax shive C	0.25316239

Levels not connected by same letter are significantly different.

Thickness swell (2 hours) – acrodur particleboards

Analysis of Variance -Logarithm transformation

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample	2	1.0559071	0.527954	85.3002	<.0001*
Error	45	0.2785210	0.006189		
C. Total	47	1.3344281			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	16	1.31159	0.01967	1.2720	1.3512
flax shive	16	1.27159	0.01967	1.2320	1.3112
wood	16	0.97887	0.01967	0.9393	1.0185

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.42362 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
hemp hurd	A	1.3115870
flax shive	A	1.2715885
wood	B	0.9788716

Levels not connected by same letter are significantly different.

Thickness swell (24 hours) – acrodur particleboards

Analysis of Variance – Logarithm transformation

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample	2	1.1412320	0.570616	211.2047	<.0001*
Error	45	0.1215774	0.002702		
C. Total	47	1.2628094			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	16	1.41229	0.01299	1.3861	1.4385
flax shive	16	1.46277	0.01299	1.4366	1.4889
wood	16	1.11337	0.01299	1.0872	1.1395

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.42362 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
flax shive	A	1.4627737
hemp hurd	B	1.4122883
wood	C	1.1133722

Levels not connected by same letter are significantly different.

Water absorption (2 hours) – acrodur particleboards

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample	2	523.0537	261.527	2.1534	0.1279
Error	45	5465.1919	121.449		
C. Total	47	5988.2456			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	16	70.4948	2.7551	64.946	76.044
flax shive	16	63.5627	2.7551	58.014	69.112
wood	16	63.4238	2.7551	57.875	68.973

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.42362 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
hemp hurd	A	70.494803
flax shive	A	63.562723
wood	A	63.423768

Water absorption (24 hours) – acrodur particleboards

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample	2	1201.2958	600.648	12.5240	<.0001*
Error	45	2158.1933	47.960		
C. Total	47	3359.4891			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd	16	98.7657	1.7313	95.279	102.25
flax shive	16	98.1299	1.7313	94.643	101.62
wood	16	87.8498	1.7313	84.363	91.34

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.42362 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
hemp hurd A	98.765741
flax shive A	98.129936
wood B	87.849806

Levels not connected by same letter are significantly different.

Appendix D: statistical analysis - chapter 5

Moisture content – pMDI-lignin particleboards

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Samples	5	3.799336	0.759867	2.2011	0.0645
Error	66	22.784344	0.345217		
C. Total	71	26.583680			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-20L	12	10.8380	0.16961	10.499	11.177
hemp hurd-5L	12	10.8441	0.16961	10.505	11.183
flax shive-20L	12	10.9198	0.16961	10.581	11.258
flax shive-5L	12	11.1907	0.16961	10.852	11.529
wood-20L	12	10.5310	0.16961	10.192	10.870
wood-5L	12	10.5274	0.16961	10.189	10.866

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.93510 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
flax shive-5L	A 11.190696
flax shive-20L	A 10.919810
hemp hurd-5L	A 10.844125
hemp hurd-20L	A 10.838021
wood-20L	A 10.531033
wood-5L	A 10.527396

Levels not connected by same letter are significantly different.

Average board density – pMDI-lignin particleboards

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Board type	5	13133.873	2626.77	6.1807	<.0001*
Error	65	27624.781	425.00		
C. Total	70	40758.654			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-20L	12	521.460	5.9512	509.57	533.34
hemp hurd-5L	12	522.335	5.9512	510.45	534.22
flax shive-20L	12	542.972	5.9512	531.09	554.86
flax shive-5L	11	545.984	6.2158	533.57	558.40
wood-20L	12	556.068	5.9512	544.18	567.95
wood-5L	12	551.783	5.9512	539.90	563.67

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha
2.93643 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level			Mean
wood-20L	A		556.06810
wood-5L	A		551.78321
flax shive-5L	A	B	545.98399
flax shive-20L	A	B	542.97200
hemp hurd-5L		B	522.33487
hemp hurd-20L		B	521.45951

Levels not connected by same letter are significantly different.

DSC analysis - pMDI and Kraft lignin (moisture added)**Onset temperature – pMDI and Kraft lignin (moisture added)**

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample	2	3.750833	1.87542	0.7360	0.5494
Error	3	7.643850	2.54795		
C. Total	5	11.394683			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
pMDI+10L	2	107.010	1.1287	103.42	110.60
pMDI+20L	2	108.735	1.1287	105.14	112.33
pMDI+5L	2	107.110	1.1287	103.52	110.70

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
4.17871	0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
pMDI+20L	A 108.73500
pMDI+5L	A 107.11000
pMDI+10L	A 107.01000

Levels not connected by same letter are significantly different.

Peak temperature – pMDI and Kraft lignin (moisture added)

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample	2	9.0003000	4.50015	46.6417	0.0055*
Error	3	0.2894500	0.09648		
C. Total	5	9.2897500			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
pMDI+10L	2	124.390	0.21964	123.69	125.09
pMDI+20L	2	123.505	0.21964	122.81	124.20
pMDI+5L	2	126.430	0.21964	125.73	127.13

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
4.17871	0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
pMDI+5L	A 126.43000
pMDI+10L	B 124.39000
pMDI+20L	B 123.50500

Levels not connected by same letter are significantly different.

Reaction heat – pMDI and Kraft lignin (moisture added)

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample	2	2911.9156	1455.96	66.3342	0.0033*
Error	3	65.8465	21.95		
C. Total	5	2977.7621			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
pMDI+10L	2	231.400	3.3128	220.86	241.94
pMDI+20L	2	199.300	3.3128	188.76	209.84
pMDI+5L	2	252.915	3.3128	242.37	263.46

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
4.17871	0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
pMDI+5L A	252.91500
pMDI+10L B	231.40000
pMDI+20L C	199.30000

Levels not connected by same letter are significantly different.

DSC analysis - pMDI and 5 weight % lignin substitution (pMDI+5lignin) Onset temperature - pMDI+5lignin resin and residues

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	2	55.387400	27.6937	27.5258	0.0009*
Error	6	6.036600	1.0061		
C. Total	8	61.424000			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-5L	3	109.763	0.57911	108.35	111.18
flax shive-5L	3	110.733	0.57911	109.32	112.15
wood-5L	3	105.053	0.57911	103.64	106.47

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
3.06815	0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
flax shive-5L A	110.73333
hemp hurd-5L A	109.76333
wood-5L B	105.05333

Onset temperature – comparison of neat pMDI+5lignin resin and residues

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	3	58.625291	19.5418	21.6259	0.0006*
Error	7	6.325400	0.9036		
C. Total	10	64.950691			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-5L	3	109.763	0.54883	108.47	111.06
pMDI+5L	2	107.110	0.67217	105.52	108.70
flax shive-5L	3	110.733	0.54883	109.44	112.03
wood-5L	3	105.053	0.54883	103.76	106.35

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
3.31014	0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
flax shive-5L A	110.73333
hemp hurd-5L A B	109.76333
pMDI+5L B C	107.11000
wood-5L C	105.05333

Peak temperature - pMDI+5lignin resin and residues

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	2	4.641867	2.32093	0.7022	0.5321
Error	6	19.832533	3.30542		
C. Total	8	24.474400			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-5L	3	134.437	1.0497	131.87	137.01
flax shive-5L	3	134.670	1.0497	132.10	137.24
wood-5L	3	136.063	1.0497	133.49	138.63

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
3.06815	0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
wood-5L A	136.06333
flax shive-5L A	134.67000
hemp hurd-5L A	134.43667

Levels not connected by same letter are significantly different.

Peak temperature – comparison of neat pMDI+5lignin resin and residues

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	3	126.41903	42.1397	14.8613	0.0020*
Error	7	19.84873	2.8355		
C. Total	10	146.26776			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-5L	3	134.437	0.9722	132.14	136.74
pMDI+5L	2	126.430	1.1907	123.61	129.25
flax shive-5L	3	134.670	0.9722	132.37	136.97
wood-5L	3	136.063	0.9722	133.76	138.36

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
3.31014	0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
wood-5L	A 136.06333
flax shive-5L	A 134.67000
hemp hurd-5L	A 134.43667
pMDI+5L	B 126.43000

Levels not connected by same letter are significantly different.

Reaction heat - pMDI+5lignin resin and residues

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	2	381.04889	190.524	8.0902	0.0198*
Error	6	141.30000	23.550		
C. Total	8	522.34889			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-5L	3	118.033	2.8018	111.18	124.89
flax shive-5L	3	122.433	2.8018	115.58	129.29
wood-5L	3	106.967	2.8018	100.11	113.82

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
3.06815	0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
flax shive-5L	A 122.43333
hemp hurd-5L	A B 118.03333
wood-5L	B 106.96667

Levels not connected by same letter are significantly different.

Reaction heat – comparison of neat pMDI+5lignin resin and residues

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	3	31140.556	10380.2	483.6141	<.0001*
Error	7	150.246	21.5		
C. Total	10	31290.802			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-5L	3	118.033	2.6748	111.71	124.36
pMDI+5L	2	252.915	3.2760	245.17	260.66
flax shive-5L	3	122.433	2.6748	116.11	128.76
wood-5L	3	106.967	2.6748	100.64	113.29

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha
3.31014 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
pMDI+5L A	252.91500
flax shive-5L B	122.43333
hemp hurd-5L B C	118.03333
wood-5L C	106.96667

Levels not connected by same letter are significantly different.

DSC analysis - pMDI and 10 weight % lignin substitution (pMDI+10lignin)

Onset temperature - pMDI+10lignin resin and residues

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	2	48.200022	24.1000	44.8057	0.0002*
Error	6	3.227267	0.5379		
C. Total	8	51.427289			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-10L	3	109.010	0.42343	107.97	110.05
flax shive-10L	3	110.013	0.42343	108.98	111.05
wood-10L	3	104.680	0.42343	103.64	105.72

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
3.06815	0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
flax shive-10L	A	110.01333
hemp hurd-10L	A	109.01000
wood-10L	B	104.68000

Levels not connected by same letter are significantly different.

Onset temperature – comparison of neat pMDI+10lignin resin and residues

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	3	49.499424	16.4998	11.4670	0.0043*
Error	7	10.072267	1.4389		
C. Total	10	59.571691			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-10L	3	109.010	0.69255	107.37	110.65
pMDI+10L	2	107.010	0.84820	105.00	109.02
flax shive-10L	3	110.013	0.69255	108.38	111.65
wood-10L	3	104.680	0.69255	103.04	106.32

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
3.31014	0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
flax shive-10L	A	110.01333
hemp hurd-10L	A	109.01000
pMDI+10L	A B	107.01000
wood-10L	B	104.68000

Levels not connected by same letter are significantly different.

Peak temperature - pMDI+10lignin resin and residues

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	2	0.6402667	0.32013	0.2165	0.8113
Error	6	8.8711333	1.47852		
C. Total	8	9.5114000			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-10L	3	132.337	0.70203	130.62	134.05
flax shive-10L	3	132.943	0.70203	131.23	134.66
wood-10L	3	132.430	0.70203	130.71	134.15

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
3.06815	0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
flax shive-10L	A	132.94333
wood-10L	A	132.43000
hemp hurd-10L	A	132.33667

Levels not connected by same letter are significantly different.

Peak temperature – comparison of neat pMDI+10lignin resin and residues

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	3	110.13328	36.7111	28.3142	0.0003*
Error	7	9.07593	1.2966		
C. Total	10	119.20922			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-10L	3	132.337	0.65741	130.78	133.89
pMDI+10L	2	124.390	0.80516	122.49	126.29
flax shive-10L	3	132.943	0.65741	131.39	134.50
wood-10L	3	132.430	0.65741	130.88	133.98

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha
3.31014 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
flax shive-10L	A
wood-10L	A
hemp hurd-10L	A
pMDI+10L	B

Levels not connected by same letter are significantly different.

Reaction heat - pMDI+10lignin resin and residues

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	2	57.55556	28.7778	2.1842	0.1938
Error	6	79.05333	13.1756		
C. Total	8	136.60889			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-10L	3	105.433	2.0957	100.31	110.56
flax shive-10L	3	111.433	2.0957	106.31	116.56
wood-10L	3	107.100	2.0957	101.97	112.23

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha
3.06815 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
flax shive-10L A	111.43333
wood-10L A	107.10000
hemp hurd-10L A	105.43333

Levels not connected by same letter are significantly different.

Reaction heat – comparison of neat pMDI+10lignin resin and residues

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	3	24979.868	8326.62	480.2238	<.0001*
Error	7	121.373	17.34		
C. Total	10	25101.242			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-10L	3	105.433	2.4041	99.75	111.12
pMDI+10L	2	231.400	2.9444	224.44	238.36
flax shive-10L	3	111.433	2.4041	105.75	117.12
wood-10L	3	107.100	2.4041	101.42	112.78

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha
3.31014 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
pMDI+10L A	231.40000
flax shive-10L B	111.43333
wood-10L B	107.10000
hemp hurd-10L B	105.43333

Levels not connected by same letter are significantly different.

**DSC analysis - pMDI and 20 weight % lignin substitution (pMDI+20lignin)
Onset temperature - pMDI+20lignin resin and residues**

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	2	45.545400	22.7727	26.4737	0.0011*
Error	6	5.161200	0.8602		
C. Total	8	50.706600			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-20L	3	108.170	0.53547	106.86	109.48
flax shive-20L	3	109.550	0.53547	108.24	110.86
wood-20L	3	104.240	0.53547	102.93	105.55

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha
3.06815 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
flax shive-20L	A	109.55000
hemp hurd-20L	A	108.17000
wood-20L	B	104.24000

Levels not connected by same letter are significantly different.

Onset temperature – comparison of neat pMDI+20lignin resin and residues

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	3	48.821768	16.2739	20.0868	0.0008*
Error	7	5.671250	0.8102		
C. Total	10	54.493018			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-20L	3	108.170	0.51967	106.94	109.40
pMDI+20L	2	108.735	0.63647	107.23	110.24
flax shive-20L	3	109.550	0.51967	108.32	110.78
wood-20L	3	104.240	0.51967	103.01	105.47

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
3.31014	0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
flax shive-20L	A 109.55000
pMDI+20L	A 108.73500
hemp hurd-20L	A 108.17000
wood-20L	B 104.24000

Peak temperature - pMDI+20lignin resin and residues

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	2	4.120622	2.06031	0.6869	0.5387
Error	6	17.996667	2.99944		
C. Total	8	22.117289			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-20L	3	130.773	0.99991	128.33	133.22
flax shive-20L	3	131.280	0.99991	128.83	133.73
wood-20L	3	129.660	0.99991	127.21	132.11

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
3.06815	0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
flax shive-20L	A 131.28000
hemp hurd-20L	A 130.77333
wood-20L	A 129.66000

Peak temperature – comparison of neat pMDI+20lignin resin and residues

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	3	85.82414	28.6080	11.0852	0.0047*
Error	7	18.06512	2.5807		
C. Total	10	103.88925			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-20L	3	130.773	0.9275	128.58	132.97
pMDI+20L	2	123.505	1.1359	120.82	126.19
flax shive-20L	3	131.280	0.9275	129.09	133.47
wood-20L	3	129.660	0.9275	127.47	131.85

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha
3.31014 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
flax shive-20L A	131.28000
hemp hurd-20L A	130.77333
wood-20L A	129.66000
pMDI+20L B	123.50500

Levels not connected by same letter are significantly different.

Reaction heat - pMDI+20lignin resin and residues

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	2	19.421956	9.71098	1.8956	0.2301
Error	6	30.738133	5.12302		
C. Total	8	50.160089			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-20L	3	103.133	1.3068	99.936	106.33
flax shive-20L	3	102.020	1.3068	98.822	105.22
wood-20L	3	99.613	1.3068	96.416	102.81

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
3.06815	0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
hemp hurd-20L	A 103.13333
flax shive-20L	A 102.02000
wood-20L	A 99.61333

Levels not connected by same letter are significantly different.

Reaction heat – comparison of neat pMDI+20lignin resin and residues

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	3	15642.540	5214.18	805.4008	<.0001*
Error	7	45.318	6.47		
C. Total	10	15687.858			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-20L	3	103.133	1.4690	99.66	106.61
pMDI+20L	2	199.300	1.7992	195.05	203.55
flax shive-20L	3	102.020	1.4690	98.55	105.49
wood-20L	3	99.613	1.4690	96.14	103.09

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q*	Alpha
3.31014	0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
pMDI+20L	A 199.30000
hemp hurd-20L	B 103.13333
flax shive-20L	B 102.02000
wood-20L	B 99.61333

Levels not connected by same letter are significantly different.

Internal bond – pMDI-lignin particleboards

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample	5	2.0047571	0.400951	66.8552	<.0001*
Error	186	1.1154990	0.005997		
C. Total	191	3.1202561			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-20L	32	0.623169	0.01369	0.59616	0.65018
hemp hurd-5L	32	0.804734	0.01369	0.77773	0.83174
flax shive-20L	32	0.501123	0.01369	0.47411	0.52813
flax shive-5L	32	0.592696	0.01369	0.56569	0.61970
wood-20L	32	0.498009	0.01369	0.47100	0.52502
wood-5L	32	0.596883	0.01369	0.56988	0.62389

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.87966 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
hemp hurd-5L	A 0.80473359
hemp hurd-20L	B 0.62316889
wood-5L	B 0.59688300
flax shive-5L	B 0.59269646
flax shive-20L	C 0.50112252
wood-20L	C 0.49800879

Levels not connected by same letter are significantly different.

Modulus of rupture – pMDI-lignin particleboard

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Board	5	1058.4731	211.695	102.066	<.0001*
Type				6	
Error	65	134.8155	2.074		
C. Total	70	1193.2886			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-20L	12	10.2090	0.41574	9.379	11.039
hemp hurd-5L	12	10.7235	0.41574	9.893	11.554
flax shive-20L	12	9.5736	0.41574	8.743	10.404
flax shive-5L	11	11.7007	0.43423	10.834	12.568
wood-20L	12	2.5695	0.41574	1.739	3.400
wood-5L	12	2.3774	0.41574	1.547	3.208

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.93643 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
flax shive-5L	A 11.700741
hemp hurd-5L	A B 10.723491
hemp hurd-20L	A B 10.208989
flax shive-20L	B 9.573646
wood-20L	C 2.569493
wood-5L	C 2.377419

Levels not connected by same letter are significantly different.

Modulus of elasticity – pMDI-lignin particleboards

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Board	5	2.6735065	0.534701	65.5968	<.0001*
Type					
Error	65	0.5298365	0.008151		
C. Total	70	3.2033430			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-20L	12	0.23896	0.02606	0.1869	0.2910
hemp hurd-5L	12	0.27148	0.02606	0.2194	0.3235
flax shive-20L	12	0.29727	0.02606	0.2452	0.3493
flax shive-5L	11	0.37043	0.02722	0.3161	0.4248
wood-20L	12	-0.11101	0.02606	-0.1631	-0.0590
wood-5L	12	-0.10702	0.02606	-0.1591	-0.0550

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.93643 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
flax shive-5L A	0.3704315
flax shive-20L A B	0.2972723
hemp hurd-5L A B	0.2714808
hemp hurd-20L B	0.2389559
wood-5L C	-0.1070160
wood-20L C	-0.1110148

Levels not connected by same letter are significantly different.

Linear expansion – pMDI-lignin particleboards

Analysis of Variance- Logarithm transformation

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample	5	2.5238767	0.504775	458.5904	<.0001*
Error	90	0.0990640	0.001101		
C. Total	95	2.6229407			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-20L	16	-0.38838	0.00829	-0.4049	-0.3719
hemp hurd-5L	16	-0.49294	0.00829	-0.5094	-0.4765
flax shive-20L	16	-0.58128	0.00829	-0.5978	-0.5648
flax shive-5L	16	-0.64817	0.00829	-0.6646	-0.6317
wood-20L	16	-0.21391	0.00829	-0.2304	-0.1974
wood-5L	16	-0.24319	0.00829	-0.2597	-0.2267

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.91203 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
wood-20L	A	-0.2139143
wood-5L	A	-0.2431907
hemp hurd-20L	B	-0.3883798
hemp hurd-5L	C	-0.4929448
flax shive-20L	D	-0.5812767
flax shive-5L	E	-0.6481677

Levels not connected by same letter are significantly different.

Thickness swell – pMDI-lignin particleboards (2 hours)

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Samples	5	468.94066	93.7881	131.5052	<.0001*
Error	66	47.07052	0.7132		
C. Total	71	516.01119			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-20L	12	11.6177	0.24379	11.131	12.104
hemp hurd-5L	12	6.6414	0.24379	6.155	7.128
flax shive-20L	12	6.9608	0.24379	6.474	7.448
flax shive-5L	12	5.2772	0.24379	4.790	5.764
wood-20L	12	4.1622	0.24379	3.676	4.649
wood-5L	12	4.1118	0.24379	3.625	4.599

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.93510 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
hemp hurd-20L	A	11.617707
flax shive-20L	B	6.960774
hemp hurd-5L	B	6.641394
flax shive-5L	C	5.277212
wood-20L	D	4.162243
wood-5L	D	4.111784

Levels not connected by same letter are significantly different.

Thickness swell – pMDI-lignin particleboards (24 hours)

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Samples	5	6077.6341	1215.53	462.9502	<.0001*
Error	66	173.2903	2.63		
C. Total	71	6250.9244			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-20L	12	38.4745	0.46776	37.541	39.408
hemp hurd-5L	12	18.4844	0.46776	17.551	19.418
flax shive-20L	12	19.3375	0.46776	18.404	20.271
flax shive-5L	12	15.5952	0.46776	14.661	16.529
wood-20L	12	11.7092	0.46776	10.775	12.643
wood-5L	12	11.1151	0.46776	10.181	12.049

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.93510 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
hemp hurd-20L	A
flax shive-20L	B
hemp hurd-5L	B
flax shive-5L	C
wood-20L	D
wood-5L	D

Levels not connected by same letter are significantly different.

Water absorption – pMDI-lignin particleboards (2 hours)

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Samples	5	3047.1387	609.428	347.0993	<.0001*
Error	66	115.8810	1.756		
C. Total	71	3163.0198			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-20L	12	30.0627	0.38251	29.299	30.826
hemp hurd-5L	12	16.1042	0.38251	15.340	16.868
flax shive-20L	12	17.5820	0.38251	16.818	18.346
flax shive-5L	12	13.5960	0.38251	12.832	14.360
wood-20L	12	11.3020	0.38251	10.538	12.066
wood-5L	12	10.7358	0.38251	9.972	11.500

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.93510 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
hemp hurd-20L	A 30.062718
flax shive-20L	B 17.581987
hemp hurd-5L	B 16.104202
flax shive-5L	C 13.595975
wood-20L	D 11.302004
wood-5L	D 10.735848

Levels not connected by same letter are significantly different.

Water absorption – pMDI-lignin particleboards (24 hours)

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Samples	5	51001.468	10200.3	528.9538	<.0001*
Error	66	1272.738	19.3		
C. Total	71	52274.206			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hemp hurd-20L	12	114.127	1.2677	111.60	116.66
hemp hurd-5L	12	52.561	1.2677	50.03	55.09
flax shive-20L	12	48.743	1.2677	46.21	51.27
flax shive-5L	12	40.713	1.2677	38.18	43.24
wood-20L	12	39.651	1.2677	37.12	42.18
wood-5L	12	38.495	1.2677	35.96	41.03

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.93510 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
hemp hurd-20L	A	114.12669
hemp hurd-5L	B	52.56149
flax shive-20L	B	48.74273
flax shive-5L	C	40.71288
wood-20L	C	39.65108
wood-5L	C	38.49506

Levels not connected by same letter are significantly different.

Appendix E: statistical analysis - chapter 6

Moisture content – fiber-reinforced particleboards

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Board Type	3	0.322028	0.10734	0.0595	0.9807
Error	39	70.389797	1.80487		
C. Total	42	70.711825			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hh-h	10	9.70415	0.42484	8.8448	10.563
fs-f	11	9.57592	0.40507	8.7566	10.395
w-f	11	9.55750	0.40507	8.7382	10.377
w-h	11	9.76213	0.40507	8.9428	10.581

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.68337 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
w-h A	9.7621290
hh-h A	9.7041475
fs-f A	9.5759204
w-f A	9.5575003

Levels not connected by same letter are significantly different.

Average board density – fiber-reinforced particleboards

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Board Type	3	6194.919	2064.97	1.8780	0.1493
Error	39	42882.749	1099.56		
C. Total	42	49077.669			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hh-h	10	765.864	10.486	744.65	787.07
fs-f	11	794.168	9.998	773.95	814.39
w-f	11	775.310	9.998	755.09	795.53
w-h	11	764.055	9.998	743.83	784.28

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.68337 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level	Mean
fs-f A	794.16801
w-f A	775.30959
hh-h A	765.86403
w-h A	764.05462

Levels not connected by same letter are significantly different.

Internal bond – fiber-reinforced particleboards

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Board Type	3	1.684823	0.561608	3.7019	0.0136*
Error	124	18.811781	0.151708		
C. Total	127	20.496605			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
hh-h	32	1.03604	0.06885	0.8998	1.1723
fs-f	32	1.29340	0.06885	1.1571	1.4297
w-f	32	1.07215	0.06885	0.9359	1.2084
w-h	32	1.26961	0.06885	1.1333	1.4059

Comparisons for all pairs using Tukey-Kramer HSD

Confidence Quantile

q* Alpha

2.60422 0.05

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level		Mean
fs-f	A	1.2933974
w-h	A B	1.2696124
w-f	A B	1.0721519
hh-h	B	1.0360362

Levels not connected by same letter are significantly different.

Internal bond - analysis of covariance Wood fiber-reinforced particleboards

Multiple Comparisons for Board Type

Least Squares Means Estimates

Standard errors are based on square transformed response.

Board Type	Estimate	Std Error	DF	Lower 95%	Upper 95%
w	1.6791866	0.15939737	92	1.5821159	1.7709445
w-f	0.9843755	0.12642612	92	0.8472909	1.1045761
w-h	1.1688873	0.13256166	92	1.0502469	1.2765486

Tukey HSD All Pairwise Comparisons

Quantile = 2.38225 , Adjusted DF = 92.0 , Adjustment = Tukey-Kramer

Differences are based on transformed response.

All Pairwise Differences

Board Type	-Board Type	Difference	Std Error	t Ratio	Prob> t	Lower 95%	Upper 95%
w	w-f	1.85067	0.2266730	8.16	<.0001*	1.31068	2.390665
w	w-h	1.45337	0.2369552	6.13	<.0001*	0.88888	2.017857
w-f	w-h	-0.39730	0.1669607	-2.38	0.0503	-0.79504	0.000440

Level		Least Sq Mean
w	A	1.6791866
w-h	B	1.1688873
w-f	B	0.9843755

Levels not connected by same letter are significantly different

Internal bond - analysis of covariance

Hemp hurd fiber-reinforced particleboards

Multiple Comparisons for Board Type

Least Squares Means Estimates

Board Type	Estimate	Std Error	DF	Lower 95%	Upper 95%
h	1.2773607	0.07644961	61	1.1244903	1.4302312
hh	0.9056917	0.07644961	61	0.7528213	1.0585621

Tukey HSD All Pairwise Comparisons

Quantile = 1.99968 , Adjusted DF = 61.0 , Adjustment = Tukey-Kramer

All Pairwise Differences

Board Type	Board Type	Difference	Std Error	t Ratio	Prob> t	Lower 95%	Upper 95%
h	hh	0.3716690	0.1370395	2.71	0.0087*	0.0976333	0.6457048

Level	Least Sq Mean
h A	1.2773607
hh B	0.9056917

Levels not connected by same letter are significantly different.

Internal bond - analysis of covariance

Flax shive fiber-reinforced particleboards

Multiple Comparisons for Board Type

Least Squares Means Estimates

Board Type	Estimate	Std Error	DF	Lower 95%	Upper 95%
fs	1.3105975	0.07724770	60	1.1560791	1.4651159
fs-f	0.7084195	0.06592059	60	0.5765587	0.8402803

Tukey HSD All Pairwise Comparisons

Quantile = 2.00036 , Adjusted DF = 60.0 , Adjustment = Tukey-Kramer

All Pairwise Differences

Board Type	-Board Type	Difference	Std Error	t Ratio	Prob> t	Lower 95%	Upper 95%
S	SF	0.6021780	0.1015516	5.93	<.0001*	0.3990385	0.8053175

Modulus of rupture – fiber-reinforced particleboard

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	1610.5098	536.837	12.2835
Error	52	2272.5944	43.704	Prob > F
C. Total	55	3883.1042		<.0001*

Least Squares Means Table

Level	Least Sq Mean	Std Error	Lower 95%	Upper 95%	Mean
hh-h	34.607764	1.7668321	31.062356	38.153172	34.6078
fs-f	25.199717	1.7668321	21.654309	28.745125	25.1997
w-f	21.011605	1.7668321	17.466197	24.557013	21.0116
w-h	22.031558	1.7668321	18.486150	25.576966	22.0316

Level		Least Sq Mean
hh-h	A	34.607764
fs-f	B	25.199717
w-h	B	22.031558
w-f	B	21.011605

Levels not connected by same letter are significantly different.

Modulus of elasticity – fiber-reinforced particleboard

Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Board Type	3	3	22779902	12.0196	<.0001*

Least Squares Means Table

Level	Least Sq Mean	Std Error	Lower 95%	Upper 95%	Mean
hh-h	4875.7112	212.42505	4449.4491	5301.9732	4875.71
fs-f	4606.1988	212.42505	4179.9368	5032.4608	4606.20
w-f	3501.2568	212.42505	3074.9947	3927.5188	3501.26
w-h	3458.8151	212.42505	3032.5531	3885.0772	3458.82

Level		Least Sq Mean
hh-h	A	4875.7112
fs-f	A	4606.1988
w-f	B	3501.2568
w-h	B	3458.8151

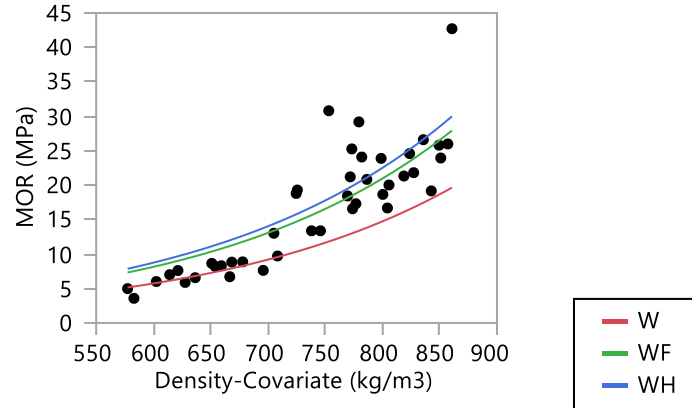
Levels not connected by same letter are significantly different.

Modulus of rupture and elasticity -analysis of covariance Wood fiber-reinforced particleboards

Response Log(MOR (MPa))

Whole Model

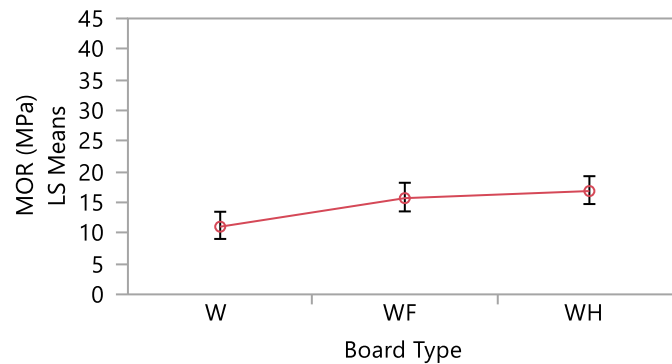
Regression Plot



Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Board Type	2	2	0.4189957	4.6327	0.0158*
Density-Covariate (kg/m3)	1	1	1.5720950	34.7646	<.0001*

LS Means Plot



LSMeans Differences Tukey HSD

Differences are on transformed Y's

$\alpha = 0.050$ $Q = 2.43883$

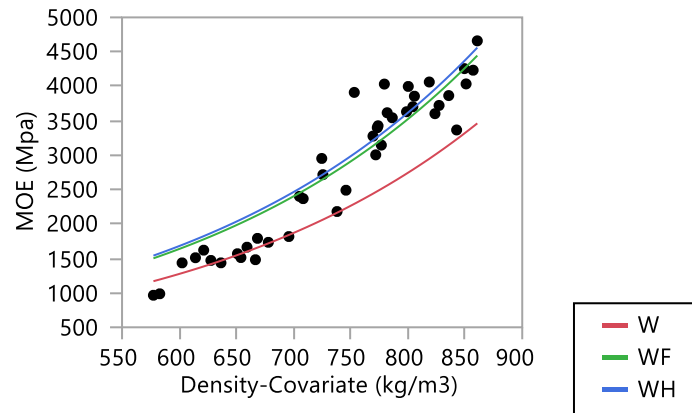
Level	Least Sq Mean
w-h A	16.852470
w-f A B	15.694807
w B	11.038838

Levels not connected by same letter are significantly different.

Response Log(MOE (Mpa))

Whole Model

Regression Plot



Effect Tests

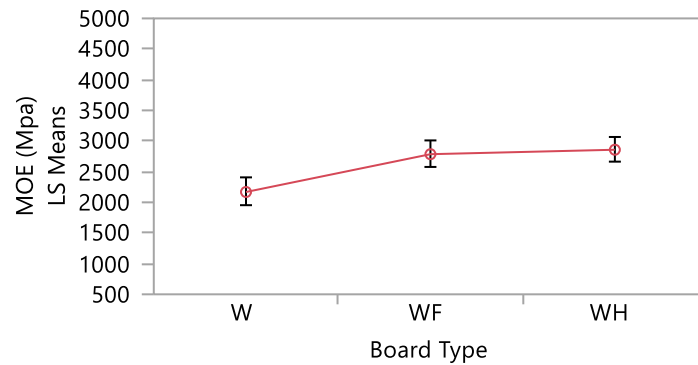
Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Board Type	2	2	0.1753894	7.0791	0.0024*
Density-Covariate (kg/m3)	1	1	1.0336852	83.4438	<.0001*

Least Squares Means Table

Level	Least Sq Mean	Std Error	Mean
w	2168.7195	0.05155969	1476.15
w-f	2785.2749	0.03831975	3472.99
w-h	2857.8759	0.03474599	3367.29

* Std Errors are on transformed Y's

LS Means Plot



LSMeans Differences Tukey HSD

Differences are on transformed Y's

$\alpha = 0.050$ $Q = 2.43883$

Level		Least Sq Mean
w-h	A	2857.8759
w-f	A	2785.2749
w	B	2168.7195

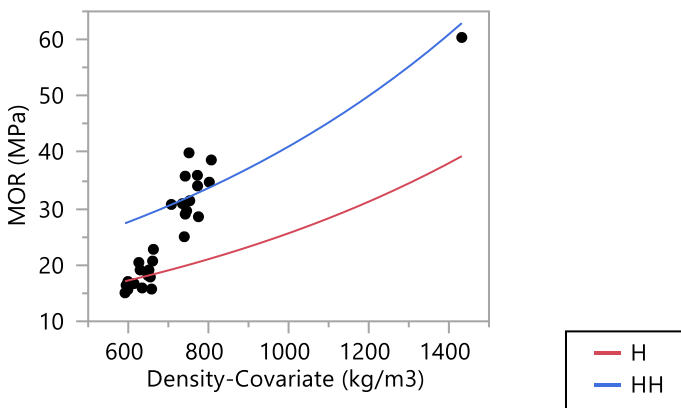
Levels not connected by same letter are significantly different

Modulus of rupture and elasticity - analysis of covariance Hemp hurd fiber-reinforced particleboards

Response Log(MOR (MPa))

Whole Model

Regression Plot



Effect Tests

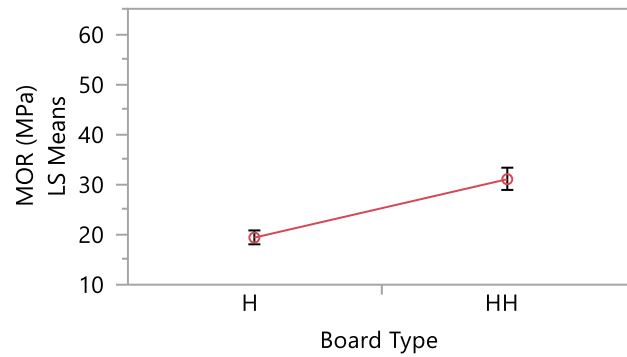
Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Board Type	1	1	1.0417958	77.0986	<.0001*
Density-Covariate (kg/m3)	1	1	0.4321789	31.9836	<.0001*

Least Squares Means Table

Level	Least Sq Mean	Std Error	Mean
hh	19.385077	0.03464258	17.7755
hh-h	31.028514	0.03464258	33.8382

* Std Errors are on transformed Y's

LS Means Plot



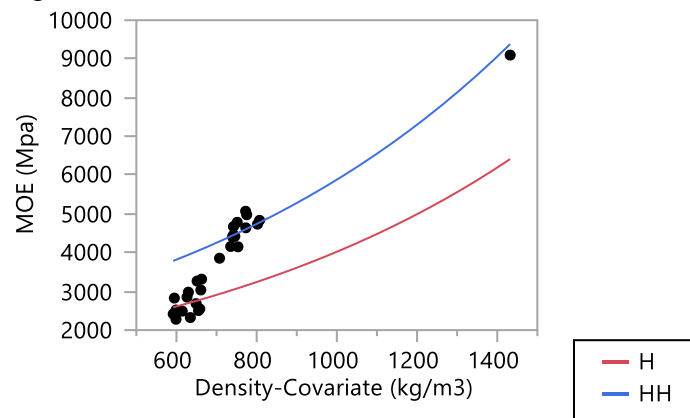
LSMeans Differences Student's t
Differences are on transformed Y's
 $\alpha = 0.050$ $t = 2.05954$

Level	Least Sq Mean
hh-h A	31.028514
hh B	19.385077

Response Log(MOE (Mpa))

Whole Model

Regression Plot



Parameter Estimates

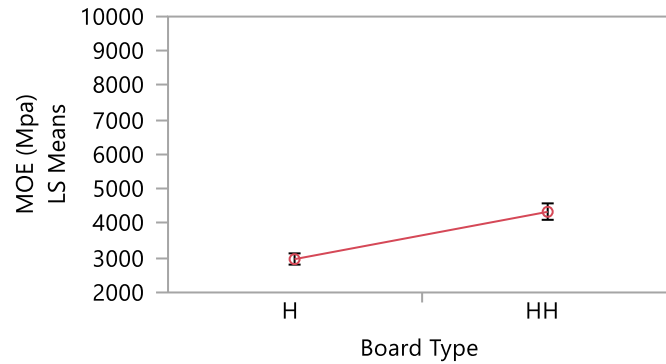
Term	Estimate	Std Error	t Ratio	Prob> t
Intercept	7.4095259	0.098232	75.43	<.0001*
Board Type[H]	-0.190134	0.020622	-9.22	<.0001*
Density-Covariate (kg/m3)	0.0010802	0.000135	8.02	<.0001*

Least Squares Means Table

Level	Least Sq Mean	Std Error	Mean
hh	2966.2961	0.02667004	2698.58
hh-h	4338.7326	0.02667004	4769.17

* Std Errors are on transformed Y's

LS Means Plot



LSMeans Differences Student's t

Differences are on transformed Y's

$\alpha = 0.050$ $t = 2.05954$

Level	Least Sq Mean
hh-h A	4338.7326
hh B	2966.2961

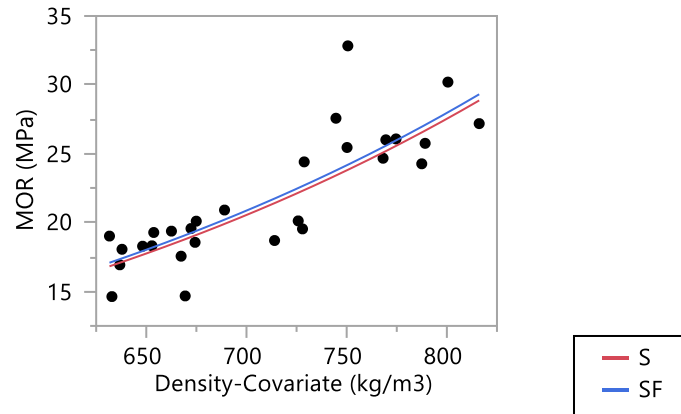
Levels not connected by same letter are significantly different

Modulus of rupture and elasticity - analysis of covariance Flax shive fiber-reinforced particleboards

Response Log(MOR (MPa))

Whole Model

Regression Plot



Effect Tests

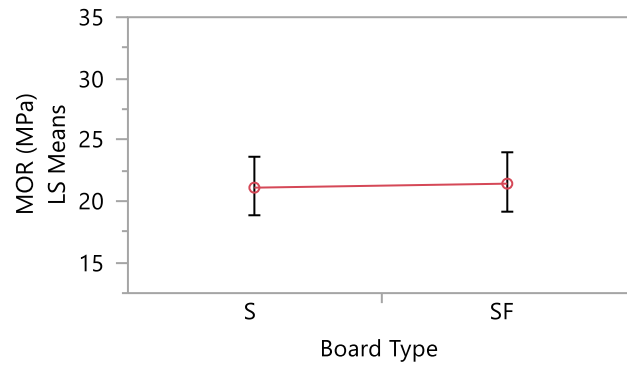
Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Board Type	1	1	0.00030323	0.0233	0.8800
Density-Covariate (kg/m3)	1	1	0.14336908	10.9968	0.0028*

Least Squares Means Table

Level	Least Sq Mean	Std Error	Mean
fs	21.099177	0.05478026	18.1446
fs-f	21.425720	0.05478026	24.9146

* Std Errors are on transformed Y's

LS Means Plot

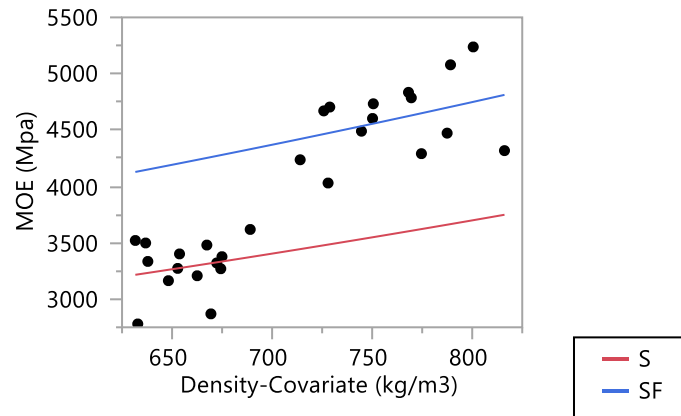


LSMeans Differences Student's t
Differences are on transformed Y's
 $\alpha = 0.050$ $t = 2.05954$

Level	Least Sq Mean
fs-f A	21.425720
fs A	21.099177

Levels not connected by same letter are significantly different.

Response Log(MOE (Mpa))
Whole Model
Regression Plot



Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Board Type	1	1	0.08023213	15.7807	0.0005*
Density-Covariate (kg/m3)	1	1	0.01156269	2.2742	0.1441

Least Squares Means Table

Level	Least Sq Mean	Std Error	Mean
fs	3429.3109	0.03420898	3285.49
fs-f	4402.5121	0.03420898	4595.23

* Std Errors are on transformed Y's

Level	Least Sq Mean
fs-f A	4402.5121
fs B	3429.3109

Levels not connected by same letter are significantly different.

Thickness swell – fiber-reinforced particleboard (2 hours)

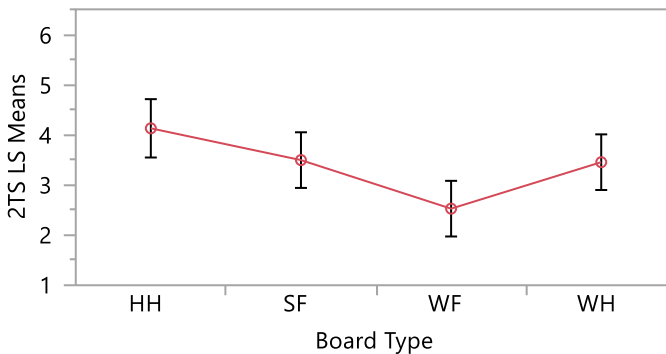
Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	13.810049	4.60335	5.5669
Error	39	32.249668	0.82691	Prob > F
C. Total	42	46.059717		0.0028*

Least Squares Means Table

Level	Least Sq Mean	Std Error	Lower 95%	Upper 95%	Mean
hh-h	4.1281838	0.28756122	3.5465363	4.7098312	4.12818
fs-f	3.4946795	0.27417887	2.9401004	4.0492586	3.49468
w-f	2.5267122	0.27417887	1.9721331	3.0812913	2.52671
w-h	3.4533085	0.27417887	2.8987294	4.0078876	3.45331

LS Means Plot



LSMeans Differences Tukey HSD

$\alpha = 0.050$ $Q = 2.68337$

Level		Least Sq Mean
hh-h	A	4.1281838
fs-f	A B	3.4946795
w-h	A B	3.4533085
w-f	B	2.5267122

Thickness swell – fiber-reinforced particleboard (24 hours)

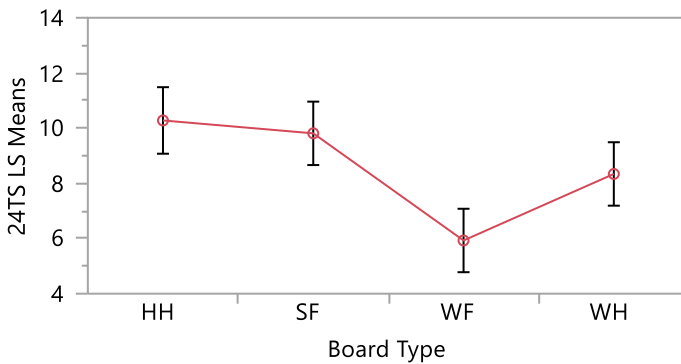
Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	123.49685	41.1656	11.5866
Error	39	138.56226	3.5529	Prob > F
C. Total	42	262.05912		<.0001*

Least Squares Means Table

Level	Least Sq Mean	Std Error	Lower 95%	Upper 95%	Mean
hh-h	10.278517	0.59606027	9.0728714	11.484163	10.2785
fs-f	9.811489	0.56832117	8.6619514	10.961028	9.8115
w-f	5.927975	0.56832117	4.7784374	7.077514	5.9280
w-h	8.339138	0.56832117	7.1895998	9.488676	8.3391

LS Means Plot



LSMeans Differences Tukey HSD

 $\alpha = 0.050$ $Q = 2.68337$

Level		Least Sq Mean
hh-h	A	10.278517
fs-f	A	9.811489
w-h	A	8.339138
w-f	B	5.927975

Levels not connected by same letter are significantly different.

Water absorption – fiber-reinforced particleboard (2 hours)

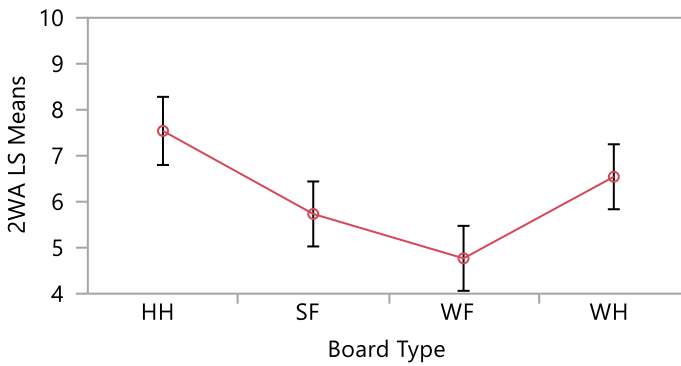
Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	43.862802	14.6209	10.8910
Error	39	52.356521	1.3425	Prob > F
C. Total	42	96.219323		<.0001*

Least Squares Means Table

Level	Least Sq Mean	Std Error	Lower 95%	Upper 95%	Mean
hh-h	7.5398011	0.36639799	6.7986913	8.2809110	7.53980
fs-f	5.7335269	0.34934678	5.0269064	6.4401475	5.73353
w-f	4.7682448	0.34934678	4.0616242	5.4748653	4.76824
w-h	6.5424999	0.34934678	5.8358794	7.2491205	6.54250

LS Means Plot



LSMeans Differences Tukey HSD

 $\alpha = 0.050$ $Q = 2.68337$

Level	Least Sq Mean
hh-h A	7.5398011
w-h A B	6.5424999
fs-f B C	5.7335269
w-f C	4.7682448

Levels not connected by same letter are significantly different.

Water absorption – fiber-reinforced particleboard (24 hours)

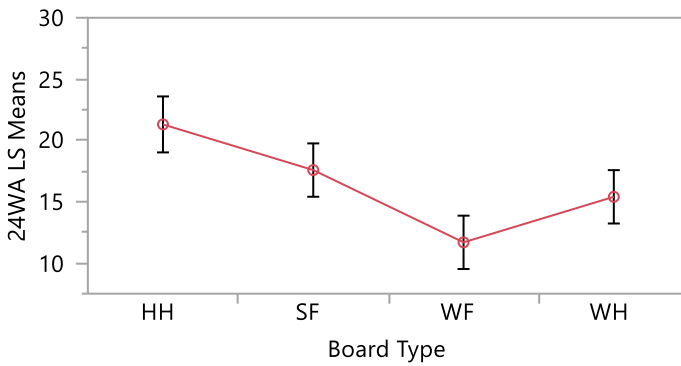
Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	511.1400	170.380	13.3918
Error	39	496.1844	12.723	Prob > F
C. Total	42	1007.3244		<.0001*

Least Squares Means Table

Level	Least Sq Mean	Std Error	Lower 95%	Upper 95%	Mean
hh-h	21.303210	1.1279484	19.021719	23.584701	21.3032
fs-f	17.579278	1.0754566	15.403961	19.754594	17.5793
w-f	11.686276	1.0754566	9.510960	13.861592	11.6863
w-h	15.398463	1.0754566	13.223146	17.573779	15.3985

LS Means Plot



LSMeans Differences Tukey HSD

$\alpha = 0.050$ $Q = 2.68337$

Level		Least Sq Mean
HH	A	21.303210
SF	A B	17.579278
WH	B C	15.398463
WF	C	11.686276

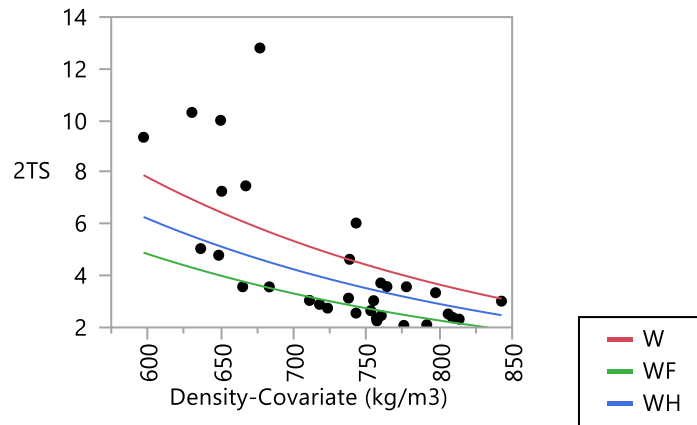
Levels not connected by same letter are significantly different.

Thickness swell (2 hours) - analysis of covariance Wood fiber-reinforced particleboards

Response Log(2TS)

Whole Model

Regression Plot



Effect Tests

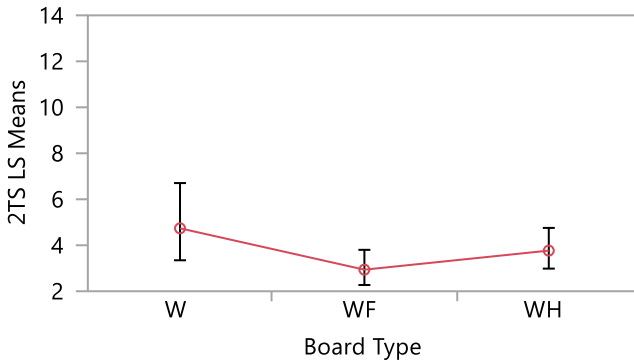
Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Board Type	2	2	0.49412393	2.3773	0.1106
Density-Covariate (kg/m3)	1	1	0.44011661	4.2349	0.0487*

Least Squares Means Table

Level	Least Sq Mean	Std Error	Lower 95%	Upper 95%	Mean
w	4.7373305	0.16986503	3.3469879	6.7052231	6.31010
w-f	2.9382563	0.12589269	2.2712648	3.8011200	2.49221
w-h	3.7671204	0.11386047	2.9845234	4.7549286	3.33436

* Std Errors are on transformed Y's

LS Means Plot



LSMeans Differences Tukey HSD

Differences are on transformed Y's

$\alpha = 0.050$ $Q = 2.46966$

Level	Least Sq Mean
w A	4.7373305
w-h A	3.7671204
w-f A	2.9382563

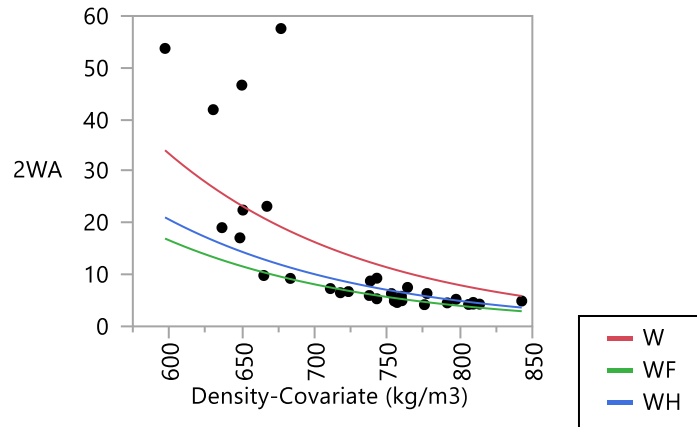
Levels not connected by same letter are significantly different.

Water absorption (2 hours) - analysis of covariance Wood fiber-reinforced particleboards

Response Log(2WA)

Whole Model

Regression Plot



Effect Tests

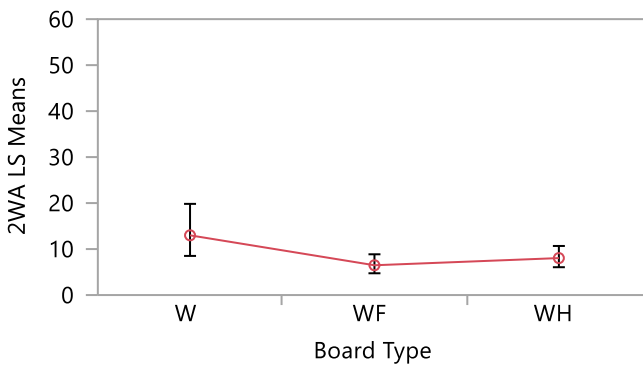
Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Board Type	2	2	0.7821098	2.5386	0.0964
Density-Covariate (kg/m3)	1	1	1.5718926	10.2043	0.0034*

Least Squares Means Table

Level	Least Sq Mean	Std Error	Lower 95%	Upper 95%	Mean
w	12.988189	0.20680527	8.5085889	19.826207	22.3275
w-f	6.478241	0.15327035	4.7349726	8.863326	4.7460
w-h	8.038541	0.13862150	6.0540944	10.673461	6.3829

* Std Errors are on transformed Y's

LS Means Plot



LSMeans Differences Tukey HSD

Differences are on transformed Y's

$\alpha = 0.050$ $Q = 2.46966$

Level	Least Sq Mean
w A	12.988189
w-h A	8.038541
w-f A	6.478241

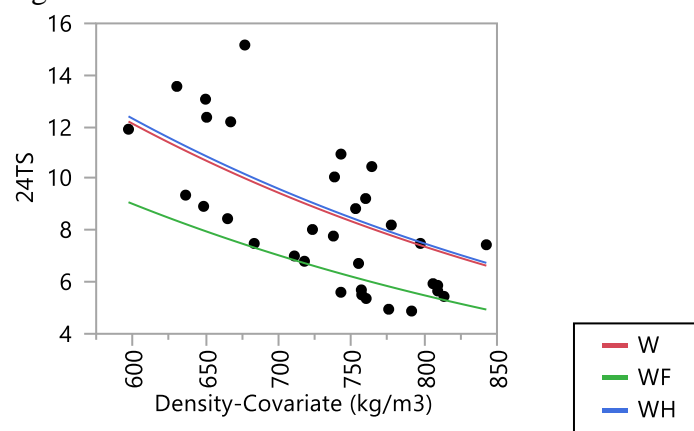
Levels not connected by same letter are significantly different.

Thickness swell (24 hours) - analysis of covariance Wood fiber-reinforced particleboards

Response Log(24TS)

Whole Model

Regression Plot



Effect Tests

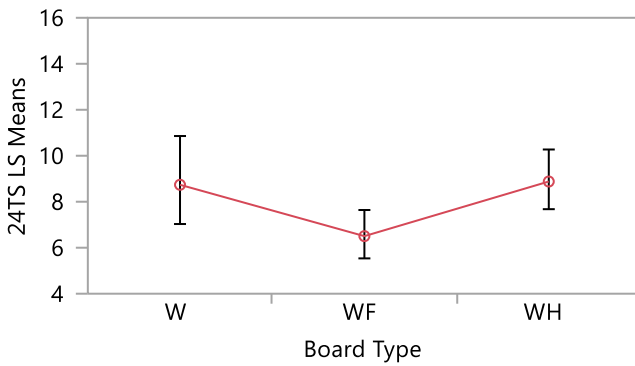
Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Board Type	2	2	0.53047583	6.5235	0.0046*
Density-Covariate (kg/m3)	1	1	0.18935077	4.6571	0.0393*

Least Squares Means Table

Level	Least Sq Mean	Std Error	Lower 95%	Upper 95%	Mean
w	8.7369400	0.10624742	7.0305127	10.857547	10.5444
w-f	6.5034107	0.07874354	5.5360328	7.639830	5.8377
w-h	8.8795358	0.07121761	7.6759564	10.271835	8.1965

* Std Errors are on transformed Y's

LS Means Plot



LSMeans Differences Tukey HSD

Differences are on transformed Y's

$\alpha = 0.050$ $Q = 2.46966$

Level	Least Sq Mean
w-h A	8.8795358
w A B	8.7369400
w-f B	6.5034107

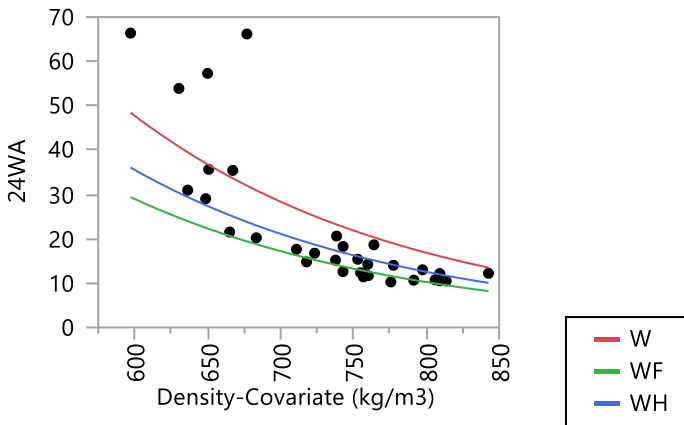
Levels not connected by same letter are significantly different.

Water absorption (42 hours) - analysis of covariance Wood fiber-reinforced particleboards

Response Log(24WA)

Whole Model

Regression Plot



Effect Tests

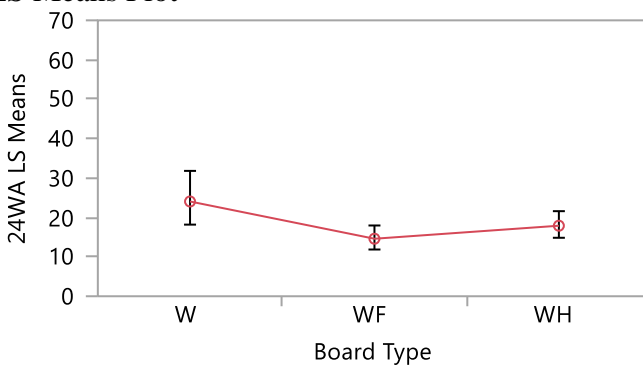
Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Board Type	2	2	0.45000993	3.3373	0.0496*
Density-Covariate (kg/m3)	1	1	0.83143178	12.3319	0.0015*

Least Squares Means Table

Level	Least Sq Mean	Std Error	Lower 95%	Upper 95%	Mean
w	24.040566	0.13681689	18.172704	31.803127	35.6506
w-f	14.590172	0.10139960	11.857525	17.952577	11.6354
w-h	17.893750	0.09170831	14.833482	21.585376	15.1307

* Std Errors are on transformed Y's

LS Means Plot



LSMeans Differences Tukey HSD

Differences are on transformed Y's

$\alpha = 0.050$ $Q = 2.46966$

Level	Least Sq Mean
W A	24.040566
WH A	17.893750
WF A	14.590172

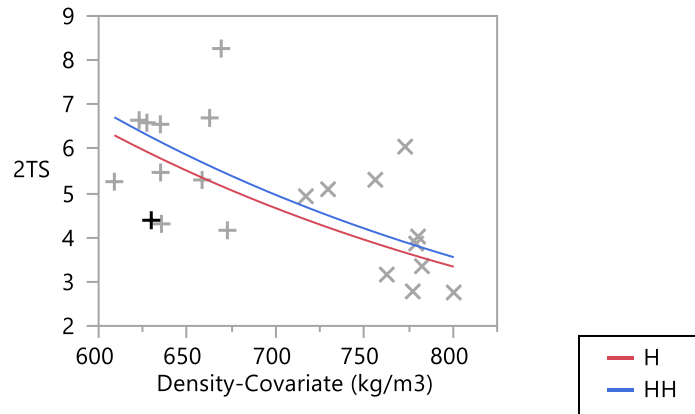
Levels not connected by same letter are significantly different.

Thickness swell (2 hours) - analysis of covariance Hemp hurd fiber-reinforced particleboards

Response Log(2TS)

Whole Model

Regression Plot



Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Board Type	1	1	0.00229061	0.0379	0.8477
Density-Covariate (kg/m3)	1	1	0.11266624	1.8661	0.1887

Least Squares Means Table

Level	Least Sq Mean	Std Error	Lower 95%	Upper 95%	Mean
hh	4.6472303	0.16188504	3.3074039	6.5298193	5.65703
hh-h	4.9468525	0.17637019	3.4151162	7.1655981	3.98469

* Std Errors are on transformed Y's

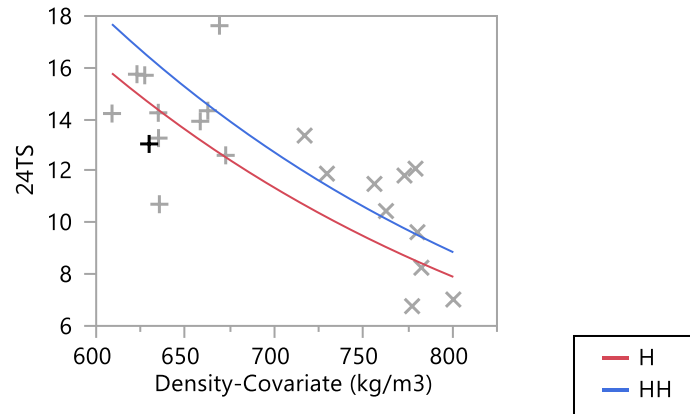
LSMeans Differences Student's t
Differences are on transformed Y's
 $\alpha = 0.050$ $t = 2.10092$

Level	Least Sq Mean
hh-h A	4.9468525
hh A	4.6472303

Levels not connected by same letter are significantly different

Thickness swell (24 hours) - analysis of covariance Hemp hurd fiber-reinforced particleboards

Response Log(24TS)
Whole Model
Regression Plot



Effect Tests

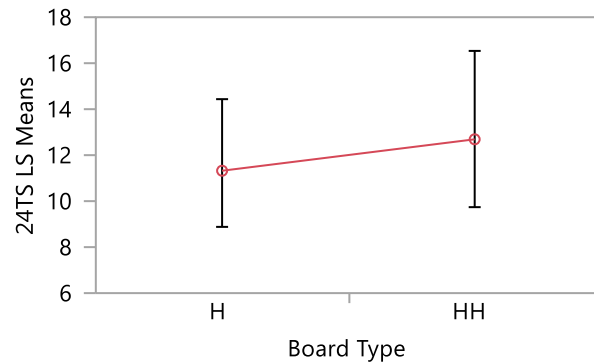
Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Board Type	1	1	0.00761864	0.2474	0.6249
Density-Covariate (kg/m3)	1	1	0.13321803	4.3256	0.0521

Least Squares Means Table

Level	Least Sq Mean	Std Error	Lower 95%	Upper 95%	Mean
hh	11.322575	0.11562097	8.8807662	14.435770	14.0217
hh-h	12.689133	0.12596650	9.7386275	16.533552	10.0297

* Std Errors are on transformed Y's

LS Means Plot



LSMeans Differences Student's t
Differences are on transformed Y's
 $\alpha = 0.050$ $t = 2.10092$

Level	Least Sq Mean
hh-h A	12.689133
hh A	11.322575

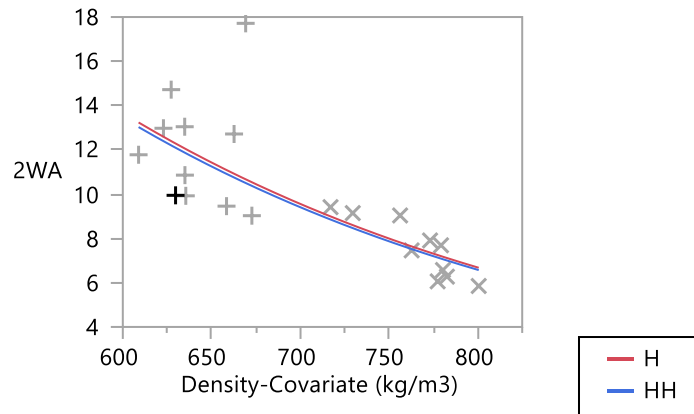
Levels not connected by same letter are significantly different.

Water absorption (2 hours) - analysis of covariance Hemp hurd fiber-reinforced particleboards

Response Log(2WA)

Whole Model

Regression Plot



Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Board Type	1	1	0.00014669	0.0046	0.9469
Density-Covariate (kg/m3)	1	1	0.13047165	4.0500	0.0594

Least Squares Means Table

Level	Least Sq Mean	Std Error	Lower 95%	Upper 95%	Mean
hh	9.5311151	0.11825308	7.4344249	12.219123	11.7771
hh-h	9.3816027	0.12883412	7.1569232	12.297808	7.4335

* Std Errors are on transformed Y's

LSMeans Differences Student's t

Differences are on transformed Y's

$\alpha = 0.050$ $t = 2.10092$

Level	Least Sq Mean
hh A	9.5311151
hh-h A	9.3816027

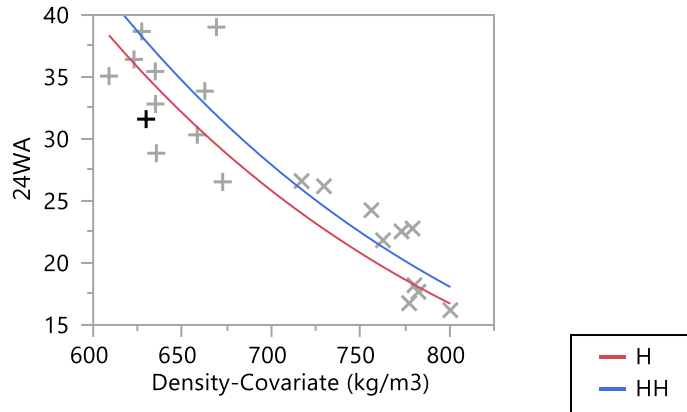
Levels not connected by same letter are significantly different.

Water absorption (24 hours) - analysis of covariance Hemp hurd fiber-reinforced particleboards

Response Log(24WA)

Whole Model

Regression Plot



Effect Tests

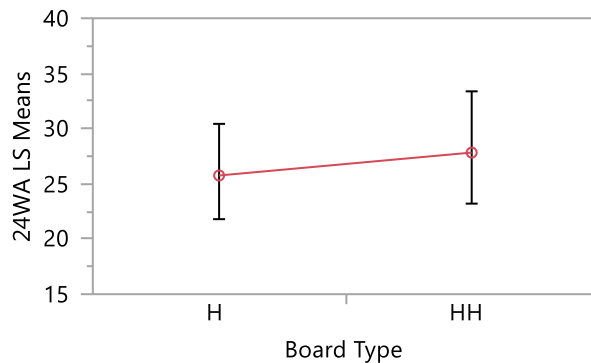
Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Board Type	1	1	0.00352438	0.2425	0.6283
Density-Covariate (kg/m3)	1	1	0.19215016	13.2230	0.0019*

Least Squares Means Table

Level	Least Sq Mean	Std Error	Lower 95%	Upper 95%	Mean
hh	25.755162	0.07942090	21.797122	30.431925	33.2954
hh-h	27.830599	0.08652732	23.204563	33.378877	20.9822

* Std Errors are on transformed Y's

LS Means Plot



LSMeans Differences Student's t
Differences are on transformed Y's

$\alpha = 0.050$ $t = 2.10092$

Level	Least Sq Mean
hh-h A	27.830599
hh A	25.755162

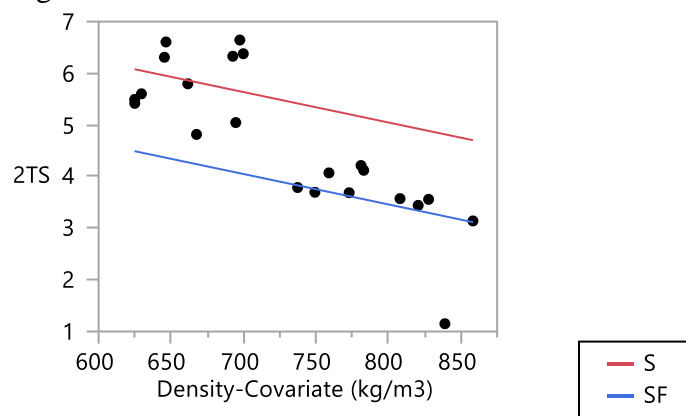
Levels not connected by same letter are significantly different.

Thickness swell (2 hours) - analysis of covariance Flax shive fiber-reinforced particleboards

Response 2TS

Whole Model

Regression Plot



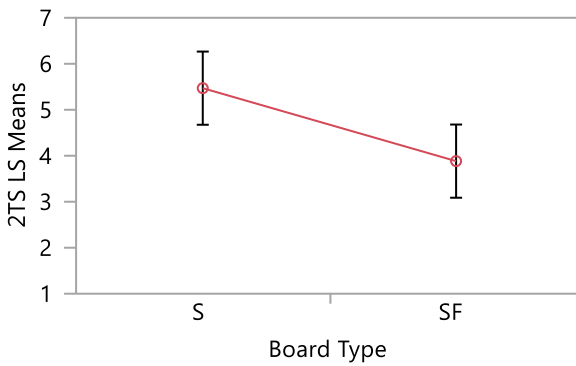
Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Board Type	1	1	2.8138536	5.2379	0.0337*
Density-Covariate (kg/m3)	1	1	0.8457346	1.5743	0.2248

Least Squares Means Table

Level	Least Sq Mean	Std Error	Lower 95%	Upper 95%	Mean
fs	5.4694958	0.38021932	4.6736876	6.2653040	5.85771
fs-f	3.8828920	0.38021932	3.0870838	4.6787002	3.49468

LS Means Plot



LSMeans Differences Student's t
 $\alpha = 0.050$ $t = 2.09302$

Level		Least Sq Mean
fs	A	5.4694958
fs-f	B	3.8828920

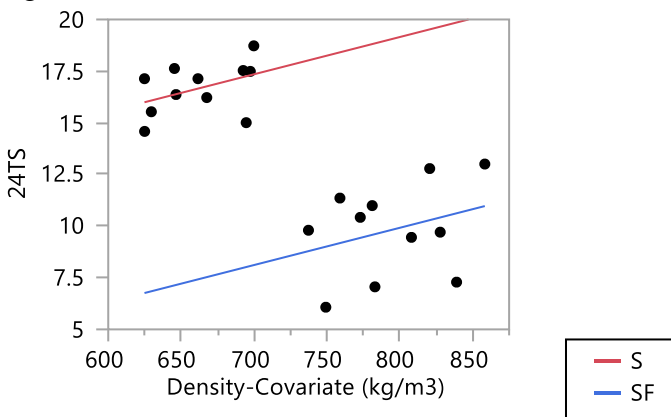
Levels not connected by same letter are significantly different.

Thickness swell (24 hours) - analysis of covariance Flax shive fiber-reinforced particleboards

Response 24TS

Whole Model

Regression Plot



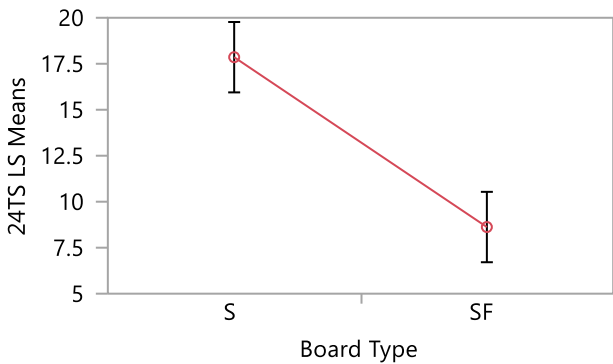
Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Board Type	1	1	95.299099	30.6976	<.0001*
Density-Covariate (kg/m3)	1	1	7.932162	2.5551	0.1264

Least Squares Means Table

Level	Least Sq Mean	Std Error	Lower 95%	Upper 95%	Mean
fs	17.855992	0.91401976	15.942927	19.769057	16.6671
fs-f	8.622581	0.91401976	6.709516	10.535647	9.8115

LS Means Plot



LSMeans Differences Student's t

$\alpha=$	0.050	$t=$	2.09302
Level		Least Sq Mean	
fs	A	17.855992	
fs-f	B	8.622581	

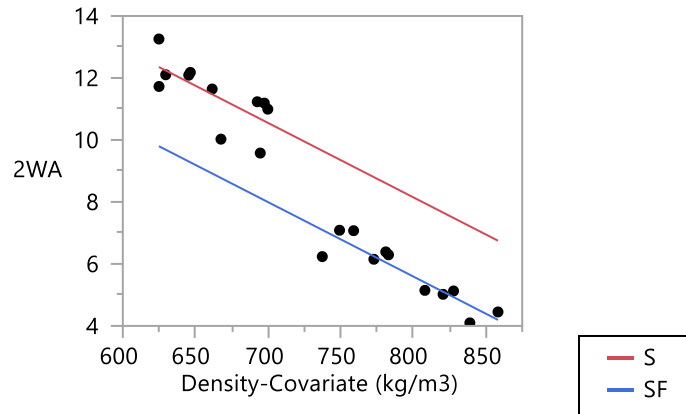
Levels not connected by same letter are significantly different

Water absorption (2 hours) - analysis of covariance Flax shive fiber-reinforced particleboards

Response 2WA

Whole Model

Regression Plot



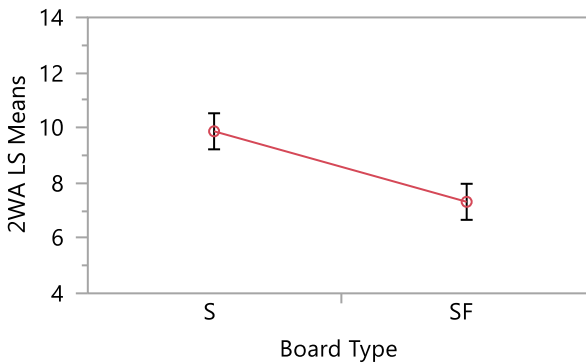
Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Board Type	1	1	7.300491	20.1676	0.0003*
Density-Covariate (kg/m3)	1	1	14.069469	38.8669	<.0001*

Least Squares Means Table

Level	Least Sq Mean	Std Error	Lower 95%	Upper 95%	Mean
fs	9.8725347	0.31211324	9.2192742	10.525795	11.4559
fs-f	7.3169294	0.31211324	6.6636689	7.970190	5.7335

LS Means Plot



LSMeans Differences Student's t

$\alpha = 0.050$ $t = 2.09302$

Level	Least Sq Mean
fs A	9.8725347
fs-f B	7.3169294

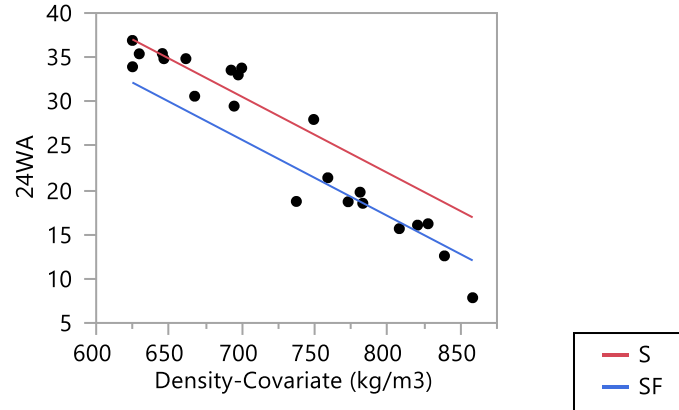
Levels not connected by same letter are significantly different.

Water absorption (24 hours) - analysis of covariance Flax shive fiber-reinforced particleboards

Response 24WA

Whole Model

Regression Plot



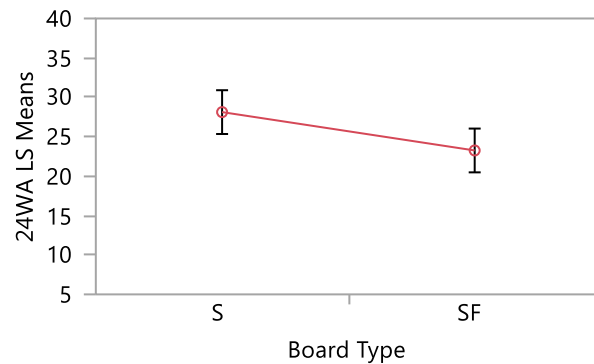
Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Board Type	1	1	26.45465	4.0447	0.0587
Density-Covariate (kg/m3)	1	1	180.84774	27.6500	<.0001*

Least Squares Means Table

Level	Least Sq Mean	Std Error	Lower 95%	Upper 95%	Mean
fs	28.120991	1.3266989	25.344178	30.897804	33.7979
fs-f	23.256148	1.3266989	20.479336	26.032961	17.5793

LS Means Plot



LSMeans Differences Student's t

$\alpha = 0.050$ $t = 2.09302$

Level		Least Sq Mean
S	A	28.120991
SF	A	23.256148

Levels not connected by same letter are significantly different.