STEAM PRE-TREATMENT AND LEACHING FOR THE PRODUCTION OF HIGH-QUALITY OIL PALM RESIDUE PELLETS

by

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ABSTRACT

It is estimated that about 26 million metric tons of oil palm oil residues are produced annually in Malaysia. The oil palm residues contain high ash content, high alkali metals and low lignin content. Ash and alkali metals lead to slagging in a combustion reactor and fouling of surfaces in convective tubes of heat exchangers. Low lignin content leads to disintegration of pellets during pellet handling and storage. This research thesis investigated the pelletization performance and the ash reduction on Empty Fruit Bunches (EFB) and Palm Kernel Shell (PKS) of oil palm residues by two different pre-treatment processes: steam explosion and water leaching. Steam explosion for increasing binding ability was performed by reacting EFB/PKS with saturated steam ranging from 120 to 220 °C for 5 minutes in a batch reactor. Water leaching for reducing the ash and alkali metals of EFB and PKS was investigated by immersing the samples in water from 25 to 55 °C. The immersion time ranged from instantaneous to 240 minutes. Single pellets were made from water treated and steam treated samples. A significant ash reduction from 5.47 to 2.47 % was found for the EFB pellet due to water leaching. A slight reduction of pellet's porosity and a significant reduction in ash content suggested that water leaching may be used to improve the quality of EFB as a biofuel. On the other hand, pre-treatment processes were not necessary for PKS, considering low binding ability and no ash reduction after pre-treatments. Kinetic models of EFB and PKS leaching were developed. A mass balance of cross flow process was investigated for evaluating the number of stages for a continuous leaching system of EFB and PKS. The techno-economic analysis showed that the additional leaching process costs \$25.75 per tonne in a pellet making line.

PREFACE

The literature review and the design of the research plan in Chapter 1 were prepared by the major author (Pak Yiu Lam) for the M.A.Sc. comprehensive exam. The construction of experimental set up (Chapter 2) was done by the major author. The results and discussions (Chapter 3) will be published in peer review journal. The major supervisors, Professor Sokhansanj and Professor Lim, provided guidelines and advice on the work from Chapter 1 to Chapter 5.

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NOMENCLATURE

C_{sat}	saturated leaching concentration (g/L)
C_t	concentration of water soluble compounds (g/L)
d_p	mean particle size (mm)
E	mechanical energy (J)
F	force (N)
F_N	maximum force when the pellet is crushed (N)
h	initial leaching rate (g/(L min))
i	distance interval
k	second-order leaching rate constant (g-min/L)
L	flow rate of the dry biomass (g/min)
<i>HHV</i> _{dry}	dry basis high heating value (MJ/kg)
HHV _{wet}	wet basis high heating value (MJ/kg)
h_i	indentation depth (mm)
H_M	Meyer Hardness (N/mm ²)
т	mass of the sample (kg)
МС	moisture content of the sample (w. b %)
P_{i}	pressure reading after pressurizing the reference volume (Pa)
P_2	pressure reading after including V_c (Pa)
t	leaching duration in kinetic models (min)
V	distilled water flow rate (g/min)
V_{c}	volume of sample cell (m ³)
Vo	input flow rate of the distilled water (g/min)
V_p	true volume of grinded biomass (m ³)

$V_{\scriptscriptstyle R}$	reference volume (m ³)
Vn	output flow rate of the leachate (g/min)
Wi	mass fraction (%)
X _n	mass ratio of input of the soluble and insoluble ash in the dry biomass (g/g)
x _{n+1}	mass ratio of output of the soluble and insoluble ash in the dry biomass (g/g)
y _n	mass ratio of input of the soluble ash in the leachate (g/g)
y_{n+1}	mass ratio of output of the soluble ash in the leachate (g/g)
Zi	geometric mean of the sieves (mm)
ΔL	deformation at distance (mm)
6 0	porosity (%)
$ ho_{\scriptscriptstyle b}$	pellet density (kg/m ³)
$ ho_{\scriptscriptstyle P}$	particle density (kg/m ³)

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Chapter 1 Introduction

1.1 Introduction of Oil Palm Residues

Oil palm residues, by-products of palm oil production, are considered to be a potential for pellet production. It is reported that 85 % of palm oil is produced in Malaysia and Indonesia (Timms, 2007). In addition, palm oil accounts for 1/3 of the world's vegetable oil market (Malaysia Palm Oil Council, 2010). It is estimated that Malaysia and Indonesia produce a combined 232 million bone dry tones (M bdt) of oil palm residues (Wan Rosli et al., 2004; Menon et al., 2003). These figures raised the question on how these abundant oil palm residues can be used to generate energy. The bulk density of pellets with less than 10% moisture content is around 600 – 700 kg/m³ and the bulk density of woodchips with 25 % moisture content is around 200 - 350 kg/m³ (Eco Link Power Ltd, 2009). Pellets are favored over wood chips for long distance transportation. Oil palm pellets are compatible with existing co-fired power facilities. Co-firing 10 to 100 % biomass with coal is proven to be possible (Peksa et al., 2007).

Figure 1.1 shows the unit operations of palm oil processing (Poku, 2002). The oil palm bunches with fresh fruits initially arrive at the oil palm mill. The raw bunches are sterilized by low-pressurized steam at 140 °C and 0.28 MPa (Shamsudin et al., 2012). Empty fruit bunches (Figure 1.2) are further extracted during the threshing process, in which a rotary drum or fixed drum equipped with rotary beater bars detaches the fruits from bunches. Fruit digestion releases the palm oil from the fruit by breaking down the oil-bearing cells within a steam-heated stirring tank at 80°C to 90°C (Kasim, 2009). Pulp pressing applies a

mechanical pressure on the digesting mixture, yielding a mixture of oil, moisture, fiber and nut. The oil mixture is clarified, dried and stored to yield pure palm oil. The fiber and nuts are dried and crushed so that the palm kernel shell can be extracted.



Figure 1.1 Unit Operations of Palm Oil Processing

1.1.1 Empty Fruit Bunch

Empty Fruit Bunch (EFB) is a bunch of foliage that is surrounding the oil palm fruit (Figure 1.2). EFB is pre-processed by sterilizing fresh fruits and pericarp along with brunches. Sterilization inactivates enzymatic process, avoiding an excessive production of free fatty acids (Department of Industrial Work, 2006). The EFB is separated from fruit by a rotary drum after sterilization. It is stated that EFB can be used for a variety of industrial applications, such as pharmaceutical coating, auto motive interior, pulp and paper, fiberboard, mattress, cushion, building materials, etc (Rahman et al., 2007;Khalid et al., 2008;Law et al., 2007; Nasrin et al., 2008; Prasertsan et al., 2006; Ramli et al., 2002). EFB pellets exhibit high energy content due to the decrease in moisture content, uniform size, superior combustion performance and high mechanical strength (Narsrin et al., 2008).



Figure.1.2 Empty Fruit Bunch (EFB)

1.1.2 Palm Kernel Shell

Palm Kernel Shell (PKS) is the endocarp, which is the surrounding layer of the oil palm fruit seed (Figure 1.3). PKS is further crushed into pieces (Figure 1.4) during oil extraction. PKS has high calorific value of 18.51 MJ/kg and low sulfur content less than 0.1 % (Table 1.1). PKS is a green renewable fuel for biomass power plants. It is stated that carbonized PKS can substitute charcoal for barbeque or residential heating applications (Elham, 2001). Carbonized PKS with specific surface area of $600 - 1200 \text{ m}^2/\text{g}$ can be used as absorbent in liquid and gaseous phase filtration (Ng et al., 2002; Oritiz et al., 2005).



Figure 1.3 Overall Structure of Oil Palm Fruit



Figure 1.4 Palm Kernel Shell (PKS)

1.1.3 Challenges in Utilizing EFB and PKS as a Renewable Fuel

Table 1.1 lists published data on composition of PKS and EFB. PKS has a larger content of lignin but a smaller content of cellulose than EFB. There are more extractives in PKS than in EFB. A high lignin may prove to be useful in making pellets from PKS. But its higher extractive may make it more prone to affinity to water absorption (Kalyan and Motrey, 2009). However, the high heat value measured for the PKS and EFB are almost identical.

Biomass feedstock	PKS	EFB
Proximate analysis* (wt.%)		
Moisture	5.92	7.38
Volatiles	71.31	76.41
Fixed carbon	17.81	11.57
HHV (MJ/kg)	18.51	18.74
Elemental analysis* (wt.%)		
С	44.6	51.8
Н	6.5	7.0
Ο	40.2	40.3
Ν	2.92	0.7
S	< 0.1	0.2
Component analysis (wt.%)		
Cellulose	30.59	43.80 ^a
Hemicellulose	30.64	35.00 ^a
Lignin	20.41	16.04 ^a
Extractives	18.36	4.80^{a}

Table 1.1 Compositions of EFB and PKS (Kim et al., 2013 and Hamzah et al., 2011.^a)

*Analytical Procedure of Proximate and Elemental Analysis can be found in Table A.2.

From proximate analysis, both PKS and EFB have comparable values in volatiles, fixed carbon and moisture content, which concurs that the high heat values are comparable as shown in Table 1.1. From elemental analysis, PKS has a high nitrogen content, which may yield nitrogen oxide at high temperature during the combustion process (Idris et al., 2012).

1.1.3.1 Size Reduction

EFB and PKS are different components of the oil palm tree and therefore exhibit different material properties (Figure 1.5). Tammelin et al. (2011) stated that cellulose confers tensile strength and the hemicelluloses/lignin confers ductility and flexibility. The high abundance in cellulose around 43.8 % by weight (Table 1.1) indicates that EFB material has

higher tensile strength. Higher tensile strength of EFB suggests that EFB can withstand higher maximum stress while being stretch before failing. EFB requires higher elastic energy to stretch than PKS, given that EFB and PKS have similar percentages of the hemicelluloses/lignin. Particle size distribution of EFB and PKS has to be no more than 3 mm geometric mean diameter for standard-size pellet mills (Ciolkosz, 2009). In addition, the alkali content, an ash deposit that causes slagging and fouling in combustion unit, is found to be more abundant in smaller particle sizes (Liu et al, 2011). Although the elimination of the smaller particles can improve combustion performance, the complete elimination of the alkali content is not an economical option. With a large specific surface area of Kaolin, Konsomboon et al. (2011) indicated that the addition of kaolin during combustion as an alternative can effectively absorb volatile potassium compounds from smaller particle sizes of EFB.



Figure 1.5 Different Parts of a Palm Oil Tree and the Structure of Fibres (Abdul Khalil et al., 2012)

1.1.3.2 Impact of Ash and Moisture Content on Combustion

Ash content, alkali content and moisture content of EFB and PKS impose challenges during combustion. Ash content and alkali content cause corrosion, slagging and fouling in the furnace, where a substantial cost is required in order to repair the furnace (Heinzel et al., 1998; Pronobis, 2005; Nutalapati et al., 2007). In addition, the suspension of the fly ash in the flue gas stream reduces the convective heat transfer to the heating surface, thereby decreasing the combustion efficiency (Benson, 1992). Shuit et al. (2009) suggested that asreceived oil palm residues with high moisture content of 38 % (w.b.) required extra drying for EFB and PKS down to 20 % (w.b.) for an efficient combustion (Helin, 2005; Francescato et al., 2008; Forest Research, 2011).

1.2 Pelletization

With the increasing demand of energy fuel, EFB and PKS residues are considered to be potential fuels for power generation in East Asia (Cocchi et al, 2011). Pelletization may facilitate handling and transporting of EFB and PKS for domestic and international markets.

High moisture content and low lignin content prevent effective binding of ground biomass to produce durable pellets. High moisture content showed negative influences in mechanical properties as well as in pellet density (Nielsen et al., 2009; Carone et al., 2011; Odogherty et al., 1984). In addition, low lignin content decreases the binding of pellet, which could lead to the disintegration of pellets during transportation. Dust generated during pellet handling may become a potent fuel for explosion. Amrogowicz et al. (1991) investigated the effect of different dust explosion suppression agent on four explosion dusts: melamine, wood dust, wheat flour and coal dust. Calle et al. (2005) studied the influence of the size distribution and concentration on wood dust explosion by both experiment and reaction modeling. The results showed that a decrease of the violence of the explosion with the increase of the particle size. Melin (2008) compared the particle size distribution of dust from white wood pellets and bark pellets and found that dust from bark pellets are smaller and can become airborne.

1.3 Pre-treatment Processes

1.3.1 Principle and Application of Steam Explosion

Steam explosion is a process involving steaming the biomass in the temperature range of 180 - 240 °C, steam pressure 1.03 - 3.45 MPa for 0 - 10 min and followed by the explosive decompression (Delong, 1981; Lam, 2011). Saturated steam activates the lignin in the middle lamella of the plant cell when the steam temperature is above the glass transition temperature of the lignin. The lignin will melt, migrate and relocate as beads condensing on the fiber surface (Donohoe et al., 2008). The exposure of lignin increases the binding ability in the pellet production process.

Steam hydrolysed cellular structure comprises of carbohydrates (cellulose and hemicelluloses) and lignin (Ramos et al., 2003). Steam cleaves the hydroxyl group of the hemicellulose to form acetic acid. In the presence of acetic acid, hemicelluloses and lignin yield low molecular weight components: mono-sugar and acid-soluble lignin. For example, steam treatment of hardwood produces the mono-sugar, Xylose, and part of Xylose further dehydrates to form furfural under high treatment temperature (Ramos et al., 2003). Lignin

goes through repolymerization and condensation so that lower molecular lignin is produced (Miranda et al., 1979). By reducing the high molecular weight lignin to low molecular weight lignin, the new structure of the biomass allows more accessible sites for pellet binding, thereby increasing the durability of the pellets (Lam et al., 2011).

Extraction of sugar with steam pre-treatments on EFB has been introduced as a pretreatment process for ethanol conversion (Sendelius et al., 2005). Different steam explosion conditions on EFB have been studied for enhancement of its biodegradability (Baharuddin et al., 2013). To the knowledge of the author, there is no published report on the pelletization performance of steam treated EFB and PKS.

1.3.2 Water Leaching

Water leaching is defined as the removal of water soluble and ion-exchangeable inorganic constituents from solid substrate using water. Water leaching is reported as a low cost pre-treatment to reduce inorganic constituents in the biomass content (Jenkins et al., 1996; Turn et al., 1997; Arvelakis et al., 2003). Table A.1 summarizes the research findings for biomass leaching. Typical inorganic constituents are Potassium, Sodium, Calcium, Aluminum and Magnesium. Potassium exists in an ionic form and thus is highly mobile in water. Potassium is crucial to plant metabolism and is therefore mostly concentrated in regions where rapid growth occurs, such as leaves and plant tops; Sodium and Calcium are found with small concentrations in plants and are important for metabolism and structure integrity. Aluminum, one of the ingredients of many soils, is toxic to the plants. Magnesium is part of the chlorophyll and is responsible for photosynthesis.

Inorganic constituents cause fouling, slagging, agglomeration and corrosion during combustions. Fouling occurs when the inorganic constituents form ash deposit on the surfaces of the combustion furnace. Slagging, in which a glass layer rich in Fe₂O₃ and K₂O (Kostakis, 2011), is a process formed by the melting of the deposits. Agglomeration of Fe₂O₃ and K₂O leads to the increase of the thickness of the glass layer. Interaction of the deposit layers with the metal surfaces within the furnace accelerates corrosion. Potassium compound, such as Potassium Chloride (KCl) in EFB, is reported to evolve into gas –phase condenses and deposits on low-temperature surface of the heat exchanger, causing slagging (Konsomboon et al., 2011). This retards the heat transfer between the heat exchanger and also corrodes the surface (Madhiyanon et al., 2012).

The removal of inorganic constituents of the feedstock can improve the combustion quality of fuel. The effectiveness of the leaching process relies on material characteristics, particle size distribution and treatment conditions, such as biomass to water mass ratio, temperature, leaching duration (Liu et al., 2011). The effluent recovered after the leaching process can be recovered by reverse osmosis or distillation (Jenkins, 2003).

Jenkins et al. (1996) reported that a significant proportion of Potassium and Chlorine 90 % - 91 % were removed after leaching banagrass with water. Wheat straw and rice straw showed a similar trend in responding to water leaching in the reduction of Potassium and Chlorine. A substantial reduction of soluble metal salts after water leaching on pine barks and switchgrass was reported (Liu et al., 2011).

1.4 Objectives

The challenges of EFB and PKS pellet production as fuel for combustion are their high ash content, high moisture content (up to 65 % in EFB), high alkali content, and low lignin content (in EFB) (Singh,1999). High ash content leads to high disposal costs of bottom ash. High moisture content of feedstock requires a substantial amount of energy in the drying process prior to pelletization. High alkali and chlorine contents cause slagging in the combustion furnace and corrosion of the heat exchange surfaces. Low lignin content reduces the binding characteristics of biomass to form durable pellets.

The specific objectives of this research are:

1. To investigate steam and leaching pre-treatments and their effect on durability of pellets made from EFB and PKS.

2. To investigate the effect of pre-treatments to reduce the alkali and ash contents of pellets made from EFB and PKS.

3. To analyze the techno economics of leaching pre-treatment.

1.5 Scope and Organization of Thesis

Since the binding capability and the ash content of wood pellet are the main concerns for combustion process, a series of research approaches were developed to tackle the existing challenges. In the first part of research, physical characterizations were performed to evaluate the physical properties of the EFB and PKS pellets and their potential use for combustion. In the second part of the research, an analysis of material balance and ash content at different pre-treatment conditions were studied. This study recommended the optimal pre-treatment conditions to upgrade the quality of these species for combustion. In the third part of the research, metal content analysis and leaching behavior of the steam exploded EFB and PKS material were performed and discussed. In the fourth part of the research, the feasibility of establishing a leaching facility will be analyzed through cost estimation as well as sensitivity analysis.

This thesis is presented in four chapters. Chapter 1 reviews literature related to pretreatments and pelletization of EFB and PKS and develops objectives of the research. Chapter 2 describes experiments on steam treatment, water leaching, and pelletization. Chapter 3 presents experimental data on pre-treatments of EFB and PKS and outlines the modeling of pre-treatments of EFB and PKS. Chapter 4 discusses the overall techno economic implications of leaching pre-treatment.

Chapter 2 Experimental Set Up and Methodology

2.1 Sample Preparation Methods

2.1.1 Materials

Empty Fruit Branches (EFB) and Palm Kernel Shell (PKS) with approximately 25 % moisture content (w.b.) were supplied by TorchLight Bioresources Inc, Vancouver (Figure 2.1). These materials were originated from the rain forest in Malaysia. The general process flow diagram of the experiments is shown in Figure 2.2.

Material	As-Received	Ground Material
EFB		
PKS		

Figure 2.1 As-received Material and Ground Material of EFB and PKS



Figure 2.2 Schematic Diagram of the Experimental Procedures of Pre-treatments and Pelletization

2.1.2 Drying

The as-received EFB and PKS were conditioned to moisture contents of average 15 % with ± 1 % (w.b.) at a drying temperature of 50 °C in a convection oven. The drying duration was 24 hours. Moisture conditioning is required to ensure the ground material to maintain at desired moisture content for pre-treatments and pelletization. The low drying temperature was applied to prevent denature of the enzymatic activity of the woody biomass.

2.1.3 Size Reduction – Milling

The materials were further processed into ground particles by a Retsch grinder SM100 model (Retsch Inc. Newtown, PA) with a 4 mm screen size. Particle size analysis was done on a set of sieves and a sieve shaker. The sieving analysis will be described in section 2.2.1).

2.1.4 Pre-treatment: Steam Explosion

The steam explosion unit shown in Figure 2.3 was used to treat the materials prior to pelletization and it consists of a 1 L steam reactor and a 2 L boiler (Lam, 2011). A process flow diagram of the steam explosion unit is documented in Appendix B. To begin with, 30.0 g (w.b.) ground biomass was fed into the reactor. After that, 300 ml of water were pumped into the boiler to produce saturated steam. When the steam temperature reached the desired experimental condition, the saturated steam was allowed to flow from the boiler to the steam temperature reached the steam reactor and to steam the material for 5 minutes. At this point of time, the automatic solenoid valve was activated through a computer program. The treated material was ejected at once

into a container from the steam reactor. The ejected material and the residual in the reactor were collected in a plastic bag to determine the total dry mass recovery. The steam temperatures investigated for this research were 120, 140, 160, 180, 200 and 220 $^{\circ}$ C.



Figure 2.3 Apparatus Set Up of Steam Explosion Unit

2.1.5 Pre-treatment: Water Leaching

For water leaching, 5.0 g biomass was loaded into a beaker mixed uniformly with 100.0 g distilled water on a hot plate stirrer (Ika Works, Inc. NC) as shown in Figure 2.4. When the target leaching duration had reached, the mixture was poured into a filtration unit (i.e. a glass funnel) connected to the vacuum pump. Filter paper was placed on top of the funnel so that the solid material can be recovered from the filter paper. The summation of the weight measurement of moisture contents of both the filtrate and the residual of the drain-out mixture were used to determine the total dry mass recovery. The weight measurement was

carried on a ALC-80.4 (Acculab, Edgewood, NY) analytical balance with 0.1 mg precision. The moisture content was measured by drying the filter paper and wet biomass in a precision oven at 105 $^{\circ}$ C for 24 hours. The investigated leaching durations were 1, 3, 5, 10, 30, 60, 120 and 240 min. Temperature effect was studied at 25, 40 and 55 $^{\circ}$ C.



Figure 2.4 Apparatus Set Up of Leaching Process

2.1.6 Pelletization

Ground material, which was smaller than 4 mm screen size, was used to make a pellet in a single pellet piston-cylinder assembly (Figure 2.5). The fixture was composed of the top and the bottom plates. The top plate with four holes at the corner was installed with a 6.30 mm diameter piston at the center for pellet compression and extrusion. A die with 70 mm channel length and 6.35 mm diameter was fixed at the center of the bottom part. At the corners of the bottom plate, there were four installed well aligned collars to prevent the piston from bending due to misalignment. This fixture was portable and deployable with the MTI - 50K universal press (Measurement Technology Inc. Roswell. GA) (Figure 2.5). MTI – 50K press is a universal compression machine with its flange exerting downward force on the top part of the fixture (Figure 2.5). A heating tape wrapped around the die maintained a constant die temperature of 100 $^{\circ}$ C to facilitate the fabrication process.



Figure 2.5 Piston Cylinder Assembly (Left) and MTI - 50K Universal Press (Right) for Pelletization Unit

To begin with, a small block was inserted at the bottom of the die channel. The block can support counter upward force during the compression. 0.85 g of ground particle was filled into the die channel to form an initial loose bulk packing. The MTI machine was preset at a maximum of 4000 N downward compressive force. The moving speed of the piston was 10 mm/min downward. When the maximum compression force reached 4000 N, the piston stopped the movement and halted at that position for 15 s to undergo stress relaxation for achieving a dimensional stability of the pellet. After 15 s, the small block underneath the die channel was removed and the piston extruded the pellet out of the die channel. Three replicates of pellets were made for each sample.

2.2 Characterization Methods

2.2.1 Particle Size Analysis

The particle size analysis was performed according to the ASABE S319.3 (1997). The experimental set up was a Ro-Tap sieve shaker (Tyler Industrial Products, OH, USA). Exactly 20 g of the ground sample was placed on top of the stack of sieves from the smallest to the largest mesh number. The mesh numbers of sieves for particles size distribution were: 7, 10, 14, 18, 25, 35, 45, 60, 80 and 100. The nominal sieve openings corresponded to the mesh numbers were 4, 2, 1.41, 1, 0.707, 0.5, 0.354, 0.25, 0.177 and 0.149 mm. Sieving duration was 5 minutes. Five replicate measurements were carried out for each sample. The mass fraction was calculated by the retained mass at a specific nominal diameter divided by the total input amount of samples. The mean particle size (d_p) was determined by the mass fraction and the geometric mean of the sieve:

$$d_p = \left(\sum \frac{w_i}{z_i}\right)^{-1} \tag{2-1}$$

where w_i is the mass fraction (decimal) and z_i is the geometric mean of the sieves (mm). z_i can be computed by the square root of the product of two sieves size.

2.2.2 Moisture Content, Heat Value and Ash Content

The moisture content measurement of the untreated and treated sample was performed according to ASABE S358.2 (2010). 1 g sample was used for moisture content measurement. The sample was placed in a precision oven at 105 °C for 24 hours. The weight difference before and after 24 hours drying over the initial mass of material was equal to the moisture content of the samples (wet mass basis). Three replicates were performed in order to credit the accuracy of the reported values in wet basis.

The heat (calorific) value of the ground particle was measured by an adiabatic oxygen bomb calorimeter (Parr Instrument Company, IL) using the procedure of standard method for gross calorific value of coal and coke by the adiabatic bomb calorimeter (ASTM D2015-96, 1996). Approximately 0.5 g of ground material was placed in a nickel crucible and fired inside the bomb calorimeter using an ignition wire in the presence of oxygen. The calorific value of the sample was indicated in HHV_{wet} and reported as HHV_{dry} according to eq. (2-2). The measurements were repeated three times and the average values on dry basis were reported.

$$HHV_{wet} = HHV_{dry} \times \left(1 - \frac{MC}{100}\right)$$
(2-2)

where HHV_{wet} is wet basis high heating value(MJ/kg), HHV_{dry} is the dry basis high heating value(MJ/kg) and *MC* is the wet basis of moisture content of the sample (%).

The ash content of the samples was determined based on NREL biomass chemical composition analysis (NREL Standard TP-510-42622, 2005). Around 0.5 g of the sample was placed in a porcelain crucible. The crucible was placed inside a muffle furnace (Blue M Electric Company, IL). The temperature of the furnace was increased to 250°C for 30 minutes, and followed by increasing to 575°C for 3 hours. The samples were cooled down to 105°C for 8 hours until the samples were collected. The cooled samples were covered, removed from the furnace and cooled to room temperature inside a glass desiccator. The mass retained in the crucible was expressed as percent ash content. The ash content measurements were repeated for three times for each sample.

2.2.3 Ash Compositional Analysis - Inductive Coupling Plasma (ICP) Spectrometry

The metal content of the untreated and treated biomass material was determined by the Inductive Coupling Plasma Spectrometry (Thermo Scientific iCAP 6500 Radial View ICP-OES Spectrometer, England). For each analysis, 25 g of biomass material or 40 ml of leachate was sent to ALS Laboratory Inc., Vancouver. Preliminary material treatment required acid digestion of biomass material inside either a hot-block or an oven (EPA Method 3005A, 2013). After the acid pre-treatment, the sample was loaded into inductively coupled plasma - optical emission spectrometry (EPA Method 6010B, 2013) for metal analysis. The result yielded the metal content of alkali metals, alkaline earth metals and transition metals based on the mass ratio or mass concentration of the samples.

2.2.4 Pellet Density, Particle Density and Porosity

Pellet density (ρ_b) was defined as dividing the pellet mass by the pellet bulk volume. A digital caliper (MastercraftMD, Miami, FL, USA) was used to measure the length and diameter of the pellet to determine the bulk volume. A digital balance (Acculab ALC-80.4, Edgewood, NY) with four decimal accuracy was used to measure the pellet mass. Five replicates were reported.

Particle density was defined as a void free pellet density. A Quantachrome Multipycnometer (Quantachrome, Boyton Beach, FL, USA) was used to measure the void volume of the pellets. The mechanism of the pycnometer was determining the pressure difference between two cells due to nitrogen injection, a reference cell without sample and a cell with samples. The user manual (Quantachrome Instruments, Multipycnometer Operating Manual) of the pycnometer suggested the ideal gas equation to deduce the particle density based on the pressure difference:

$$V_p = V_c - V_R \left(\frac{P_1}{P_2} - 1\right)$$
(2-3)

where: V_p = true volume of biomass grinds (m³), V_c = volume of sample cell (m³), V_k = reference volume (m³), P_i = pressure reading after pressurizing the reference volume (Pa) and P_2 = pressure reading after including V_c (Pa).

$$\rho_p = \frac{m}{v_p} \tag{2-4}$$

where: m = mass of the sample (kg), $V_p = \text{true volume of biomass grinds (m³)}$.

Porosity (ϵ_0) was further calculated from pellet density and particle density in the following equation:
$$\varepsilon_0 = 1 - \frac{\rho_b}{\rho_p} \tag{2-5}$$

where ε_0 is the porosity, ρ_b = pellet density of a pellet (kg/m³), ρ_p = particle density of a pellet (kg/m³).

2.2.5 Energy Input for Pelletization

Force v.s. displacements data of compression and expulsion were collected from the data logging system during pelletization. The mechanical energy input, excluding external heat input, can be integrated through the area under the force v.s. displacement curve. The mechanical energy equation is presented as (Tooyserkani et al., 2013):

$$E = \sum_{i=1}^{n} F_i \cdot \Delta L_i \tag{2-6}$$

where *E* is the mechanical energy (J), F is the force (N) at distance i interval and ΔL is deformation (m) at distance i. Mechanical Energy refers to either compression energy or extrusion energy. The displacement of compression is measured from the initial position up to the position at maximum applied force. The displacement of extrusion is measured from the displacement of rod before initiating the movement of the pellet due to static friction between the pellet and the die channel. The specific mechanical energy is normalized with the pellet mass in J/g. Five replicates were calculated for compression and extrusion energy respectively.

2.2.6 Breakage Test

The breakage test was used to determine the mechanical strength of the samples according to the procedure of Tabil et al. (2002). A single pellet was placed on a metal platform of the MTI machine, where the 6.35 mm diameter compression rod was set to indent the pellet at the middle as shown in Figure 2.6. To begin with, the compression rod was brought down in contact with the pellet without applying a pre-loading force. When the pellet was barely fixed between the platform and the compression rod, loading started to apply for indentation to measure the mechanical strength of the pellet. During indentation, the maximum applied force (*F*) and displacements of the piston (*d*) were recorded. The Meyer Hardness (H_M) is defined as the applied force divided by the projected indentation area and was calculated according to the following equation (Tabil et al., 2002; Lam et al., 2011; Lam et al., 2013b):

$$H_{M} = \frac{F_{N}}{\pi (Dh_{i} - h_{i}^{2})}$$
(2-7)

where H_M is the Meyer Hardness (N/mm²) and h_i is the indentation depth (mm), D is the initial diameter of a pellet cross section (mm), and F_N is the maximum force when the pellet is crushed (N). Five replicates were performed for each breakage test.



Figure 2.6 Apparatus Set Up of Breakage Test

2.3 Modeling of Water Leaching

2.3.1 Batch Leaching

A mass balance model is introduced to design a unit operation process of ash removal from biomass by leaching method. For modeling purpose, it is assumed that leaching metals from biomass takes place in several stages. Figure 2.7 illustrates a single-pass flow into and out from a single stage. The single stage is then assembled into multiple stage processes for effective ash removal.



Figure 2.7 A Model of Flows Into and Out of a Single Stage Leaching Process. y_n is the Input Mass Ratio of the Soluble Ash in the Leachate, y_{n+1} is the Output Mass Ratio of the Soluble Ash in the Leachate, x_n is the Input Mass Ratio of the Soluble and Insoluble Ash in the Dry Biomass, x_{n+1} is the Output Mass Ratio of the Soluble and Insoluble Ash in the Dry Biomass. L is the Flow Rate of the Dry Biomass. V_o is the Input Flow Rate of the Distilled Water. V_n is the Output Flow Rate of the Leachate.

Mass Balance of ash removal during leaching process (Earle, 1983):

$$y_{n+1} = \frac{L}{V_n} (x_n - x_{n+1}) + y_n$$
(2-8)

where y_n is the input mass ratio of the soluble ash in the leachate (g/g), y_{n+1} is the output mass ratio of the soluble ash in the leachate (g/g), x_n is the input mass ratio of the soluble and insoluble ash in the dry biomass (g/g), x_{n+1} is the output mass ratio of the soluble and insoluble ash in the dry biomass (g/g), L is the flow rate of the dry biomass, V_o is the input flow rate of the distilled water (g/min), V_n is the output flow rate (g/min) of the leachate. Thus, L/V_n is the ratio of the flow rate of the dry biomass (g/min) to the output flow rate of the leachate (g/min).

2.3.2 Determination of the Leaching Rate Constant and Leaching Saturation Capacity

The leaching behavior of a certain materials (e.g. organic or inorganic constituents) from a substrate can be studied by the leaching experiment. A kinetic model can be used to describe the leaching rate. The governing model parameters generally include the initial concentration of leachate, the leaching rate constant (k) and the maximum possible concentration of metal in the solution (C_{sat}). The leaching capacity is also defined as the concentration of water-soluble compounds at saturation. An overall mass balance has been proposed for leaching operation of EFB or PKS and is described as the following (Ho et al., 2005):

$$EFB/PKS_{(s)} + Distilled Water_{(l)} \rightarrow (water-soluble compounds)_{(aq)}$$
 (2-9)

The model assumes that the water –soluble compounds are the ash (e.g. organic or inorganic compounds) from the biomass after the immersion of the biomass into the distilled water. The model also assumes as an irreversible process.

In order to determine the leaching rate constant, Ho et al. (2005) suggested the second –order rate law to predict the leaching kinetics for Tilia sapwood. The second-order kinetic differential equation was formulated as following:

$$\frac{\mathrm{d}C_{\mathrm{t}}}{\mathrm{d}\mathrm{t}} = \mathrm{k}(\mathrm{C}_{\mathrm{sat}} - \mathrm{C}_{\mathrm{t}})^2 \tag{2-10}$$

where k is the second-order leaching rate constant (g-min/L), C_{sat} is the leaching capacity (g/L) which is the saturated concentration of water soluble compounds within the suspension,

 C_t is the concentration of water soluble compounds (g/L) within the suspension at any time t (min). The concentration of the water soluble compounds was measured by the weight of the dissolved dry matter within the volume of the leaching fluid.

Given that the boundary conditions are t = 0 to t and $C_t = 0$ to C_t , integrating the differential equation Eq. (2-10) to yield an Eq. (2-11) and rearranged in a linear form Eq. (2-12):

$$C_{t} = \frac{C_{sat}^{2}kt}{1+C_{sat}kt}$$
(2-11)

$$\frac{t}{C_t} = \frac{1}{kC_{sat}^2} + \frac{t}{C_{sat}}$$
(2-12)

By taking the reciprocal of Eq. (2-12), the leaching rate, C_t/t , can be obtained as:

$$\frac{C_{t}}{t} = \frac{1}{\frac{1}{kC_{sat}^{2}} + \frac{t}{C_{sat}}}$$
(2-13)

When t approaches zero, the initial leaching rate (g/(L min)), h, can be computed as:

$$h = \lim_{t \to 0} \frac{C_t}{t} = kC_{\text{sat}}^2$$
(2-14)

Chapter 3 Results and Discussion

3.1 Physical Characterization of EFB and PKS Pellets

The physical characterization of untreated/control, steam exploded and water leached pellet was carried out. EFB was ground to a mean particle size of 0.31 mm and PKS was ground to mean size of 0.72 mm as shown in Figure 3.1. Steam explosion was studied at 220 °C for 5 minutes resident time in the reactor and water leaching was studied at room temperature for 240 minutes leaching duration. Figure 3.2 and 3.3 show the ground particles and pellets of control, water leached and steam exploded, respectively. There was no physical changes in color of the water leached biomass, however, steam exploded biomass became darker than the untreated biomass. The dark color of the steam exploded biomass can be explained by the caramelization of sugars due to Maillard reaction (Lam et al., 2012). The color changes were also caused by extractives degradation (McDonald et al., 1997; Sundqvist et al., 2002).



Figure 3.1 Size Distribution of EFB and PKS Ground using a Knife Mill Equipped with 4 mm Square Screen

Material	Untreated	Leached	Steam treated
EFB			
PKS			

Figure 3.2 Untreated, Leached and Steam Treated EFB and PKS Ground Material

Material	Untreated	Leached	Steam treated
EFB			
PKS			

Figure 3.3 Untreated, Leached and Steam Treated EFB and PKS Pellets

Table 3.1 lists the physical properties of ground EFB and PKS particles before and after treatments. The reported moisture content of the control samples represent moisture content immediately after size reduction operation. A portion of the control samples was sampled to prepare the leached and steam exploded ground material. Since the high water content of the material after leaching was not favorable for pelletization, the moisture content of leached material was conditioned to 13 - 18 % MC by using the drying oven at 50°C. The steam exploded EFB and PKS ground material was pelletized at 7.3 and 6.7 % MC, respectively. The highest high heat value was found for steam exploded EFB material at 22.42 MJ/kg as compared to the control sample of 18.54 MJ/kg. The HHV of PKS was 21.51 MJ/kg for the control sample and it did not have any significant changes regardless of different applied pretreatments. Negro et al. (2003) pointed out that the increase of HHV is due to the increase in the C/H ratio after steam explosion, which removed – OH group of hemicelluloses. Since EFB C/H ratio (51.8 %/7.0 % = 7.4) is higher than PKS (44.6%/6.5%=6.9) (Table 1.1), it is reasonable to expect that steam explosion may have a larger influence on HHV of EFB than that of PKS. On the other hand, the low C/H ratio of PKS suggests that there is less room in increasing the HHV brought by steam explosion. The ash content of EFB control sample dropped from 5.47 to 2.47 % due to water leaching. Similar results were observed for PKS that leaching dropped its content of ash from 1.35 to 0.72 %. Jain (1992) reported that the ash content in hardwood and softwood varied from 1.2 to 7.5 % and 0.3 to 1.8 %, respectively. Water leaching is a viable option in reducing the high ash content in hardwood, softwood and oil palm residue in order to meet the ash content requirement (< 0.7 wt% of dry matter) of the wood pellets in CEN Standards (Alakangas et al.,2006). On the other hand, the ash content of steam exploded EFB and PKS showed a

dramatic rise. Lam et al. (2011) showed that steam explosion removes a substantial amount of hemicelluloses, which results in an increase in the net ash content.

		Moisture	HHV-dry basis	Ash Content				
Species	Pre-treatment	Content (%)	(MJ/kg)	(%)				
		Average	Average	Average				
	Control	13.5 ± 0.7	18.54 ± 0.80	5.47 ± 0.08				
EFB	Leaching	17.8 ± 0.6	19.07 ± 0.19	2.47 ± 0.19				
	Steam Explosion	7.3 ± 0.4	22.42 ± 0.91	8.08 ± 0.27				
	Control	13.5 ± 0.3	21.51 ± 1.14	1.35 ± 0.22				
PKS -	Leaching	13.5 ± 0.4	21.26 ± 0.22	0.72 ± 0.35				
	Steam Explosion	6.7 ± 0.3	21.99 ± 0.20	1.85 ± 0.21				

Table 3.1 Physical Characterization of EFB and PKS Ground Material (n = 3)

Note: n = number of repeated measurements.

Table 3.2 lists the physical dimensions and particle densities of pellets with and without pretreatment. The mass of a single pellet ranged from 0.80 to 0.82 g for EFB pellet and 0.57 to 0.79 g for PKS pellet. The diameter did not show much variation at 6.5 mm, which is close to the fixed inner diameter of the die channel. A shorter pellet length in pellets with water leaching was found in PKS, but not in EFB. The pellet densities of both steam exploded EFB and PKS pellets were larger than the pellets made from control or leached biomass, which concurred with the calculated results in the decrease of the porosity. Therefore, steam explosion is able to increase packing proximity due to lignin activation (Lam et al., 2011). EFB pellets showed higher particle densities than PKS, which possibly attribute to a larger number of small particles (Figure 3.1) and denser packing. All pre-treated pellets, except leached EFB pellets, showed reductions in porosity.

					Pellet	Particle	
	Pre-	Mass	Diameter	Length	density, ρ_b	density, ρ_p	Porosity,
Species	treatment	(g)	(mm)	(mm)	(g/cm^3)	(g/cm^3)	Eo (%)
		0.82					
		\pm		$21.9 \pm$			$19.0 \pm$
	Control	0.01	6.5 ± 0.0	0.5	1.14 ± 0.02	1.40 ± 0.00	1.42
		0.80					
EFB		\pm		$21.3 \pm$			$18.8 \pm$
	Leaching	0.03	6.5 ± 0.0	0.8	1.14 ± 0.03	1.41 ± 0.01	1.91
		0.81					
	Steam	±		$20.5 \pm$			$14.1 \pm$
	Explosion	0.03	6.5 ± 0.0	0.7	1.18 ± 0.02	1.37 ± 0.00	1.36
		0.76					
		±		$20.4 \pm$			$17.9 \pm$
	Control	0.02	6.5 ± 0.0	0.5	1.13 ± 0.03	1.37 ± 0.03	3.04
DVS		0.57					
глэ		±		$15.5 \pm$			$15.3 \pm$
	Leaching	0.02	6.5 ± 0.0	0.6	1.11 ± 0.04	1.31 ± 0.01	3.52
		0.79					
	Steam	±		$19.7 \pm$			$10.1 \pm$
	Explosion	0.03	6.5 ± 0.0	0.9	1.21 ± 0.03	1.35 ± 0.00	2.31

Table 3.2 Particle Densities and Dimensions of EFB and PKS Pellets (n = 5)

Note: n = number of repeated measurements.

Table 3.3 lists the mechanical energy input for pelletization. Metal concentration is a function of binding characteristics of the lignin functional group (Werner et al., 2000). Leached EFB pellets required lower compression energy, which indicated the removal of metal contents was effective while metal content removal was not effective in PKS pellets. Water leaching pretreatment did not result in a huge increase in pellet density, but it greatly reduced the energy input for making EFB pellet. Although the overall energy input for PKS was higher than EFB, PKS did not show any variation in compression energy and extrusion energy between control and leached PKS. The possible reason is that no physical structure alteration of PKS was due to leaching. Steam explosion increased the compression energy, reasoning that the increase in compression energy for steam exploded materials may be due

to the extra energy required to break the large and hard particles into small particles and to overcome the friction between rough particles in order to fill the pores between particles (Lam, 2011). Higher extrusion energy was due to the presence of higher extractives content and monosugars generated due to steam explosion (Lam et al., 2013b).

rubie 515 Micenanical Energy of Compression and Endaston of Peners (n - C)					
Species	Pre-treatment	Compression Energy (J/g)	Extrusion Energy (J/g)		
EFB	Control	30.15 ± 1.51	0.99 ± 0.10		
	Leaching	26.81 ± 2.04	1.68 ± 0.31		
	Steam Explosion	44.50 ± 4.56	5.81 ± 1.34		
PKS	Control	32.61 ± 1.04	3.95 ± 1.51		
	Leaching	32.24 ± 1.95	4.73 ± 0.49		
	Steam Explosion	31.81 ± 1.38	5.16 ± 0.45		

Table 3.3 Mechanical Energy of Compression and Extrusion of Pellets (n = 5)

Note: n = number of repeated measurements.

Table 3.4 shows the performance values of hardness tests. Shang et al. (2012) reported that the durability of pellets is well correlated with hardness values measured from the material testers. The hardness values are important in relating to the stress distribution of pellets is silo. The experimental data suggests that EFB had higher Meyer Hardness than PKS. This was because smaller particle size of EFB with high particle surface area allowing more lignin available for binding during pelletization. As a result, PKS pellets were much more brittle than EFB pellets. In the leaching process of EFB, the removal of metal content exposed more lignin for binding, thereby increasing the Meyer Hardness. The decrease of Meyer Hardness of leached PKS pellets was due to a short pellet length, in which small indentation on the pellet surface was sufficient enough to break the pellet apart. Thus, shorter pellet length showed much more brittle nature. Steam treatment had an effect in increasing Meyer Hardness of EFB and PKS pellets.

	Meyer Hardn	ness (N/mm ²)	Displacement (mm)			
Pre-treatment	EFB	PKS	EFB	PKS		
Control	1.82 ± 0.68	1.35 ± 0.23	0.35 ± 0.14	0.46 ± 0.49		
leaching	2.44 ± 0.47	0.97 ± 0.11	0.19 ± 0.03	0.14 ± 0.08		
Steam	2.97 ± 0.23	1.95 ± 0.89	0.28 ± 0.09	0.21 ± 0.16		

Table 3.4 Mechanical Impact due to Axial Loading on a Pellet (n = 3)

Note: n = number of repeated measurements.

3.2 Effectiveness of Pre-treatments on Ash Removal

3.2.1 Mass Recovery of EFB and PKS after Steam Explosion

Table 3.5 lists the operating conditions of steam explosion on EFB and PKS. The computer data logging systems recorded the pressure and temperature of the boiler (P1 and T1) and the reactor (P2 and T2), respectively. The pressure of saturated steam dropped slightly due to the transfer of saturated steam from the boiler to steam line and reactor under isothermal conditions. Pressure and temperatures of steam transfer before and after 5 minutes in the reactor were recorded for analyzing the severity of the steam explosion in accordance with the steam ejection. Typically, the steam temperature and pressure were maintained for 5 minutes for steam explosion reaction.

	Steam conditions at boiler before		Steam at rea	conditions ctor after	Steam Conditions at reactor after 5		
Temperature (°C)	steam	n transfer	steam	ı transfer		minutes	
	T1 (°C)	P1 (MPa)	T2 (°C)	P2 (MPa)	T2 (°C)	P2 (MPa)	
EFB							
120	117.8	0.17	127.8	0.17	123.0	0.13	
140	141.9	0.30	153.5	0.30	150.9	0.28	
160	164.8	0.58	165.4	0.55	178.2	0.62	
180	178.6	0.79	164.4	0.78	183.5	0.95	
200	192.4	1.17	182.3	1.14	187.0	1.16	
PKS							
120	122.4	0.17	127.5	0.17	124.9	0.17	
140	138.8	0.28	145.2	0.28	142.2	0.28	
160	163.2	0.53	140.4	0.52	164.4	0.55	
180	176.7	0.75	165.7	0.75	165.2	0.75	
200	196.3	1.26	183.5	1.25	184.7	1.25	

Table 3.5 Operating Condition of Steam Explosion on EFB and PKS

Table 3.6 shows the mass recovery of EFB after steam explosion at the reactor temperature from 120 to 200 °C. The decrease of the wet mass at 120 and 140 °C compared to the EFB at room temperature was due to the condensation of steam. Water was formed from the condensed steam at low pressure so that some of the biomass might be dissolved into the water. From 160 to 200 °C, hydrolysis of hemicelluloses took place within the reactor, but no significant dry mass loss was observed. In contrast, EFB showed a significant drop in dry mass from 27.0 to 20.7 g at 200 °C, reasoning that evaporation of volatile matter caused a significant total dry mass loss of 6.3 g in condensate. The total dry mass recovery of EFB was reported between 76.7 and 94.2%. Low dry mass recovery of 76.7 % at 200 °C steaming condition suggests that severe steaming condition took place. Thus, 180 °C was an adequate steaming condition, which required less energy input compared to 200 °C steaming condition.

Temperature (°C)	25	120	140	160	180	200
Total Wet Mass (g)	30.0	27.7	27.9	26.5	28.0	25.4
Total Dry Mass (g)	27.0	25.4	26.0	25.2	25.9	20.7
Dry Mass Loss in Condensate (g)	-	1.6	1.0	1.8	1.1	6.3
Total Dry Mass Recovery (%)	-	94.2	96.4	93.4	96.0	76.7

Table 3.6 Dry Mass Recovery after Steam Explosion on EFB with Mean Particle Size of 0.31 mm for 5 Minutes Reaction.

Note: 25 °C represents the input temperature of EFB.

Table 3.7 lists the mass recovery of PKS after steam explosion at the reactor temperature from 120 to 200 $^{\circ}$ C. The decrease of wet mass from 120 to 200 $^{\circ}$ C was solely due to the condensation of steam. As a result, the steam treatment was not successful in improving the binding quality of PKS pellets. Dry mass recovery was reported between 94.3 and 98.5%. Comparing to EFB, this suggests that evaporation of volatile matter was not prominent after 160 $^{\circ}$ C.

Table 3.7 Dry Mass Recovery after Steam Explosion on PKS with Mean Particle Size of 0.72 mm for 5 Minutes Reaction

Temperature (°C)	25	120	140	160	180	200
Total Wet Mass (g)	30.0	28.3	28.5	28.2	27.6	28.6
Total Dry Mass (g)	27.0	25.8	26.4	25.4	25.8	26.6
Dry Mass Loss in Condensate (g)	-	1.2	0.6	1.5	1.1	0.4
Dry Mass Recovery (%)	-	95.6	98.0	94.3	95.8	98.5

Note: 25 °C represents the input temperature of PKS.

3.2.2 Effect of Steam Explosion on Ash Content

Figure 3.4 demonstrates the ash content of EFB and PKS at different steam explosion conditions. Steam explosion increased ash content of EFB from 8.41 % to 9.77 %. From 120 to 140 $^{\circ}$ C, ash content did not increase, which shows steam explosion severity has not taken place. From 160 to 200 $^{\circ}$ C, the ash content showed a linear relationship with the increase of steam explosion severity. Compared to the ash content of the control PKS, the ash content of steam exploded PKS did not show much fluctuation throughout from 120 to 200 $^{\circ}$ C. EFB had a good linear fit of R² value 0.8942 while PKS showed a poor fit of R² value 0.1516. This suggests that linear relationship was only applicable on steam exploded EFB. Since PKS has lower accessible amount of cellulose than EFB, the effect of steam explosion of PKS was not as significant as on EFB. Generally speaking, loss of organic volatiles is the main reason causing the increase of the ash content of EFB after 160 $^{\circ}$ C (Horn et al, 2011).



Figure 3.4 Ash Content of Steam Exploded EFB and PKS at Different Steam Explosion Conditions

3.2.3 Mass Recovery of EFB and PKS due to Water Leaching

Water leaching was investigated for the mass recovery of EFB and PKS. The aim of water leaching was to reduce the ash content and metal contents. In this study, the effectiveness of water leaching on the ash content of EFB and PKS was evaluated from instantaneous to 120 minutes leaching time. The amount of the total dry and wet mass was measured from the recovery within the leachate and the residual on the filter paper. The mass balance of ash considered three components: the insoluble ash, the soluble ash in the leachate and the soluble ash in the moisture of the biomass. The insoluble ash was the amount of ash in the dry biomass. The soluble ash was the amount of ash dissolved into the leachate. After separating the biomass from the leachate, the remaining moisture on the biomass was calculated based on the amount of the moisture on biomass and the concentration of dissolved ash in the leachate.

Table 3.8 lists the wet and dry mass recovery of EFB due to water leaching. A significant amount of water uptake of EFB within the leachate was observed. The hydrophilic nature of EFB required a substantial amount of energy in the drying process. There was a decrease in the total dry mass from 4.4 to 3.9 g from 1 minute to 30 minutes leaching, followed by a slightly increase of dry mass from 30 to 120 minutes. It is believed that mineral re-absorption of EFB contributed to the increase of dry mass after 30 minutes. Mineral re-absorption could occur when the metal contents are leached from the smaller biomass particles faster than from the larger particles due to the larger surface area (Asiagwu, 2012). With this concern, leaching duration should be reduced and continuous leaching process should be introduced instead of a batch leaching process. The dry mass recovery was

reported from 87.9 to 95.1 %. The soluble ash in the moisture of EFB was around 10 % of the insoluble ash of the input EFB. The soluble ash in the moisture could be eliminated by introducing multiple stage leaching process. Approximately 50 % ash reductions from the input EFB was observed at 5 minutes with the decrease of insoluble ash from 0.2423 to 0.1201 g. Although extra approximately 0.2 g reduction was achieved in insoluble ash at 120 minutes, long leaching duration was not preferred within a continuous leaching process.

Duration (min)	0	1	3	5
Material Residues from the Leachate				
Wet Mass (g)	5.000	22.18	25.81	23.43
Dry Mass (g)	4.429	4.158	4.085	3.912
Material Residues from the Filtrate				
Wet Mass (g)	-	0.0866	0.0744	0.2795
Dry Mass (g)	-	0.0530	0.0460	0.1570
Total Material Residues				
Total Wet Mass (g)	5.000	22.27	25.88	23.71
Total Dry Mass (g)	4.429	4.211	4.131	4.069
Dry Mass Recovery (%)	-	95.1	93.3	91.9
Dry Mass Loss into Leachate (g)	-	0.2181	0.2981	0.3601
Mass Balance of Ash				
Ash Content in Total Dry Mass (%)	5.47	3.91	3.58	3.54
Insoluble and Soluble Ash in Biomass (g)	0.2423	0.1647	0.1478	0.1439
Soluble Ash in Leachate (g)	-	0.0776	0.0945	0.0984
Concentration of Soluble Ash (g Ash/g Leachate)	-	0.0009	0.0012	0.0012
Soluble Ash in the Moisture of Biomass (g)	-	0.0170	0.0260	0.0238
Insoluble Ash (g)	0.2423	0.1477	0.1218	0.1201
Soluble Ash (g)	-	0.0946	0.1205	0.1222

Table 3.8 Dry Mass Recovery after Leaching on EFB with Mean Particle Size of 0.31 mm under Room Temperature

Duration (min)	10	30	60	120
Material Residues from the Leachate				
Wet Mass (g)	24.98	23.25	25.09	25.36
Dry Mass (g)	3.869	3.672	3.840	3.831
Material Residues from the Filtrate				
Wet Mass (g)	0.2459	0.4012	0.3849	0.3646
Dry Mass (g)	0.1350	0.2190	0.2230	0.1970
Total Material Residues				
Total Wet Mass (g)	25.23	23.65	25.47	25.73
Total Dry Mass (g)	4.004	3.891	4.063	4.028
Dry Mass Recovery (%)	90.4	87.9	91.7	90.9
Dry Mass Loss into Leachate (g)	0.4251	0.5381	0.3661	0.4011
Mass Balance of Ash				
Ash Content in Total Dry Mass (%)	3.83	3.56	3.40	3.32
Insoluble and Soluble Ash in Biomass (g)	0.1535	0.1385	0.1380	0.1337
Soluble Ash in Leachate (g)	0.0888	0.1038	0.1043	0.1087
Concentration of Soluble Ash (g Ash/g	0.0011	0.0013	0.0013	0.0014
Leachate)	0.0011	0.0013	0.0013	0.0014
Soluble Ash in the Molsture of Blomass (g)	0.0237	0.0253	0.0281	0.0298
Insoluble Ash (g)	0.1298	0.1133	0.1099	0.1039
Soluble Ash (g)	0.1125	0.1291	0.1324	0.1384

Note: 0 min duration column shows the initial mass of EFB.

Table 3.9 lists the dry mass recovery of PKS after leaching. Comparing to EFB, PKS within the leachate had lower water uptake capacity. The low water uptake capacity of PKS is favorable in pellet storage under a humid environment, where the pellets would not disintegrate easily. Although all leaching durations showed reduction in dry mass, however, there was no specific point indicating that the mineral re-absorption had happened. The dry mass recovery was reported from 89.9 to 94.1 %. An insignificant amount of the soluble ash in the moisture of PKS was due to the low water uptake capacity. The insoluble ash at 1 minute showed the greatest reduction (approximately 25 %) from 0.0606 to 0.0455 g. After

one minute, the reduction of insoluble ash was almost identical. This suggests that long leaching duration for PKS was not necessary.

Duration (min)	0	1	3	5
Material Residues from the Leachate				
Wet Mass (g)	5.000	8.452	8.660	8.495
Dry Mass (g)	4.495	4.191	3.974	3.891
Material Residues from the Filtrate				
Wet Mass (g)	-	0.0611	0.1424	0.3144
Dry Mass (g)	-	0.0390	0.0940	0.1890
Total Material Residues				
Total Wet Mass (g)	5.000	8.513	8.803	8.809
Total Dry Mass (g)	4.495	4.230	4.068	4.080
Dry Mass Recovery (%)	-	94.1	90.5	90.8
Dry Mass Loss into Leachate (g)	-	0.2645	0.4265	0.4145
Mass Balance of Ash				
Ash Content in Total Dry Mass (%)	1.35	1.09	1.26	1.15
Insoluble and Soluble Ash in Biomass (g)	0.0606	0.0461	0.0512	0.0468
Soluble Ash in Leachate (g)	-	0.0145	0.0094	0.0138
Concentration of Soluble Ash (g Ash/g				
Leachate)	-	0.0002	0.0001	0.0001
Soluble Ash in the Moisture of Biomass				
(g)	-	0.0006	0.0005	0.0007
Insoluble Ash (g)	0.0606	0.0455	0.0507	0.0461
Soluble Ash (g)	-	0.0151	0.0099	0.0145

Table 3.9 Dry Mass Recovery after Leaching on PKS with Mean Particle Size of 0.72 mm under Room Temperature

Duration (min)	10	30	60	120
Material Residues from the Leachate				
Wet Mass (g)	9.141	8.243	8.912	8.874
Dry Mass (g)	4.020	3.683	3.958	3.889
Material Residues from the Filtrate				
Wet Mass (g)	0.1982	0.7333	0.2946	0.2675
Dry Mass (g)	0.1330	0.4930	0.1790	0.1510
Total Material Residues				
Total Wet Mass (g)	9.339	8.976	9.207	9.141
Total Dry Mass (g)	4.153	4.176	4.137	4.040
Dry Mass Recovery (%)	92.4	92.9	92.0	89.9
Dry Mass Loss into Leachate (g)	0.3415	0.3185	0.3575	0.4545
Mass Balance of Ash				
Ash Content in Total Dry Mass (%)	1.24	1.32	1.30	1.33
Insoluble and Soluble Ash in Biomass (g)	0.0513	0.0553	0.0536	0.0535
Soluble Ash in Leachate (g)	0.0093	0.0053	0.0070	0.0077
Concentration of Soluble Ash (g Ash/g Leachate)	0.0001	0.0001	0.0001	0.0001
Soluble Ash in the Moisture of Biomass				
(g)	0.0005	0.0003	0.0004	0.0004
Insoluble Ash (g)	0.0508	0.0550	0.0532	0.0532
Soluble Ash (g)	0.0098	0.0056	0.0074	0.0074

Note: 0 min duration column shows the initial mass of PKS.

3.2.4 Effect of Water Leaching on Ash Content

Similar to the ash content analysis of steam exploded materials at different conditions, the ash content of leached biomass was analyzed. Metal contents, such as Potassium or Calcium, cause slagging and fouling in the combustion furnace. The optimized leaching conditions for ash reduction with respect to the temperature or duration were studied by removing the metal contents through leaching. The optimized leaching duration was first determined, and then the optimized temperature was studied based on the optimized leaching duration.

Figures 3.5 and 3.6 show the ash content of water leached EFB and PKS at the first five minutes and the extended leaching times, respectively. EFB leaching achieved significant ash reduction from 5.47 to 3.91 % at one minute. However, mineral re-absorption caused the re-bounce on the ash content after five minutes as shown on Figure 3.6, as explained in the previous section. Thus, leaching duration should be short in facilitating an effective leaching process. On the contrary, ash reduction was not significant in PKS leaching throughout 120 minutes. Therefore, leaching process is not necessary for PKS.



Figure 3.5 Ash Content of Leached EFB and PKS at the First 5Minutes (The Initial Ash Contents of EFB and PKS before Leaching were Presented at 0 Minute. Water to Biomass Ratio = 20:1 and Temperature = $25 \ ^{\circ}C$)



Figure 3.6 Ash Content of Leached EFB and PKS after 5Minutes (The Initial Ash Contents of EFB and PKS before Leaching were Presented at 0 Minute in Figure 3.5. Water to Biomass Ratio = 20:1 and Temperature = $25 \ ^{\circ}C$)

EFB was further investigated at different temperatures for 5 minutes leaching duration, where the greatest reduction before mineral re-absorption took place. Figure 3.7 presents temperature effect from 25 to 55 $^{\circ}$ C for EFB. The ash content was ranging between 3.54 and 3.75 % and showed neither upward nor downward trend from 25 to 55 $^{\circ}$ C. It was believed that the short leaching duration did not allow sufficient heat transfer towards the EFB. Moreover, EFB could not increase the leaching kinetic rate with slow heat transfer. Therefore, increasing the temperature of water did not affect the ash removal performance during leaching.



Figure 3.7 Ash Content of Leached EFB from 25 to 55 °C for 5 Minutes (The Initial Ash Content of EFB before Leaching were Presented as 5.47 % in Table 3.1.)

3.2.5 Mass Balance Model on Ash Removal due to Leaching

Four stages of leaching process were carried out on EFB and PKS under room temperature for 5 minutes in each stage. The biomass to water ratio was *L* to V_n throughout different stages for both EFB and PKS. Figure E.1 shows multiple stage leaching of EFB. Since fresh water was used as input in every stage, it was assumed that the input mass ratio of the soluble ash in the distilled water was $y_n = 0.000000$. This ensured that no impurity was found in the input stream of distilled water at each stage. The values of x_n and x_{n+1} were the input and output mass ratio of soluble and insoluble ash in the biomass, respectively. x_{n+1} value was updated on x_n value in the subsequent stage so that the biomass with the ash reduction from previous stage could perform leaching on next stage. With the given ash content at different leaching stages and the L/V_n ratio, y_n values were calculated by Eq. (2-8). Table 3.10 shows different parameters of ash removal in the mass balance model of EFB and PKS at four different stages. The input ash content of EFB decreased from 5.47 to 2.63 % throughout 3 leaching stages and the input ash content of PKS decreased from 1.35 to 1.15 % by a single leaching stage. No more ash reduction was observed after stage 3 for EFB and stage 1 for PKS, respectively. The y_{n+1} values of EFB were almost identical at stage 2 and stage 3, which suggested that the majority of ash was removed at stage 1. The ash reduction in EFB at stage 1 was the greatest, suggesting that convection effect on EFB was dominant in removing ash. For stage 2 and stage 3 of EFB leaching, diffusion effect on PKS was dominant. Single stage leaching of PKS suggested that convection effect on PKS was dominant in removing ash, it is reasonable to hypothesize that a substantial amount of ash is exposed on the PKS particle surfaces for leaching.

	Input Ash Content (%)	Xn	y _n	x _{n+1}	y_{n+1}
EFB					
Stage 1	5.47 ± 0.08	0.057865	0.000000	0.036699	0.001059
Stage 2	$3.54~\pm~0.24$	0.036699	0.000000	0.031672	0.000205
Stage 3	$3.07~\pm~0.19$	0.031672	0.000000	0.027010	0.000190
Stage 4	$2.63~\pm~0.33$	0.027010	0.000000	-	-
PKS					
Stage 1	1.35 ± 0.23	0.013685	0.000000	0.011634	0.000103
Stage 2	1.15 ± 0.34	0.011634	0.000000	-	-
Stage 3	1.21 ± 0.09	0.012248	0.000000	-	-
Stage 4	1.21 ± 0.09	0.012248	0.000000	-	-

Table 3.10 Parameters of Ash Removal in Mass Balance Model of EFB and PKS at Four Different Stages (n = 3)

Note: n = number of repeated measurements.

3.2.6 Kinetic Models of Ash from EFB and PKS during Water Leaching

Kinetic models studied the leaching rate of ash removal from EFB and PKS. The particle size and metal distribution of biomass affected leaching performance. In this study, however, the studied reaction rates ignored these factors because of small particle size. It was assumed that there was no effect of metal distribution on the leaching rate. A typical leaching kinetic model is dependent on leaching duration and temperature (Ho et al, 2005). From Section 3.2.3, the temperature effect on ash removal was not significant in ash removal of both species. Therefore, the kinetic models were solely based on duration. The kinetics are able to study the leaching process in a microscopic level, such as initial leaching rate and leaching saturating capacity, which are critical parameters in designing the continuous process of leaching. The kinetic parameters are also important for batch process design.

Figure 3.8 illustrates the concentration of soluble ash (C_t) v.s. leaching duration of EFB and PKS at 25 °C. The concentration (C_t) of soluble ash of EFB and PKS was calculated from the given amount of leachate and soluble ash in Table 3.8 and Table 3.9. The concentration of soluble ash in EFB leachate increased steadily at the first 5 minutes from 0.94 to 1.21 g/L before saturation. It was observed that the concentration of soluble ash in PKS leachate saturated in the solution between 0.10 and 0.15 g/L after a slight increase from instantaneous to 1 minute. Ho et al. (2005) adopted a second order leaching model in predicting the leaching condition of Tilia Sapwood. Figure 3.9 shows the linear curve fittings of Eq. (2-12) to the C_t data for EFB and PKS species throughout 5 minutes. The intercept and the slope of the curves can estimate h, k and C_{sat} values. The estimated h, k and C_{sat} are listed on Table 3.11. The linearization leaching models of EFB and PKS were well fitted

within 5 minutes leaching time, of which R^2 values of EFB and PKS were 0.999 and 0.861, respectively. The modeled leaching trends of EFB and PKS were plotted on Figure 3.8. The trends level off after 3 minutes and 1 minute for EFB and PKS, repsectively. The concentrations increased with slower leaching rates. This is an indication that diffusion effect on ash migrating from biomass to leachate is dominant when the trends level off. Within the level off region, the convection effect on ash migration from biomass to leachate is dominant. The models suggested that the saturation capacity of both species would take place within 5 minutes. PKS showed a lower leaching capacity at saturation (C_{sat}) so that PKS was expected to require less amount of time to achieve saturation. Compared to PKS, a substantial amount of ash was exposed on the surface of EFB particles, which explained the initial leaching rate (h) was much greater than PKS.

Species	C _{sat} (g/L)	k (g-min/L)	h (g/L/min)	R^2
EFB	1.31	2.17	3.70	0.999
PKS	0.15	16.35	0.36	0.861

Table 3.11 Linearization of the Kinetic Models of EFB and PKS



Figure 3.8 Plot of the Concentration of Soluble Ash v.s. Time of EFB and PKS Leaching



Figure 3.9 Second–order Leaching Kinetics of the Concentration of Soluble Ash of EFB and PKS

3.3 Ash Composition for EFB and PKS at Different Leaching Temperatures

Although temperature did not affect the dry mass leaching rate, the removal of metal contents in dry mass might have been influenced by different temperatures. For instance, ion exchange of chemicals involved the passive diffusion of Potassium and Sodium between the intracellular and the extracellular matrix of the biomass. In the following, metal analysis of EFB and PKS leachate were conducted from 25 to 55 $^{\circ}$ C.

Table 3.12 illustrates the recovery of dry EFB and PKS ground particles after leaching from 25 to 55 °C. Considering that the amounts of EFB and PKS ground were limited for metal analysis, no replications were performed. Initially, 4.427 g EFB ground particles and 4.495 g PKS ground particles were used in leaching at three different temperature conditions at 25, 40 and 55 °C respectively. The data in EFB showed that the leaching recovery of all conditions was able to achieve 90.0 – 97.2 % recovery. PKS had higher material recovery than EFB, reasoning that ground EFB had higher ash content.

EFB			PKS			
Temperature	Dry	Dry biomass		Dry	Dry biomass	
(°C)	biomass	After leached	Recovery	biomass	After leached	Recovery
	(g)	(g)	(%)	(g)	(g)	(%)
25	4.427	4.069	91.9	4.495	4.080	90.8
40	4.427	3.987	90.0	4.495	4.287	95.4
55	4.427	4.087	92.3	4.495	4.367	97.2

Table 3.12 Recovery of the Dry EFB and PKS Ground due Water Leaching from 25 to 55 $^{\rm o}{\rm C}$ for 5 Minutes

Table 3.13 presents the metal contents of dry EFB and PKS before water leaching. The mass percentage of the metal content is defined as the amount of the metal content per the amount of dry biomass before leaching. The amount of the metal content was calculated by multiplying the weight of dry biomass with the measured amount of metal content in the dry biomass from the ICP results. The mass percentage was the amount of metal content in the dry biomass. Substantial amount of Potassium, 2.420 %, was found in EFB dry ground material. Approximately 0.314 and 0.171 % was reported for Calcium and Magnesium, respectively. Aluminum, Iron and Sodium were negligible with less than 0.1 %. PKS had a much lower metal content. Potassium had an amount of 0.233 %, which was around one tenth of EFB. Other metal contents are lower than 0.05 %. These results concurred to the lower ash content presented in PKS.

		,	U,	U,		
		EFB			PKS	
Metal			Mass			Mass
Content	Mass Ratio	Amount	Percentage	Mass Ratio	Amount	Percentage
	(mg/kg) ^a	(mg)	(%)	(mg/kg) ^a	(mg)	(%)
Al	88	0.39	0.009	50	0.22	0.005
Ca	3140	13.91	0.314	349	1.57	0.035
Fe	170	0.75	0.017	132	0.59	0.013
K	24200	107.18	2.420	2330	10.47	0.233
Mg	1710	7.57	0.171	341	1.53	0.034
Na	120	0.53	0.012	100	0.45	0.010

Table 3.13 Metal Contents of EFB and PKS Ground Particles before Water Leaching (Dry Biomass Weight (Dry Basis): EFB = 4.427 g, PKS = 4.495 g)

^aThe mass ratio is in mg of element per kg of dry biomass.

Table 3.14 depicts the metal contents in dry EFB and PKS after water leaching. The amount of metal content in the leachate was calculated by multiplying the measured mass concentration of metals from the ICP results with the leachate weight. The amount of metal

content in the leached biomass was calculated by subtracting the amount of metal content in the leachate from the original amount of metal content from the dry biomass (Table 3.13). The mass percentage was the amount of metal content in the leached biomass. At 25 °C leaching temperature, EFB and PKS showed reductions in all metal contents. A temperature rise from 25 to 55 °C did not have any significant reduction of Aluminum, Iron and Magnesium of EFB. From 25 to 40 °C, the percentage of Calcium and Potassium was halved in EFB. This is due to the active transport of these metal ions in biomass cell structure with the supply of heat. It is believed that the transport of Calcium and Potassium ions in EFB is governed by microbial activities (Karley et al., 2009). Sodium in EFB was almost completely removed, reasoning that the Sodium is available on the cell surface allowing it to be washed away completely. PKS showed similar trends in the ion exchange of Aluminum, Calcium, Potassium and Sodium, but not for Iron and Magnesium. This ion exchange occurred more rapidly at temperatures higher than 40 °C, resulting in greater reduction. Therefore, Iron and Magnesium ions may be more exposed on the surface of the PKS ground particles.

Metal Content	Al	Ca	Fe	K	Mg	Na
EFB 25 °C						
Mass Concentration (mg/L) ^b	2.2	98.0	4.5	986.0	37.0	6.0
Amount in leachate (mg)	0.18	7.88	0.36	79.27	2.97	0.48
Amount in leached biomass (mg)	0.21	6.03	0.39	27.91	4.60	0.05
Mass Percentage (%)	0.005	0.148	0.010	0.686	0.113	0.001
EFB 40 °C						
Mass Concentration (mg/L) ^b	2.1	134.0	4.1	1140.0	44.7	5.5
Amount in leachate (mg)	0.17	11.03	0.34	93.83	3.68	0.45
Amount in leached biomass (mg)	0.22	2.88	0.41	13.35	3.89	0.08
Mass Percentage (%)	0.005	0.072	0.010	0.335	0.098	0.002
EFB 55 °C						
Mass Concentration (mg/L) ^b	2.0	126.0	4.0	1140.0	41.6	6.5
Amount in leachate (mg)	0.16	10.24	0.32	92.65	3.38	0.53
Amount in leached biomass (mg)	0.23	3.67	0.43	14.54	4.19	< 0.1
Mass Percentage (%)	0.006	0.09	0.011	0.356	0.103	~ 0
PKS 25 °C						
Mass Concentration (mg/L) ^b	0.2	1.6	0.5	31.4	2.3	2.0
Amount in leachate (mg)	0.02	0.15	0.05	2.99	0.21	0.19
Amount in leached biomass (mg)	0.21	1.41	0.55	7.48	1.32	0.26
Mass Percentage (%)	0.005	0.035	0.013	0.183	0.032	0.006
PKS 40 °C						
Mass Concentration (mg/L) ^b	1.1	10.5	4.0	95.3	9.9	2.4
Amount in leachate (mg)	0.09	0.91	0.35	8.29	0.86	0.21
Amount in leached biomass (mg)	0.13	0.66	0.25	2.19	0.67	0.24
Mass Percentage (%)	0.003	0.015	0.006	0.051	0.016	0.006
PKS 55 °C						
Mass Concentration (mg/L) ^b	0.7	8.8	2.8	100.0	9.8	2.2
Amount in leachate (mg)	0.05	0.71	0.23	8.09	0.79	0.18
Amount in leached biomass (mg)	0.17	0.86	0.36	2.38	0.74	0.27
Mass Percentage (%)	0.004	0.020	0.008	0.054	0.017	0.006

Table 3.14 Metal Contents of Leached EFB and PKS Ground Particles from 25 to 55 $^{\circ}$ C for 5 Minutes (Water to Biomass Ratio = 20 : 1. Dry Biomass Weight (Dry Basis): EFB = 4.427 g, PKS = 4.495 g. Leachate Weight: EFB = 80.4 g, PKS = 95.3 g)

^bThe mass concentration is conducted through suspension (mg of metals per L of suspension)

Chapter 4 Techno-Economic Analysis of Water Leaching

4.1 Process Economy of Pilot Scale Water Leaching Facility

A proposed leaching facility is assumed to be installed at a 25 MW power plant. The capacity of a leaching facility for a 25 MW power plant should be 135 metric ton/ day (dry basis) EFB. EFB ground is also assumed with HHV of 16 MJ/kg for 20 % power plant conversion efficiency (Bakker, 2000). Table 4.1 lists the system components of 135 metric ton/ day (dry basis) EFB leaching facility. The list presents the estimated throughput capacity, the power requirement and the estimated capital cost. The component list was established in Bakker's thesis (Bakker, 2000). The capacity of the components with more than 5.7 metric ton/ hour is sourced in order to match up with 135 metric ton/ day for daily of biomass power generation input. It is assumed that the tap water used in the facility has the same leaching performance as the distilled water in this research. Water to biomass ratio and leaching duration are critical in designing the water bath component. For five minute leaching duration, the water bath capacity should be able to fill up 10 metric ton water and 0.5 metric ton EFB. It is assumed that each batch loading and unloading requires between 2 to 3 minutes. This can be achieved by a 4 m by 4 m square bath of 1 m depth. Shorter leaching duration can allow smaller size of water bath, leading to capital cost reduction. An additional 0.5 % parasitic load for the 25 MW power plant is contributed by the total power requirement of leaching (126 kW). The capital cost of the components should be lower than the estimated amount due to technology advancement of the components since ten years (Bakker, 2000). The system boundary of the leaching process does not include the cost of biomass. Since the input is EFB ground material, the system boundary of the leaching

process ignores the capital cost of size reduction unit. The system boundary of the leaching process also ignores the leachate disposal cost because the drainage of the leachate could be sold as fertilizer. Other cost with 15 % total component costs are estimated based on the taxation system and the insurance plan in British Columbia, Canada.

Component	EFB Capacity (Metric tons/hour)	Power Requirement (kW)	Capital Cost (\$)	Source
Inclined feeder table + sprays	4 - 8	37	100,000	Cameco, Thibodeaux, LA
Wash bath with drag chain	variable	22	125,000	Cameco, Thibodeaux, LA
Dewatering	4 - 6	52	350,000	Fulton Iron Works St. Louis, MO
Pumps for water/leachate		15	20,000	Hugot, 1986
Foundation, concrete flooring			62,050	Hugot, 1986
Other (15%)			98,557	
Total		126	755607	

Table 4.1 System Components of a 135 metric tons/ day Industrial EFB Leaching Facility

Table 4.2 presents the increased fuel cost of 135 metric ton/ day EFB leaching facility. Capital recovery cost is calculated based on 10 year economic life and 12 % annual rate of capital investment obtained from Table 4.1. Electricity cost for leaching process is calculated based on system availability of 7000 hour per year and an electricity rate of \$ 0.0942 kWh⁻¹ in British Columbia, Canada (BC Hydro, 2013). The electricity rate is based on medium general service conservation rate with peak demand of facility between 85 – 150 kW. Labour cost is estimated at \$ 20 per hour in accordance with 1.33 man-hours needed per hour of operation. This cost includes an operator during day time and a technician to carry out maintenance. Water cost is based on water rate at \$ 0.68 per metric ton water in the City of

Surrey, British Columbia (City of Surrey, 2013) and water to biomass ratio of 20 to 1. Assumption has been made to EFB so that EFB combustion would not reduce steam generating capacity of the power plant. Maintenance is considered at 10 % of the capital cost. It is computed that 52.8 % of total increased cost of \$ 25.75 per metric ton is substantially contributed by the water cost of \$ 13.60 per metric ton. The water rate was increased substantially from \$ 0.22 to 0.68 per metric ton water from 2004 to 2013 (City of Surrey, 2013). The significant increase was due to the expansion of the Greater Vancouver Water District's (GVWD) infrastructure. In addition, water scarcity due to high water demand leads to increase of water rate globally throughout decades (Maxwell, 2012). As a result, water rate is the primary concern in establishing a leaching facility.

Capital Investment (\$)	\$	755,607
Capital Recovery Cost	\$/metric ton	3.39
Electricity (\$ 0.0942 per kWh)	\$/metric ton	2.11
Labour cost (\$20/hour)	\$/metric ton	4.73
Water cost (\$0.68/metric ton water)	\$/metric ton	13.60
Maintenance (10% capital cost)	\$/metric ton	1.92
Total Increased cost	\$/metric ton	25.75

Table 4.2 Total Increased Fuel Cost for EFB Leaching:135 metric ton/ day System Size

4.2 Sensitivity Analysis of Water Leaching Process

Figure 4.1 shows the total increased cost of leaching systems at different daily capacity. The capacity of the base system is 135 metric ton per day and the other four scaleup systems are 169, 203, 236, 270 metric ton/ day. The base system and other scaled-up systems are compatible with 20% power input for 25, 31, 38, 44, 50 MW power plants, respectively. A scale factor 0.64 is utilized in the scaling correction of the capital cost at
different sizes of the leaching system (Bakker, 2000). Considering the increase of the system sizes, labour requirements increases gradually (i.e. 1.33, 1.50, 1.67, 1.83, 2.00 man-hours per hour of operation from the base system to the scale-up systems). By scaling up the system size by one fold, the increased cost for EFB leaching is brought down from \$ 25.75 to 20.28 per metric ton. Scaling up the facilities reduces all the increased cost except water cost. This suggested that the larger facilities are preferable over the smaller facilities, given that the water to biomass ratio does not change.



Figure 4.1 Total Increased Cost for EFB Leaching for 135 – 270 metric ton/day Facilities

In order to evaluate the effectiveness of the leaching process on power generation performance, the total increased cost should be compared to the revenue of electricity due to EFB combustion. It is assumed that the total increased cost is equivalent to the delivered fuel cost. The revenue associated with EFB firing is dependent on the revenue of electricity (\$ kWh⁻¹), the delivered fuel cost, the production costs of the power plant and the power plant conversion efficiency. Figure 4.1 presents the revenue for leaching of EFB at 20 %

power conversion efficiency. It is also assumed that the HHV of EFB is 16 MJ/kg. The electric power production is 889 kWh per metric ton (dry basis). The production costs, including ash disposal, emission control and fuel handling, are estimated at \$ 6.50 per metric ton for a 50 MW power plant (Bakker, 2000). For example, the available revenue for fuel leaching with the delivered fuel cost of \$ 25 per metric ton is \$ 39.62 (= 889 * 0.08 - 25 - 6.50), \$48.51 and \$ 57.40 per metric ton for an electricity revenue of \$ 0.08, 0.09, 0.10 kWh⁻¹, respectively (Figure 4.2). Leaching process is feasible for the delivered fuel cost of \$ 25.75 per metric ton with electricity revenue of \$ 0.08, \$ 0.09 and \$ 0.10 kWh⁻¹. This indicates that the current business electricity rate is capable of covering all the delivered fuel cost and production cost. Still, government incentives are necessary in order to promote the biomass leaching process prior biomass combustion.



Figure 4.2 Revenue Available for Leaching of EFB at 20 % Power Conversion Efficiency

Another alternative in increasing the economic feasibility is to install a power generation system with higher conversion efficiency. Figure 4.3 shows the conversion efficiency of the base system at 20 % and the improved system with higher conversion efficiency at 30 %. The former one has lower production cost of \$ 6.50 per metric ton while the latter one has higher production cost of \$ 10.00 per metric ton. Both systems are estimated at the same electric electricity revenue (\$ 0.09 kWh⁻¹). The increase of the revenue of \$ 36.51 per metric ton has been observed in increasing the power efficiency from 20 to 30 %. It is expected that the increase of the revenue is due to higher power output per unit of fuel (i.e. 1333 kWh per metric ton instead of 889 kWh). Although the analysis assumed that the leaching facility is located in Canada, the delivered fuel cost could be greatly reduced by combining leaching and pelletization facility near the source of the feedstock (i.e. Malaysia). Although the electricity rate in Canada (CAD \$ 0.0942 kWh⁻¹) and Malaysia (USD \$ 0.09 kWh⁻¹ (Malaysia Investment Development Authority, 2013)) are comparable, the transportation cost of the feedstock could be significantly reduced. The production costs could be minimized by selling the by-products of combustion, such as the bottom ash for reinforcing cement in building industry.



Figure 4.3 Revenue Available for Leaching of EFB with Different Power Efficiency at \$ 0.09 kWh^{-1}

Chapter 5 Conclusions and Future Work

5.1 Conclusions

Physical characterizations of EFB and PKS at different pre-treatment conditions were performed for the control, leached and steam exploded pellet. Only steam exploded EFB increased the high heat value. Leaching reduced ash content, but steam explosion increased ash content of EFB and PKS pellets. Although steam explosion did show improvement in binding ability due to the increase of particle densities and hardness, steam explosion only improved the pellet quality as a fuel for transportation instead of combustion. Leached EFB showed better performance both in pelletization, transportation and combustion process with lower pelletizing energy, higher hardness and lower ash content, compared to EFB control. Neither steam explosion nor water leaching was required for PKS in improving biofuel quality.

Since the end-use of pellet was for power generation, the ash content of the feedstock was the main concern for the combustion process. Different pre-treatment conditions have been studied in steam explosion and leaching. It was reported that the ash content of EFB increased after the steam explosion, but there was no effect in PKS. Mineral re-absorption during leaching suggested that short leaching duration was critical to a small continuous stirring reactor for an effective ash removal. In addition, the mass balance model suggested that the multiple stage leaching is necessary to remove the soluble ash remained on the moisture of biomass in order to achieve the greatest reduction of ash. From the microscopic point of view, the removal kinetic of the soluble ash of EFB and PKS was only dependent on the leaching duration, but not the temperature. The experimental results recommended that the optimal leaching condition at room temperature is 5 minutes.

The metal contents of EFB were much higher than PKS from the ICP analysis. It was found that the removal quantity of a certain metals, i.e., Calcium and Potassium from EFB and Potassium, Magnesium and Iron from PKS, were increased with the leaching temperature. The significant reduction of Potassium can prevent the formation of oxide layer, which enables higher thermal power efficiency in combustion process.

The capital and the increased cost of the leaching facility have been revealed for EFB ash removal process. It was found that the increased cost heavily depends on the water price. With this regard, water price has been increased by three fold within ten years, which suggested that the feasibility of establishing a leaching facility will be decreased due to the increase of the increased cost over time. Throughout the sensitivity analysis, it was concluded that the feasibility of establishing a leaching facility can be assisted by government incentives for biomass leaching, geographic advantage in leaching prior pelletization and installation of power plant with higher thermal conversion efficiency.

5.2 Future Work

5.2.1 Reactor Design of Water Leaching Process

This research adopted a batch leaching process to study the leaching factors, such as temperature and leaching duration. However, it was found that the distilled water to biomass ratio can further be optimized to reduce the ash content. In order to bring down the equipment capital costs and the maintenance costs, a moving bed reactor with multiple stage leaching with several stirring tanks would be a viable option for an efficient and costeffective ion-exchange process. In addition, the use of a moving bed reactor can avoid the mineral re-absorption within the leaching fluid due to different particle sizes and long leaching duration. To design a moving bed reactor, different flow rates of the biomass and distilled water and different particle size of biomass could be simulated through a computational fluid dynamic (CFD) interface to obtain the suggested optimum geometry of the moving bed reactor for effective leaching. This can be further validated with the experimental data obtained from the in house built moving bed reactor.

5.2.2 Material Handling of Water Leaching Process

There are two major challenges in material handling after the biomass leaching: the dewatering of the leached biomass and the disposal of the leached liquid. The dewatering process involves the substantial amount of heat supply in removing the moisture content of the leached biomass prior to pelletization. Different drying alternatives should be investigated in order to propose a low energy intensity and fast drying process. For instance, typical drying kiln for biomass watering requires a substantial amount of heat supply, which increases the overall operating costs. One possible solution to bring down the operating costs might be a mechanical rotary drying machine, which makes use of the fluid convection in removing biomass moisture by applying a circular motion. Discharge of leached liquid into sewage might lead to the concern of disposal costs. To overcome this problem, reverse osmosis of leached liquid can recycle the water back into the leaching process. The recovered metal

contents might be utilized for other potential applications such as cement production, fertilizer productions, etc.

5.2.3 Combination of Steam Explosion and Water Leaching Pre-treatments

This research showed that steam explosion was able to increase the hydrophobicity and durability of pellets while leaching was effectively in removing metal content in ash. By combining these two pre-treatment processes prior to pelletization, the pre-treated pellets might resolve both the dust explosion during transportation and the slagging issues of the combustion furnace. This combination of the two pre-treatments is worth to investigate the sequences in order to maximize the effectiveness in increasing durability and reducing ash content of the oil palm residue pellets.

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Appendix A: Supplemental Information for Literature Review

Research	Reference
Understanding the formation of alkali-iron trisulfates and their roles in the corrosion of steam superheaters in conventional boiler systems.	Bryers, 1977
Raask's Method was developed to determine the weight percentage of quartz, kaolinite, pyrite and calcite of combustion fuel within a furnace.	Raask , 1985
Ash deposition was caused by sodium rich glass/liquid cementing phase and recrystalized alkaline earth aluminosilicates at temperature above 1090 °C and by low melting sulphate-rich phases that bond deposits at lower temperature.	Benson, 1992
The fuel quality of herbaceous specie was much lower compared to wood, due to higher ash content and high percentage of silica, potassium and sodium.	Olanders et al., 1995
Fouling rate of biomass was reduced after applying water leaching on rice straw and wheat straw.	Jenkins et al., 1996
Banagrass showed substantial reductions in ash (45%), K(90%), Cl (98%), S (55%), Na (68%), P (72%) and Mg (68%).	Turn et al., 1997
Leaching biomass effectively reduced or eliminated the release of alkali metal vapors during combustion.	Dayton et al., 1999
Leaching could improve ash quality but it was insufficient to prevent formation of agglomerate during fluidized combustion.	Arvelakis et al., 2001

Table A.1 Summary of Research Findings in Biomass Leaching

Analysis	Procedure	Reference
Proximate	Simultaneous Thermal Analyzer called for biomass sample loading and weighing at 25 °C, heating to 110 °C, holding for 10 minutes then heating to 950 °C, where the temperature was held until constant weight was achieved in nitrogen. Then, the biomass sample atmosphere was switched to air (somewhat diluted by the balance purge). When the weight was again constant after the combustion of the fixed carbon component, the analysis was ended and the apparatus cooled for the next analysis.	Cassel et al., 2012
Elemental	Elemental analysis of air-dried biomass samples was performed using CHNS-O Elemental Analyzer (Perkin-Elmer 2400 Series). Samples were ground and each agro waste (1 mg, dry basis) was weighed on tin foil and placed into elemental furnace and subjected to complete combustion in a pure oxygen environment. For oxygen content analysis, sample (1 mg) was placed inside a silver capsule and placed into furnace and heated at 1000 °C. After complete combustion, samples were screened using infrared detector to determine oxygen content.	Sugumaran S. and S. Seshadri, 2009

Table A.2 Analytical Procedure of Proximate and Elemental Analysis in Biomass

Appendix B: Process Flow Diagram of the Steam Explosion Unit



Figure B.1 Process Flow Diagram of the Steam Explosion Unit (B: Ball Valve, PS: Pressure Relief Valve, T: Thermocouple, P: Digital Pressure Transducer) (Lam, 2011)

Appendix C: Raw Data of Physical Characterization of EFB and PKS Pellets

Pre-treatment	EFB Before Drying (g)	EFB After Drying (g)	Moisture Content (%)
Control	0.6320	0.5560	13.6691
Control	0.6180	0.5480	12.7737
Control	0.6520	0.5740	13.5889
Leaching	0.6300	0.5380	17.1004
Leaching	0.6300	0.5340	17.9775
Leaching	0.6460	0.5460	18.3150
Steam Explosion	0.6200	0.5760	7.6389
Steam Explosion	0.5820	0.5420	7.3801
Steam Explosion	0.6300	0.5900	6.7797

Table C.1 Moisture Content of EFB Ground Material

Table C.2 Moisture Content of PKS Ground Material

Pre-treatment	PKS Before Drying (g)	PKS After Drying (g)	Moisture Content (%)
Control	0.6320	0.5560	13.6691
Control	0.6360	0.5620	13.1673
Control	0.6820	0.6000	13.6667
Leaching	0.6280	0.5560	12.9496
Leaching	0.6320	0.5560	13.6691
Leaching	0.6120	0.5380	13.7546
Steam Explosion	0.6600	0.6180	6.7961
Steam Explosion	0.6600	0.6200	6.4516
Steam Explosion	0.6440	0.6020	6.9767

		High Heat Value - Wet	High Heat Value - Dry Basis
Pre-treatment	EFB (g)	Basis (MJ/kg)	(MJ/kg)
Control	0.5277	15.3661	17.7635
Control	0.5739	16.0097	18.5075
Control	0.5361	16.7432	19.3554
Leaching	0.5228	15.8574	18.3315
Leaching	0.5191	15.5466	17.9722
Leaching	0.5127	15.6330	18.0720
Steam Explosion	0.5150	20.0776	23.2101
Steam Explosion	0.5092	20.6513	23.8733
Steam Explosion	0.5305	21.6412	25.0177

Table C.3 High Heat Value of EFB Ground Material

Table C.4 High Heat Value of PKS Ground Material

		High Heat Value - Wet	High Heat Value - Dry Basis
Pre-treatment	PKS (g)	Basis (MJ/kg)	(MJ/kg)
Control	0.5112	18.1104	20.9360
Control	0.5079	19.7425	22.8227
Control	0.5472	17.9671	20.7704
Leaching	0.4919	18.4209	21.2949
Leaching	0.5657	18.5822	21.4814
Leaching	0.5200	18.2022	21.0421
Steam Explosion	0.5093	20.5510	23.7574
Steam Explosion	0.5608	20.3139	23.4833
Steam Explosion	0.5209	20.6602	23.8836

Pre-treatment	EFB (g)	EFB Ash (g)	Ash Content (%)
Control	0.5380	0.0260	4.8327
Control	0.5480	0.0260	4.7445
Control	0.5740	0.0280	4.8780
Leaching	0.5380	0.0140	2.6022
Leaching	0.5340	0.0120	2.2472
Leaching	0.5460	0.0140	2.5641
Steam Explosion	0.5760	0.0480	8.3333
Steam Explosion	0.5420	0.0440	8.1181
Steam Explosion	0.5900	0.0460	7.7966

Table C.5 Ash Content of EFB Ground Material

Table C.6 Ash Content of PKS Ground Material

Pre-treatment	PKS (g)	PKS Ash (g)	Ash Content (%)
Control	0.5560	0.0060	1.0791
Control	0.5620	0.0060	1.0676
Control	0.6000	0.0085	1.4167
Leaching	0.5560	0.0040	0.7194
Leaching	0.5560	0.0060	1.0791
Leaching	0.5380	0.0020	0.3717
Steam Explosion	0.6180	0.0120	1.9417
Steam Explosion	0.6200	0.0100	1.6129
Steam Explosion	0.6020	0.0120	1.9934

	Mass	Diameter	Length	Pellet Density	Pellet Density
Pre-treatment	(g)	(mm)	(mm)	(kg/m^3)	(g/cm^3)
Control	0.8155	6.4900	21.1200	1167.2148	1.1672
Control	0.8177	6.5000	22.1600	1112.0073	1.1120
Control	0.8214	6.4900	21.7600	1141.0812	1.1411
Control	0.8257	6.4900	22.0600	1131.4556	1.1315
Control	0.8289	6.4600	22.3600	1131.0334	1.1310
Leaching	0.7680	6.4500	21.3600	1100.4002	1.1004
Leaching	0.8016	6.4500	21.2600	1153.9451	1.1539
Leaching	0.7740	6.4800	19.9700	1175.2304	1.1752
Leaching	0.8321	6.4900	22.0800	1139.1927	1.1392
Leaching	0.8294	6.4800	21.7900	1154.1624	1.1542
Steam Explosion	0.8141	6.5000	21.1100	1162.1787	1.1622
Steam Explosion	0.8113	6.5200	20.8700	1164.3243	1.1643
Steam Explosion	0.8251	6.5100	20.8300	1190.0507	1.1901
Steam Explosion	0.8213	6.5100	20.4800	1204.8140	1.2048
Steam Explosion	0.7532	6.5200	19.3600	1165.2521	1.1653

Table C.7 Pellet Density and Dimensions of EFB Pellets

Table C.8 Pellet Density and Dimensions of PKS Pellets

	Mass	Diameter	Length	Pellet Density	Pellet Density
Pre-treatment	(g)	(mm)	(mm)	(kg/m^3)	(g/cm^3)
Control	0.7329	6.4800	19.5000	1139.6466	1.1396
Control	0.7868	6.4700	20.6700	1157.7784	1.1578
Control	0.7771	6.5200	20.3900	1141.4966	1.1415
Control	0.7432	6.5100	20.8800	1069.3586	1.0694
Control	0.7604	6.4900	20.3700	1128.4227	1.1284
Leaching	0.5645	6.5000	14.9400	1138.6671	1.1387
Leaching	0.5471	6.4500	14.8600	1126.7786	1.1268
Leaching	0.5911	6.4700	15.9400	1127.9094	1.1279
Leaching	0.5864	6.4900	15.9200	1113.4523	1.1135
Leaching	0.5454	6.4900	15.8600	1039.5196	1.0395
Steam Explosion	0.7458	6.5000	19.1400	1174.2589	1.1743
Steam Explosion	0.8192	6.4900	20.6900	1196.8788	1.1969
Steam Explosion	0.8265	6.5200	20.4900	1208.1360	1.2081
Steam Explosion	0.7823	6.4800	19.4000	1222.7332	1.2227
Steam Explosion	0.7800	6.4900	18.6400	1264.9385	1.2649

							Density
Pre-treatment	P1	P2	Vp	Vc	Vr	Pm	(g/cm^3)
Control	17.0550	5.2150	2.9282	29.4200	11.6685	3.2704	1.4045
Control	17.0820	5.2240	2.9337	29.4200	11.6685	3.2699	1.4019
Control	17.0790	5.2240	2.9404	29.4200	11.6685	3.2693	1.3987
Control	17.0850	5.2240	2.9270	29.4200	11.6685	3.2705	1.4051
Control	17.1250	5.2360	2.9253	29.4200	11.6685	3.2706	1.4059
Leaching	17.0350	5.1970	2.8410	29.4200	11.6685	3.2779	1.4103
Leaching	17.0580	5.2030	2.8335	29.4200	11.6685	3.2785	1.4140
Leaching	17.0840	5.2110	2.8340	29.4200	11.6685	3.2784	1.4138
Leaching	17.0600	5.2070	2.8584	29.4200	11.6685	3.2764	1.4017
Leaching	17.0450	5.2010	2.8479	29.4200	11.6685	3.2773	1.4069
Steam Explosion	17.1020	5.2310	2.9401	29.4200	11.6685	3.2694	1.3676
Steam Explosion	17.0840	5.2240	2.9292	29.4200	11.6685	3.2703	1.3727
Steam Explosion	17.0760	5.2230	2.9398	29.4200	11.6685	3.2694	1.3678
Steam Explosion	17.0420	5.2110	2.9281	29.4200	11.6685	3.2704	1.3732
Steam Explosion	17.0590	5.2170	2.9339	29.4200	11.6685	3.2699	1.3705

Table C.9 Particle Density of EFB Pellets

Table C.10 Particle Density of PKS Pellets

							Density
Pre-treatment	P1	P2	Vp	Vc	Vr	Pm	(g/cm^3)
Control	17.0910	5.2060	2.7816	29.4200	11.6685	3.2829	1.3561
Control	17.0210	5.1850	2.7840	29.4200	11.6685	3.2827	1.3549
Control	17.0740	5.1960	2.7460	29.4200	11.6685	3.2860	1.3736
Control	17.0560	5.1940	2.7717	29.4200	11.6685	3.2838	1.3609
Control	17.0690	5.1810	2.6463	29.4200	11.6685	3.2945	1.4254
Leaching	17.0410	5.1110	2.1837	29.4200	11.6685	3.3342	1.2884
Leaching	17.0540	5.1090	2.1388	29.4200	11.6685	3.3380	1.3155
Leaching	17.0570	5.1110	2.1472	29.4200	11.6685	3.3373	1.3103
Leaching	17.0450	5.1050	2.1288	29.4200	11.6685	3.3389	1.3216
Leaching	17.0180	5.0980	2.1371	29.4200	11.6685	3.3382	1.3165
Steam Explosion	17.0350	5.2090	2.9291	29.4200	11.6685	3.2703	1.3449
Steam Explosion	17.0570	5.2150	2.9238	29.4200	11.6685	3.2708	1.3473
Steam Explosion	17.0570	5.2130	2.9091	29.4200	11.6685	3.2720	1.3541
Steam Explosion	17.0520	5.2120	2.9130	29.4200	11.6685	3.2717	1.3523
Steam Explosion	17.0560	5.2130	2.9114	29.4200	11.6685	3.2718	1.3531

Pre-treatment	Compression Energy (J)	Extrusion Energy (J)
Control	25.0635	0.7421
Control	22.3171	0.9233
Control	25.3824	0.8236
Control	24.0577	0.7256
Control	23.8007	0.7318
Leaching	22.7743	1.0607
Leaching	20.0021	1.5740
Leaching	19.3773	1.1275
Leaching	22.3496	1.6060
Leaching	22.7421	1.3392
Steam Explosion	35.3839	3.2755
Steam Explosion	39.7547	6.1909
Steam Explosion	38.3970	4.9686
Steam Explosion	33.8578	4.5913
Steam Explosion	30.5968	4.2143

Table C.11 Energy Consumption of EFB Pellets

Table C.12 Energy Consumption of PKS Pellets

Pre-treatment	Compression Energy (J)	Extrusion Energy (J)
Control	26.6476	2.4233
Control	25.5643	4.1522
Control	25.6861	2.3247
Control	27.2554	4.7601
Control	25.2787	2.1449
Leaching	26.3303	3.5236
Leaching	27.7125	3.4007
Leaching	24.9866	4.4023
Leaching	23.5882	3.6793
Leaching	26.3332	3.8733
Steam Explosion	26.1478	3.6289
Steam Explosion	26.3798	4.5364
Steam Explosion	23.6155	4.4105
Steam Explosion	25.8162	3.9900
Steam Explosion	25.2920	4.0886

Pre-treatment	Meyer Hardı	ness (N/mm ²)	Displacement (mm)	
	EFB	PKS	EFB	PKS
Control	12.1609	2.2057	0.3209	1.0194
Control	15.1554	25.2862	0.2266	0.1027
Control	3.5881	7.8824	0.5031	0.2617
Leaching	21.4382	14.4495	0.2195	0.1194
Leaching	26.6769	7.1179	0.1532	0.2271
Leaching	16.9844	19.5092	0.1861	0.0698
Steam Explosion	27.3699	11.6692	0.1857	0.3952
Steam Explosion	14.4298	44.7052	0.3129	0.0775
Steam Explosion	14.7618	10.4280	0.3528	0.1526

Table C.13 Meyer Hardness of EFB and PKS Pellets

Appendix D: Raw Data of Effectiveness of Pre-treatments on Ash Removal

			Ash			Ash
Temperature (°C)		Ash	Content			Content
	EFB (g)	(g)	(%)	PKS (g)	Ash (g)	(%)
120	0.5100	0.0440	8.6275	1.0863	0.0220	2.0253
120	0.5880	0.0480	8.1633	1.0662	0.0170	1.5945
120	0.5340	0.0450	8.4270	1.0178	0.0220	2.1616
140	0.5300	0.0450	8.4906	1.0803	0.0170	1.5736
140	0.5170	0.0440	8.5106	1.1128	0.0190	1.7074
140	0.5530	0.0490	8.8608	1.0460	0.0160	1.5297
160	0.5060	0.0440	8.6957	0.9850	0.0170	1.7259
160	0.5160	0.0440	8.5271	1.0920	0.0170	1.5568
160	0.5580	0.0490	8.7814	0.9440	0.0180	1.9068
180	0.5290	0.0480	9.0737	0.9760	0.0140	1.4344
180	0.5160	0.0470	9.1085	1.1350	0.0210	1.8502
180	0.5360	0.0500	9.3284	1.0160	0.0150	1.4764
200	0.5520	0.0536	9.7101	1.0410	0.0160	1.5370
200	0.5190	0.0500	9.6339	1.0570	0.0210	1.9868
200	0.5720	0.0570	9.9650	1.0870	0.0180	1.6559

Table D.1 Ash Content of Steam Exploded EFB and PKS

Duration			Ash Content			Ash Content
(min)	EFB (g)	Ash (g)	(%)	PKS (g)	Ash (g)	(%)
1	0.5400	0.0230	4.2593	0.5230	0.0050	0.9560
1	0.5130	0.0210	4.0936	0.5680	0.0050	0.8803
1	0.5030	0.0170	3.3797	0.5580	0.0080	1.4337
3	0.5360	0.0180	3.3582	0.5280	0.0060	1.1364
3	0.5660	0.0210	3.7102	0.5230	0.0070	1.3384
3	0.5730	0.0210	3.6649	0.5390	0.0070	1.2987
5	0.5100	0.0180	3.5294	0.5070	0.0070	1.3807
5	0.5030	0.0190	3.7773	0.5290	0.0040	0.7561
5	0.6050	0.0200	3.3058	0.5360	0.0070	1.3060
10	0.5250	0.0210	4.0000	0.5630	0.0090	1.5986
10	0.5290	0.0190	3.5917	0.5020	0.0060	1.1952
10	0.5630	0.0220	3.9076	0.5490	0.0070	1.2750
30	0.5310	0.0170	3.2015	0.5450	0.0030	0.5505
30	0.5180	0.0190	3.6680	0.5480	0.0080	1.4599
30	0.5250	0.0200	3.8095	0.5050	0.0060	1.1881
60	0.5060	0.0170	3.3597	0.5290	0.0050	0.9452
60	0.5480	0.0160	2.9197	0.5480	0.0080	1.4599
60	0.5370	0.0210	3.9106	0.5400	0.0080	1.4815
120	0.5470	0.0170	3.1079	0.5480	0.0090	1.6423
120	0.5170	0.0170	3.2882	0.5120	0.0050	0.9766
120	0.5620	0.0200	3.5587	0.5160	0.0070	1.3566

Table D.2 Ash Content of Leached EFB and PKS

Temperature (°C)	EFB (g)	Ash (g)	Ash Content (%)
25	0.5100	0.0180	3.5294
25	0.5030	0.0190	3.7773
25	0.6050	0.0200	3.3058
35	0.5240	0.0220	4.1985
35	0.5240	0.0180	3.4351
35	0.5550	0.0200	3.6036
45	0.5220	0.0190	3.6398
45	0.5630	0.0180	3.1972
45	0.5470	0.0190	3.4735
55	0.5410	0.0190	3.5120
55	0.5870	0.0200	3.4072
55	0.5110	0.0200	3.9139

Table D.3 Ash Content of Leached EFB from 25 to 55 °C

Table D.4 Ash Content of Leached EFB and PKS at Different Stages

Stage	EFB (g)	Ash (g)	Ash Content (%)	PKS (g)	Ash (g)	Ash Content (%)
1	0.5100	0.0180	3.5294	0.5070	0.0070	1.3807
1	0.5030	0.0190	3.7773	0.5290	0.0040	0.7561
1	0.6050	0.0200	3.3058	0.5360	0.0070	1.3060
2	0.5160	0.0170	3.2946	0.5160	0.0060	1.1628
2	0.5370	0.0160	2.9795	0.5220	0.0060	1.1494
2	0.5090	0.0150	2.9470	0.5340	0.0070	1.3109
3	0.5140	0.0120	2.3346	0.5130	0.0060	1.1696
3	0.5360	0.0160	2.9851	0.5280	0.0060	1.1364
3	0.5060	0.0130	2.5692	0.5330	0.0070	1.3133
4	0.5160	0.0140	2.7132	0.5230	0.0070	1.3384
4	0.5680	0.0160	2.8169	0.5320	0.0060	1.1278
4	0.5520	0.0150	2.7174	0.5500	0.0060	1.0909

Appendix E: Mass Balance Flow Diagram of the Multiple Stage Leaching on EFB



Figure E.1 Cross Flow of 3 Stages EFB Leaching