EXPERIMENTS AND MODELING OF SIZE REDUCTION OF SWITCHGRASS IN A LABORATORY ROTARY KNIFE MILL

by

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Abstract

Biomass from forestry and agricultural sources has recently drawn a lot of attention as a new source of feedstock for energy and bio products. Size reduction is an important step in preparation of biomass as a feedstock. Each conversion process needs its own specific size or size distribution of particles. Modeling the size reduction process helps to optimize the design and control of the process while ensuring biomass particle sizes for an efficient biofuel conversion process. The objective of this study was to apply the population balance method for modeling the size reduction process. The model was applied to switchgrass size reduction by a grinder. Two population balance parameters, grinding rate (s⁻¹) and breakage distribution function (dimensionless) were estimated using experimental grinding data. The time dependent balance equations were solved using the Euler technique. The accumulation and depletion of the particles belonging to each size category were simulated as a function of time. The simulation predicted the residence time of particles inside the grinder in a way that the ground particles could meet the size and size distribution specifications for the downstream process. The thesis also describes preliminary steps in size reduction. Ground particles were fractionated based on their size by sieving. Weibull distribution was found to be the best probability density function to fit the data.

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Nomenclature

| $b_M(i,j)$ | mass-based breakage distribution function, weight fraction of material broken out |
|----------------------|---|
| | of size interval <i>j</i> which falls into interval <i>i</i> |
| b _n (i,j) | number-based breakage distribution function, weight fraction of material |
| | broken from size interval j which falls less than upper size interval i; |
| cdf | cumulative distribution function; |
| d | size of particles; |
| d_i | nominal sieve opening size opening; mm |
| \overline{d}_i | geometric mean diameter of particles on i^{th} sieve (= $(d_i * d_{i+1})^{1/2}$) |
| d_{gw} | geometric mean diameter of particles by mass; mm |
| е | size of particles; |
| Ε | specific energy requirement; kWh/t |
| F | diameter of the sieve in micron that %80 of feed passes through; |
| i | size interval; |
| j | size interval; |
| k | a constant; |
| K_R | Rettinger constant; |
| K_K | Kick's constant; |
| L | particle size; |
| L_F | feed particle size; |
| L _{f, 80%} | sieve opening size that 80% of feed particles pass through; |
| L_P | product particle size; |
| L _{р, 80%} | sieve opening size that 80% of product particles pass through; |
| т | a constant; |
| M(i,t) | mass of particles belongs to size interval <i>i</i> at time <i>t</i> ; |
| M(d,t) | mass of particles that have size <i>d</i> at time <i>t</i> ; |
| n | number of data in Equation 3.1; |
| п | a constant in Equation 1.1; |
| n(d,t) | number concentration of particles between size d and $d+dd$; |
| Р | diameter of the sieve in micron that 80% of product passes through; |

| pdf | probability density function; |
|----------------|--|
| S | standard deviation; |
| S | sample space; |
| S_{gw} | geometric standard deviation of particle diameter by mass; |
| S_{log} | geometric standard deviation of log normal distribution by mass; |
| $S_M(i)$ | mass-based grinding rate of material in size interval <i>i</i> or mass fraction of |
| | particles ground in time ;s ⁻¹ |
| $S_n(i)$ | number-based grinding rate of material in size interval <i>i</i> or number fraction of |
| | particles ground in time ;s ⁻¹ |
| t | time;s |
| x_i | i th measured variable; |
| W | energy input; kWh/t |
| W_i | work index in Equation 1.4; |
| W_i | mass of particles stays on i^{th} sieve in Equation 3.11; |
| W_{1i} | mass of particles before grinding; g |
| W_{2i} | mass of particles left on sieve <i>i</i> after grinding; g |
| W(i,j) | mass of particles originally from interval <i>i</i> that goes to interval <i>j</i> ; g |
| \overline{x} | mean of the data; |
| x_i | the i^{th} measured value; |
| Х | continuous random variable; |
| μ | location parameter; |
| σ | scale parameter; |
| λ | shape parameter; |
| ρ | particle density of the material; g/cm ³ |

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Chapter 1 Introduction

Biomass comes from a variety of sources such as forest, agriculture, industrial and municipal wastes. Size reduction (grinding) is one of the major pre-processing operations for using biomass as a source of energy or for producing pulp for paper industries. Grinders are among the largest power consuming machinery (Stocks et al., 1987), consuming 10-50 kW.t⁻¹ depending on the material and grinding mechanisms (shear, impact, attrition) (Spinelli et al., 2001). The design and choice of the grinder are important for reducing the energy input in preparing biomass. Table 1.1 shows that in a pelleting plant with a throughput of 3-5 tonne/h the grinder is the second largest power user (111.9 kW) after the pellet mill. The grinder is also the second most expensive piece of equipment (Smith, 2004).

| Table 1.1 List of equipment and power requirement for a 3-5 ton/hr pelleting plant (Smith, 2004) |
|--|
| pelleting plant (Smith, 2004) |

| Process or equipment | Power (kW) | Capital(\$) |
|------------------------------|------------|-------------|
| Raw material feeders | 7.5 | 32,000 |
| Transfer conveyor | 3.7 | 14,800 |
| Hammer mill and blower | 111.9 | 87,000 |
| Live bottom/mixing surge bin | 7.5 | 9,600 |
| Pellet mill | 186.5 | 125,000 |
| Conditioner | 5.6 | 10,000 |
| Counter-flow cooler | 1.5 | 18,000 |
| Air system cooler | 11.2 | 16,000 |
| Drag conveyor | 0.7 | 6,000 |
| Bucket elevator | 2.2 | 6,000 |
| Pellet screener | 1.5 | 14,000 |
| Bagger conveyor | 0.7 | 28,000 |

This chapter presents a critical literature review on the following topics:

- 1. The importance of biomass size and size reduction processes with respect to downstream conversion process
- 2. Characteristics of fibrous material with plant origin
- 3. Basic mechanisms of size reduction
- 4. Different ways of modeling the energy consumption of size reduction

1.1 The importance of size reduction

Processes such as gasification, pyrolysis, and hydrolysis/fermentation convert biomass to energy. None of these processes can use biomass in its original form. The first step in preparing biomass as a feedstock is size reduction. Size reduction is important because it is the main consumer of energy in the preparation process of biomass. Each downstream unit operation needs a specific average size of particles and particle size distribution. The specific particle sizes needed for some of the conversion processes are summarized below.

1.1.1 Combustion

Combustion is the simplest way of converting biomass to energy. Particle size has a major role for heat transfer during combustion (Jenkins, 1998). Combustion systems can be stoker type, suspension or fluidized beds.

1.1.1.1 Stocker pile burner

In stoker (grate type) or pile combustion the system accepts particles with a wide range of size and moisture content (up to 65%). The minimum size of a particle is dictated by the grate opening, and the maximum size is limited to the feed opening to the combustion chamber. If the particles are fed to the combustion chamber by an auger, the particles should be small enough to be handled by the auger (Badger, 2002).

Ryu et al. (2005) studied the effect of particle size on biomass combustion in a fixed bed. Pine wood was cut into particles with four different sizes of 5, 10, 20 and 35 mm. The bed height was 350, 375, 360 and 360 mm, respectively. The air flow rate was kept at 361 kg/(m^2h) in all cases. It was observed that smaller particles had higher burning rates, ignition front speeds and mass loss during ignition propagation. Large particles have a slow devolatilization rate. Larger particles had a lower ignition front speed and burning rate, while a larger amount of char was left above the ignition front.

1.1.1.2 Suspension burners

Suspension combustion systems can be either cyclonic burners or pneumatic spreaderstoker systems. Generally they can accept fuel particle sizes less than 6 mm and moisture content of less than 15% (Badger, 2002). For suspension burners, particles such as wood chip and pellets are ground before they feed to the combustion chamber.

Pulverized fuel burners usually require that the biomass fuel meet certain particle size specifications. In general, burners for biomass dust (e.g. wood powder) require particle size below 1000 μ m (Kastberg, 2002; Anderl, 1999) while the particle size used for coal in pulverized coal burner is usually below 100 μ m (Siegle, 1996). The small particle size of pulverized coal permits the complete combustion of coal in approximately half a second in the furnace. Biomass particles with sizes below 1000 μ m (Kastberg, 2002) have similar residence times as the coal, which is the reason for considering the finely ground biomass as a pulverized feedstock. Esteban et al. (2006) studied different strategies for the pulverization of forest biomass (poplar chips, pine chips, and crushed pine bark), and established the specifications for the comminuted product as having 95 wt% of particles passing a 1000 μ m mesh and 12 wt% passing a 125 μ m mesh. Another study suggested that the content of very fine particles (smaller than 100 μ m) should be higher than 10 wt% in order to achieve a short ignition time (Esteban et al., 2006).

1.1.1.3 Fluidized bed combustors

In fluidized bed combustion systems the particles are fluidized in a bed of an inert (noncombustible) material like sand (La Nauze, 1987). Fuel particle size is important, because very small particles entrained from the system cannot be caught by the cyclone to recycle (Badger, 2002). Big fuel particles cannot be fluidized and efficiently burned in the system. The particle size is also controlled by the opening of the feeding system. La Nauze (1987) reported a maximum feed biomass size of 50 mm and the range of bed particle size of 0.1-4.0 mm for bubbling fluidized bed combustors. For circulating beds the maximum feed size is 10 mm with the range of bed particle size of 0.05-1.0 mm.

1.1.2 Gasification

Gasification is the partial combustion of biomass in a restricted supply of air or oxygen to produce a combustive gas mixture, which consists of carbon monoxide, hydrogen and methane. Gasifiers can be fixed beds or fluidized beds. A wide range of biomass fuels such as wood, charcoal, wood waste (branches, roots, bark, and sawdust) as well as agricultural residues- maize cobs, coconut shells, cereal straws, rice husks, can be used for gasification. Theoretically, almost all kinds of biomass with moisture content of 5-30% can be gasified. However, not every biomass can be gasified successfully. A moisture content below 15 wt% is desirable for trouble-free and economical operation of the gasifier (Chandrakant, 2006).

The fuel size affects the pressure drop across the gasifier and the power that must be supplied to push the air and gas through the gasifier. Large pressure drops lead to the reduction of the gas load in downdraft gasifiers, resulting in a low reactor temperature and high tar formation. Excessively large sizes of particles give rise to reduced reactivity of fuel, causing start-up problems and poor syngas quality.

An acceptable fuel particle size depends on the design of the gasifier. In general, wood gasifiers work well on wood chips ranging from $80 \times 40 \times 40$ mm down to $10 \times 5 \times 5$ mm.

Fixed bed gasifiers need uniform particles, an example of the accepted particles are 25 x 25 x 6 mm wood chips (Badger, 2002). Particles moisture content should be less than 20% (Badger, 2002). Fluidized bed gasifiers have the advantage of accepting particles with wide range of size distributions. Fluidized gasifiers also need an inert bed material for fluidization of biomass particles. Van Der Drift (2001) studied the gasification process of 10 biomass residual fuels, chips and granulates with maximum sizes of 40 mm, with sand of 0.4-0.6 mm as the bed material.

According to Cummer (2002), the two most common devices for comminuting biomass to sizes appropriate for gasification are knife chippers (speeds up to 1800 rpm) and hammer mills.

1.1.3 Densification

The other important use of ground biomass is for the production of densified fuels such as pellets, cubes or briquettes. Agricultural biomass is harvested by the forage harvester. The length of full or chopped stems is between 25 to 75 mm. For pellet and briquette production the harvested material should be first chopped by grinding. If the chop is compacted into cubes, it usually requires no further size reduction (Samson et al., 2005). For briquette production the particles should be 6-8 mm.

Mani et al. (2004) studied the effect of particle size on time required for rearrangement of particles during compaction for four biomass species in a single pelleting unit. Their results showed that particles rearrangement was shorter when the particle sizes of the grinds were smaller. The specification and the particle size needed for the three mentioned densified forms of biomass are summarized in Table 1.2

Mani et al. (2003) also studied the effects of particle size on mechanical properties of biomass pellets made from wheat straw, barley straw, corn stover and switchgrass. They installed screens with 3.2, 1.6 and 0.8 mm openings respectively, in the hammer mill. They reported that corn stover pellets made from the ground material of 1.6 mm screen size were 5 to 16% denser than pellets from the ground material obtained from screen size of 3.2 mm.

| | Particle size needed(mm) | Overall shape | Dimension(mm) | Bulk density (kg.m ⁻³) | Reference |
|------------|--------------------------|------------------|--|---------------------------------------|----------------------|
| Pellets | <3.2 | Cylindrical | 4.8-19.1 Diameter | 620-720 | Mani et al. (2003) |
| | | 2 | 12.7-25.4 Length | | |
| Cubes | 25-75 | Square cross-sec | 16-32 mm square cross section 32-64 mm Length | 450-600 | Samson et al.(2005) |
| Briquettes | 6-8 | Cylindrical | 50-100 Diameter 200-300 Length | 1000-1500 | Samson et al. (2005) |

Table 1.2 The particle size needed for the production of three forms of densified biomass

Yu et al. (2003) reported that the particles processed by pellet mills are generally restricted to 80% of the die-opening diameter or less. A cuber by contrast can more easily process particles of 75% smaller than 32 mm.

Kaliyan et al. (2006) studied the effects of particles size (0.56 to 0.8 mm) on the densification (briquette production) characteristics of corn stover and switchgrass. They concluded that decreasing the geometric mean particle size of ground corn stover from 0.8 to 0.66 mm increase the density of briquetts by 5 to 10%. They also reported that decreasing the particle size of corn stover grind from 0.8 to 0.66 mm increase the durability of briquettes by 50 to 58% at 100 MPa pressure, and by 62 to 75% at 150 MPa at a moisture content of 10%. They also reported that decreasing the particle size of corn stover grind from 0.8 to 0.66 mm increase the specific energy consumption by 0.8 to 1.3 MJ/t. For switchgrass briquetting, reducing particle size from 0.64 to 0.56 mm decreased the specific energy consumption by 2.5 to 4.3 MJ/t.

1.1.4 Ethanol production

Ethanol production is another end use of biomass. According to US Patent No. 5677154 this process needs a size of 1-6 mm of ground biomass (Van Draanen, 1997). Hydrolysis of cellulose in lignocellulosic materials to fermentable sugar is the first step of bioethanol production. Cellulose is always surrounded with lignin and hemicellulose. This makes the access to cellulose difficult.

Grinding is an important step to prepare biomass for hydrolysis. The size of particles is usually 10-30 mm after chipping and 0.2-2 mm after milling or grinding (Sun et al. 2001). Vibratory ball milling has been found to be more effective in breaking down the cellulose crystals of spruce and aspen chips and improving the digestibility of the biomass than ordinary ball milling (Sun et al. 2001).

1.1.5 Pyrolysis

Pyrolysis is the thermal cracking of biomass in the absence of oxygen or air supply (Sims, 2000). Products of pyrolysis are solid, liquid and gas. Careful control of heating rate, temperature and inert gas supply can promote the production of one of the products. A slow pyrolysis produces bio-char. The reaction condition is slow heating, low to intermediate temperatures and long residence time.

Fast pyrolysis is a high temperature process in which biomass is rapidly heated in the absence of oxygen (Bridgwater et al., 1999). The product of fast pyrolysis can be a liquid fuel that can be a substitute for fuel oil. Liquid production requires very low vapor residence time to minimize secondary reactions. Usually this time is less than 1 s. Bridgwater et al. (1999) suggested that maximum liquid yields are reached with high heating rates at a reaction temperature of around 500 °C. In circulating fluidized beds, sand is used to provide the majority of heat transfer. The sand particle size should not be more than 3 mm. If the particles are more than 2 mm in size, the char should be removed to avoid slow pyrolysis (Bridgwater et al., 1999). Bridgwater (1999) reported that for fluidized bed reactors the particle size should be less than 2 mm, for circulating fluidized bed the particles should be less than 6 mm and for ablative reactors particles can be even more than 10 mm.

Zanzi et al. (1996) studied the influence of particle size on pyrolysis. Fast high temperature pyrolysis was performed in a free fall reactor with a maximum operating pressure of 5 MPa and 1100 °C. The material that was tested was wood (two types: birch and white quebracho). In rapid pyrolysis the char yield is higher when smaller particles are used. An increase of particle diameter from 0.5-0.7 mm to 0.7-1.00 mm for birch at 800 °C increased the solid residue from 4.6 to 5.5 wt% after total pyrolysis; in other words an approximately 20% increase in char production. Under the experimental conditions studied, the composition of pyrolysis gas, gas yield and tar yield were not affected by a change in particle size. In slow pyrolysis for birch and white quebracho with temperatures ranging from 800 to 1000 °C, the char yield increased almost 20%.

1.1.6 Pulp and paper

For pulping in pulp and paper industries, as an end use process for ground wood, the size and uniformity of chips are important quality characteristics. These characteristics can be achieved through proper design and operation of a grinder. Chippers with sharp knives are used for size reduction. The chips produced have relatively regular shapes and limited size variation. They must be clean and free of any contaminant. The chips size can be controlled by controlling length of the time in the chipper, maintaining chipping quality and using a series of screening processes (Goulding, 1988). The length of these chips varies from 5 to 30 mm. The thickness of chips is very critical in Kraft pulping and it ranges from 1.5 to 4 mm. It has less importance in sulphite pulping (Smook, 1992). According to Smook (1992) the ideal chip is usually considered to be about 20 mm long in the grain direction and 4-5 mm thick, with all chips 10-30 mm long and 3-6 mm thick being considered as prime materials for pulping. In sulphite pulping the most important chip parameters are chip length and chip damage. In refiner-mechanical pulping, an undisturbed constant flow of chips into the refiner is important. Therefore chip dimensions must be kept constant (Hartler et al., 1979).

In the Kraft cooking process, chip thickness is of primary concern because diffusion is the predominant means for chemicals being transported into the chips. The rate of diffusion is approximately the same in the three main directions of the chips. But most of the chemicals are transported in the direction where the distance is the shortest, i.e. along the thickness direction. The lower critical limit of particle size is 2 mm. Wood chips with small thickness have a very low mechanical stability (Hartler et al., 1979). All Kraft mills have installed equipment that classifies chips by thickness. Modern disc type or roll type screens which segregate according to thickness are now widely installed in Kraft pulping systems (Smook, 1992). Modern chip slicers that reduce chip thickness as small as do not damage the fibres are used as the re-chipper. Most sulphite mills still do use chip classification using round-hole screens.

The best chipping of softwood logs leads to 85% acceptable chips, 4% over thick, and 2% over length chips, 7% pin chips, and 2% fines. The definitions of the different fractions are as follow. Overs are the oversized or overthick fraction of chips, and are retained on 45 mm diameter hole screen so they are thicker than 10 mm for softwood or 8 mm for hardwoods. Accepts are the chip fraction of ideal size distribution for pulping. These chips pass through an 8 or 10 mm screen and are retained on a screen with 7 mm holes. Pin chips are the chips that pass though a 7 mm screen, but are retained on a 3 mm hole screen. Fines (unders) are the undersized fraction of chips or sawdust, and are collected in the bottom pan. Fines generally consist of material passing through a 3 mm screen (Biermann, 1996). Table 1.3 lists the size of the particles tested in different studies for downstream conversion processes.

| Conversion process | Particle range and spec. | Biomass type | Reference |
|--|---|--|---|
| Combustors: | | | |
| Stoker or pile burner | 5, 10, 20, and 35 mm | Pine wood | Ryu et al., 2005; |
| Suspension burner | Less than 6 mm | | Badger, 2002; |
| Pulverized fuel burner Pulverized fuel burner | Below 1000 μm 95% passing 1000 μm mesh and 12% passing | Wood powder | Kastberg, 2002; Anderl, 1999; Esteban et al., 2006; |
| Bubbling fluidized bed Combustor | 125 μm mesh Max. 50 mm with bed particles of 0.1-0.4 mm | | La Nauze, 1987; |
| Bed | particles of 0.05-1.0 mm | | La Nauze, 1987; |
| Gasification | | | |
| Fixed bed | Min. 10 x 10 x 10 mm Max. 80 x 40 x 40 mm | Wood chips | Chandrakant, 2006; |
| Fixed bed | Accepted particles: 25 x 25 x 6 mm | Wood chips | Badger, 2002; |
| Fluidized bed | Max. 40 mm with bed particles of 0.4-0.6 mm | 10 biomass residual fuels, chips and granulated | Van Der Drift, 2001; |
| Pyrolysis | | 8 | |
| Fluidized bed | <2 mm with bed particles of <3 mm | 3 | Bridgwater et al., 1999; |
| Circulating fluidized Bed | <6 mm | | Bridgwater et al., 1999; |
| Ablative reactors | Can be more than 10 mm | | Bridgwater et al., 1999; |
| Free fall reactor | 0.5-1 mm | Birch and White quebracho | Zanzi et al., 1996; |
| Densification | | | |
| Pellets | <3.2 mm | Wheat straw, barley straw, corn stover, and switchgrass | Mani et al., 2003; |
| Cubes | 25-75 mm | 0 | Samson et al., 2005; |
| Briquetts | 6-8 mm | | Samson et al., 2005; |
| Ethanol production | | | |
| Ethanol production | 1-6 mm | | Van Draanen, 1997; |
| Hydrolysis | 0.2-2 mm | | Sun et al., 2001; |

Table 1.3 A summary of biomass particles size tested for different downstream conversion processes

| Conversion process | Particle range and spec. | Biomass type | Reference |
|--------------------|------------------------------------|--------------|-----------------------|
| Pulp and paper | | | |
| Kraft pulping | Chip thickness: 1,5-4 mm | Wood | Smook, 1992; |
| Kraft pulping | >2 mm | Wood | Hartler et al., 1979; |
| Sulphite pulping | Length: 20 mm Thickness: 4-5 mm | Wood | Smook, 1992; |

1.2 Characteristics of fibrous particles from plants

Biomass is defined as all plant material, including trees and grasses or the by-products from their harvesting or processing. Chemically, a biomass is composed of three main components; cellulose, hemicellulose and lignin. The physical structure and strength of biomass plays the main role in size reduction of biomass. For shearing, the cross section of a stem is made of four types of components: fibres, skin, soft cells and cavities. Plants derive their strength mainly from fibre cells which are bundles or layers of long cells with relatively small cross sections and thick wall. A fibre bundle may have a cross section diameter of 0.1-0.2 mm (Persson, 1987). Fibre cells are long, in maximum length of 500 mm; however, the fibre cells in straw and forage grasses reach a maximum length of only 30 mm (Persson, 1987).

The cell wall of a fibre consists of three layers: the middle lamella, the primary wall, and the secondary wall. The middle lamella is the main point of breakage of cell wall. The process of cutting cell walls originates in the middle lamella. The secondary wall determines the strength and flexibility of the plant. Three layers of structures are found within the secondary wall, the main structure being cellulose. Cellulose chains have a parallel organisation, and are strongly bound together in relatively long micro fibrils. The micro fibrils have a spiral structure, the spiral angle to the cell axis being 70 degrees or more. As a result the layers of secondary wall stretch easily giving cell walls their elasticity.

Besides cellulose, chemical compounds like lignin and non-cellulose polysaccharides appear in the cell wall. Lignin has lower elasticity than cellulose but higher compressive strength. The solid material in plants offers the main resistance to cutting. For many crops all the solid material is not structurally solid. Materials such as starch and sugar do not contribute to cutting resistance. So when it is needed to estimate the structural solid, it is important to know about this non-structural solid. The concentration of these non structural substances in a plant can reaches 50% of the total amount of solids.

In size reduction of biomass, different parts of a plant behave differently in shear or impact. For example for forage, the shear of leaves is usually more difficult than the shear of stems, because the leaves bend instead of being cut. Stem structure is also varies in different directions. Grass stems have nodes and internodes. Internodes are longer and weaker than nodes. Nodes are solid but elastic. Internodes have a tubular cross section. Figures 1.1 and 1.2 show the percentage of various parts of cornstover and wheat straw (Pordesimo et al., 2004; Kenney et al., 2006). Each of these parts of plant has different structure, composition, and moisture content (Table 1.4). These differences make the size reduction process a challenging one.

Trees have a wide variety of cells in comparison with other plant species. Tree trunks consist of: dead bark, living bark, sapwood, heartwood, and pith. Sapwood is serving food and water to the tree. So it has higher moisture content than heartwood. Heartwood is more brittle and it has less ability of deformation before failure. Softwood consists up to 90% (by volume) of longitudinal cells.



Figure 1.1 Distribution of various parts of corn stover (excluding grain). (Pordesiomo et al., 2004)



Figure 1.2 Distribution of various parts of straw (excluding grain) (Kenney et al., 2006).

| Source | Average fiber length (mm) | Cellulose | Lignin (%) | Hemi- cellulose (%) | Ash (%) | Silica |
|-----------------|------------------------------|-------------|---------------|------------------------|------------|---------|
| Wheat straw | 1.5 | 50 - 52 | 16 - 20 | 26 - 30 | 5 - 10 | 4 - 8 |
| Oil flax (Bast) | 30 | 47 | 23 | 25 | 5 | - |
| Barley straw | - | 47 - 48 | 14 – 15 | 24 - 29 | 5-7 | 3 – 6 |
| Oat straw | - | 44 - 53 | 16 – 19 | 27 - 38 | 6-8 | 4 - 6.5 |
| Rye straw | - | 50 - 54 | 16 – 19 | 27 - 30 | 2 - 5 | 0.5 - 4 |
| Bagasse | 1.7 | 53 - 56 | 19 – 24 | 27 - 32 | 2 - 5 | 2 - 4 |
| Rice straw | 0.5 - 1.0 | 42 - 46 | 12 – 15 | 24 - 30 | 15 - 20 | 10 - 18 |
| Hemp bast | 25 | 61 | 10 | 23 | 2 | - |
| Hemp core | 0.8 | 34 | 21 | 38 | 1 | - |
| Jute | | 72 | 13 | 13 | | |
| Sisal | | 73 | 13 | 11 | | |
| Cotton | | 92 | 6 | | | |
| Ramie | | 76 | 15 | 1 | | |
| Wood | | 45 | 23 | 27 | | |
| Coir | | 43 | <1 | 45 | | |
| Courses Atolia | 1007. Maha | and Davi 10 | 00. Dahaar | 1095 | | |

Table 1.4 Fiber length and chemical properties of fibrous crops

Source: Atchison, 1997; Mabee and Roy, 1999; Robson, 1985.

Hardwood consists of 25%-50% of fibres that are elongated cells. Along the length these fibres the cross section length varies from 0.8 to 2.5 mm. From a cutting stand point important physical properties of the cellular material are: (1) strength in compression, tension, bending and shear, (2) high density. For plant material these properties are influenced by type and age of plant, moisture content, and cellular structure. The values for these strength properties are different in different directions in the plant.

For grasses the fibre content increases during the growing period. In the middle of the growing season grasses achieve their maximum lignin content. The strength of grass leaves, dependent on the percentage of the cellulose, increases with age. Cutting energy increases with plant age and maturity (Persson, 1987).

1.2.1 Moisture content

The moisture content of biomass from agricultural crops and forestry may range from a high of 80% (wet mass basis) down to less than 5%. The moisture content depends on the crop species, maturity, plant part, and the weathering time. Crop residues from smaller grains such as wheat and barley have lower moisture content than biomass from larger grains, such as corn. Grasses, depending on the stage of maturity, can be wet at the time of harvest. The moisture content influences the grinding characteristics of plant material significantly.

Tensile and shear properties of the biomass can also influence the energy requirements for biomass size reduction. Size reduction equipment operates more efficiently by applying shear stress than by applying tensile stress, because the shear mechanism may be considered as the weakest mode of failure. Size reduction studies show that mean shear strength is approximately one-fifth of the tensile strength (Womac, 2005).

According to Halyk and Hurlbut (1968) the ultimate tensile and shear stresses of alfalfa is inversely proportional to its moisture content. Greenberg et al. (1989) reported that both ultimate stresses decreased with increasing moisture content for ryegrass. Annoussamy et al. (2000) observed that shear stress increased as moisture content decreased for wheat straw. Ige et al. (1976) provided a similar result for corn stalk and alfalfa. They concluded that increased moisture content reduced shearing energy requirement.

Igathinathane et al. (2007) showed that a mat of moist switchgrass at 51% moisture content offered more resistance to shear than a mat of dry material at 20% moisture content. Kushwaha et al. (1983) reported a range of shear strength from 7.0 to 22 MPa for stem moisture content ranging from 5 to 30% wet basis in case of wheat straw. Minimum values of shear strength (7 to 10 MPa) occurred for stem moisture contents between 8% and 10% wet basis. Mani et al. (2004) reported a positive correlation of moisture content with specific energy consumption of wheat and barley straws, corn stover and switchgrass; the higher the moisture content the higher the specific energy consumption. Balk (1964) found a similar pattern of results for alfalfa grinding.

O'Dogherty et al. (1995) also found reduced shear stresses (mean value of 5-30 MPa) for wheat straw at moisture contents of 8 and 10% wet basis, and hypothesized that dry brittle straw was weaker than moist tough straw. The results for tensile strength showed

no consistent trends with varying moisture content. The modulus of rigidity decreased with increasing moisture content over a range of 499 to 389 MPa.

1.3 Size reduction processes

Size reduction consists of breaking or cutting a solid biomass to smaller pieces. Cutting mostly involves shearing action, whereas breaking involves some degree of impact and attrition (friction). Size reduction processes can be achieved by successive compaction and tension, as in a roller mill. The basic mechanisms of size reduction, shear, impact and attrition are reviewed here.

Table 1.5 (Anynom, 1992) lists throughput, rated power and the capital cost for four systems involving a hammer mill and roller mills. On a unit basis, hammer mills use more power than roller mills and have a higher capital cost than roller mills. The overall power requirement and cost of the two systems decrease as the throughput and size of both mills increase.

| System | Equipment | Qty | Thruput (t/hr) | Power (HP) | Capital (\$) | HP/t | \$/t |
|----------|-------------|-----|-------------------|---------------|-----------------|------|-------|
| System 1 | Hammer mill | 1 | 13 | 100 | 66,564 | 7.7 | 5,120 |
| | Roller mill | 1 | 13 | 50 | 38,698 | 3.8 | 2,977 |
| System 1 | Hammer mill | 1 | 20 | 150 | 74,876 | 7.5 | 3,744 |
| | Roller mill | 1 | 20 | 75 | 89,982 | 3.8 | 4,499 |
| | Hammer mill | 2 | 95 | 400 | 300,802 | 4.2 | 3,166 |
| System 1 | Roller mill | 2 | 95 | 200 | 296,566 | 2.1 | 3,122 |
| System 1 | Hammer mill | 4 | 200 | 350 | 549,704 | 1.8 | 2,749 |
| | Roller mill | 4 | 200 | 200 | 586,764 | 1.0 | 2,934 |

Table 1.5 Comparison of two grinders reducing corn grain to 600-700 microns

Systems include the mill, conveyors, dust control (for the hammer mill), electrical installations and controls.Source: Anynom (1992)

1.3.1 Shear

Shear is one of the mechanisms of size reduction. Shear is exerted by cutting biomass using a sharp blade. When the cutting device is a knife, the knife's geometry and the direction of the cut in relation to the material being cut affects the configuration of the resulting ground material, the cutting power needed, and the quality of the chips' surface (Hakkila, 1989).

Womac et al. (2005) studied the cutting response characteristics of single stems of corn stover, hickory wood, and switchgrass. Biomass shear strength was calculated based on peak load and actual cross-sectional area. Mean shear strengths due to cutting of corn stover, hickory, and switchgrass at 30° and 45° knife bevel angles were 1.8, 16.8, and 12.0 MPa, and 2.1, 24.9, and 12.5 MPa, respectively. Specific cutting energy was calculated based on integrating the force displacement curve through the cutting distance. Mean specific cutting energies for corn stover, hickory, switchgrass (10-15 wb% moisture content) were 28, 122, 78 kN/m, and 34, 160, and 95 kN/m for 30° and 45° knife bevel angles, respectively. They concluded that, although load displacement curves may not lead to development of biomass shear models, their study shows that hickory has shear strength of 10 times that of corn stover. Womac et al. (2005) also mentioned that although the experimental shear rate was much lower than actually occurring shear within a grinder, but their study exhibit the characteristics of real shear.

Most biomass size reduction machinery operates by rotary action. Igathinathane et al. (2006) developed a device based on linear shear action. They built a prototype of a linear action knife grid model device. The device consisted of a ram, feed block, knife grid, knife holder block, product block, and bottom tray. They tested the device with dry corn stalks and switchgrass. They studied the maximum failure load, ultimate cutting stress, energy involved in cutting corn stalks and switchgrass, and the effect of knife grid spacing. Their results show that before cutting both materials, corn stalks and switchgrass went through a large amount of deformation and compression. Ultimate cutting stress and net energy needed increase with an increase in fill depth and decrease in knife grid spacing. Corn stalks needed three to four times more specific energy for cutting than switchgrass. The specific energy is calculated based on mass and surface area generated.

Igathinathane (2007) studied size reduction characteristics of corn stalks and switchgrass using the knife grid introduced in their previous study. They investigated the effect of high and low moisture contents (high and low), knife grid spacing (25.4, 50.8, and 101.6 mm) and packed bed depth (50.8, 101.6, and 152.4 mm). The goal of the study was to

determine and compare the ultimate cutting stresses and cutting energy variation between corm stalks and switchgrass. They also evaluated new surface area generated during size reduction. Overall results from this study indicate that for corn stalks the ultimate cutting stress and cutting energy required are significantly greater (2.2 times) than switchgrass. High moisture material requires significantly greater stress and energy (1.3 times) than low moisture material. Grid spacing produces a significant difference in cutting energy but not in ultimate cutting stress. Energy values required in size reduction using linear knife grid device were much smaller than that reported for reducing similar biomass using other methods of size reduction. Therefore, a pre-processing machine, based on the linear knife grid principle, with 50 to 100 mm and greater grid spacing would be an efficient first stage size reduction process for biomass materials.

1.3.2 Impact

One of the bases of size reduction is shredding using blunt impacting tools a process by which particles are damaged by compression and impact. Hammer mills are grinders that work based on impact. Hammer mills consist of rotating shafts with fixed or swing hammers are attached to them. Hammer mills using fixed hammers can have a higher kinetic energy to break down the biomass, but their maintenance is more difficult. Swing hammers can accept more contaminated feed, and are easier to maintain. In hammer mills, the size of a grind is controlled by built-in screens (Hakkila, 1989).

Yu et al. (2003) reviewed the biomass size reduction using hammer mills. They reported that using hammer mill had the advantage of low maintenance over other methods, including crushing, shearing and using roller mills. Tub grinders are small mobile hammer mills often designed as pull-behind units for agricultural use or mounted on tractor-trailers for larger waste removal use.

1.3.3 Attrition

Attrition is used primarily for grinding of tough organic materials such as wood pulp and corn grits (Perry, 1997). Size reduction takes place between two smooth or abrasive plates, which may be aligned either horizontally or vertically. Product size is controlled by changing the distance between two plates.

Stone grinders have been used for pulp making in pulp and paper industries (Liimatainen, 1999). Essentially, pulp is formed when the logs are pressed against a rotating, properly sharpened, pulp stone. Ceramic segments are attached to a steel-reinforced concrete core. Attrition between the logs and these ceramic segments cause the grinding of wood and the production of the fiber particles. Based on their feeding system they may be chain grinder, pocket grinder, pressure grinder or thermo grinder (Liimatainen, 1999). The ceramic stones are used as pulp stone. One of the main problems associated with attrition grinders is excessive heat generation that may damage the biomass.

1.4 Power consumption for size reduction

Cadoche et al. (1989) compared the energy consumption (kWh/t) of size reduction of hardwood, straw, and corn stover using a knife mill and a hammer mill. Their results showed that for hardwood, when the final size was changed from 6.35 mm to 1.60 mm the energy consumption of the knife mill increased from 25 to 130 kWh/ton. When a hammer mill was used for the same size change the energy consumption increased from 95 to 130 kWh/t. For straw, when the final particle size decreased from 2.54 to 1.6 mm the energy consumption of the knife mill increased from 6.4 to 7.5 kWh/t. In comparison, when a hammer mill was used the energy consumption changed from 29 to 42 kWh/t. They didn't mention the original size of particles.

Esteban et al. (2006) evaluated four different strategies for pulverization of three forest biomass: poplar chips, pine chips and pine bark. They designed four types of open circuit processes. Two hammer mills, one screener and one dynamic air separator were the main apparatuses they used. The arithmetic mean size of the raw material particles were measured based on ASTM E-821-81. The arithmetic mean size of particles was 9.52 mm for poplar chips, 12.2 mm for pine chips and 11.63 mm for pine bark. They introduced a reduction ratio which is the ratio of arithmetic mean of the feed particle size to arithmetic mean of product particle size. The average of this ratio obtained for pine chips was 29.91, for poplar chips 22.5, and for pine bark 29.52. The average energy requirement (considering all apparatuses) was 118.5, 85.4, and 19.7 kWh d.t⁻¹ for pine chips, poplar chips and pine bark respectively. Their results show the lowest energy consumption for pine bark.

Table 1.6 lists a summary of measured power consumption for reducing biomass size using variety types of size reduction devices.

| Crop | kWh/t | Particle size or | Type of grinde | r Reference |
|-------------------|-------|------------------|----------------|-----------------------------|
| | | screen size, mm | | |
| Wheat straw bales | 749.0 | 12.7 | Tub grinder | Arthur et al., 1982 |
| Wheat straw bales | 328.0 | 50.8 | Tub grinder | Arthur et al., 1982 |
| Switchgrass | 2.8 | 50 | Linear knife | Igathinathane et al., 2008 |
| Switchgrass | 2.8 | 100 | Linear knife | Igathinathane et al., 2008 |
| Switchgrass | 8.5 | 100.0 | Hammermill | Schell and Harwood, 1994 |
| Switchgrass | 8.5 | 200.0 | Hammermill | Schell and Harwood, 1995 |
| Switchgrass | 44.9 | 5.6 | Hammermill | Samson et al. (2000) |
| Switchgrass | 55.9 | 5.6 | Hammermill | Jannasch et al. (2001) |
| Switchgrass | 55.9 | 2.8 | Hammermill | Jannasch et al. (2001) |
| Switchgrass | 27.6 | 3.2 | Hammermill | Mani et al. (2002, 2004) |
| Switchgrass | 4.2 | 12.7 | Knife mill | Bitra et al. (2008) |
| Switchgrass | 27.6 | 3.2 | Hammermill | Mani et al. (2002, 2004) |
| Wheat straw chops | 5.1 | 12.7 | Knife mill | Bitra et al. (2008) |
| Stover | 4.4 | 12.7 | Knife mill | Bitra et al. (2008) |
| Stover | 11.0 | 3.2 | Hammermill | Mani et al. (2002, 2004) |
| Poplar chips | 85.4 | 1.5 | Hammermill | Esteban and Carrasco (2006) |
| Pine chips | 118.6 | 1.5 | Hammermill | Esteban and Carrasco (2006) |
| Pine bark | 19.7 | 1.5 | Hammermill | Esteban and Carrasco (2006) |
| Hardwood chips | 20.0 | 0.6 | | Datta (1981) |
| Hardwood chips | 21.0 | 0.6 | | Datta (1981) |
| Hardwood chips | 100.0 | 0.3 | | Datta (1981) |
| Hardwood chips | 200.0 | 0.2 | | Datta (1981) |
| Hardwood chips | 130.0 | 1.6 | Hammermill | Cadoche and López (1989) |
| Hardwood chips | 115.0 | 3.2 | Hammer mill | Cadoche and López (1989) |
| Hardwood chips | 50.0 | 3.2 | Knife mill | Cadoche and López (1989) |

Table 1.6 Summary of measured power consumption for reducing biomass size using variety types of size reduction devices

1.5 Modeling of energy and power consumption in size reduction

The earliest studies of size reduction go back to the 1930s (Perry, 1997), beginning with fundamental studies on single particle fracture. Different theories quantify the energy consumption during the process of grinding. Each theory characterises size reduction in a different way. Earlier Walker et al. (1937) and later Earle and Earle (1983) suggested Equation 1.1 as general form for these theories.

$$dE = -K\frac{dL}{L^n}$$
 1.1

In Equation 1.1, dE is the differential energy required, L is the particle size, K and n are constants. The value of n depends upon three theories on particle breakage: the Rettinger, the Kick and the Bond. Each theory is unique and its application to a particle size reduction problem results in a unique solution.

1.5.1 Rettinge Theory

The Rettinger theory was introduced in 1867 (Bond, 1961). It hypothesizes that the work done for grinding and crushing is directly proportional to the new surface area produced. Based on this theory the surface area of the feed and product has to be calculated. The theory assumes that the energy input is completely transferred to the surface area of the ground particle. So, in the general form of Equation 1.1, n is equal to 2. Integration of Equation 1.1 gives:

$$\Delta E = K_R \left(\frac{1}{L_p} - \frac{1}{L_f}\right) \tag{1.2}$$

In Equation 1.2, L_p and L_f are product particles size, and feed particles size, respectively. K_R is the Rettinger constant. Computations using the best values of surface energy obtainable indicate that probably 99% of the work input is wasted (Bond, 1952; Earle, 1983) (parasitic power). On the other hand many tests performed showed that this law is too simplified. In fact all the input energy to the grinder is not transferred to the material. The fraction of the energy transferred to the material varies for different types of machine and operating conditions (Austin et al., 1964).

1.5.2 Kick's Theory

Kick's theory was introduced in 1885 (Bond, 1961). Kick assumed that the energy required to reduce particles of the initial size L_f , a size change of dL is directly proportional to the size reduction ratio dL/L. This means that *n* equal to 1.

$$\Delta E = K_K \ln \frac{L_f}{L_p}$$
 1.3

 K_K is the Kick's Constant, L_p and L_f are product particles size and feed particles size, respectively. The Kick theory is based primarily upon the stress-strain diagram of cubes under compression, or deformation factor (Bond, 1952).

1.5.3 Bond Law

Bond (1952) introduced a third theory: the work input is proportional to the new crack tip length produced in particle (cracks first appears on the surface then penetrates in the volume), and is equal to the work represented by the product minus that represented by the feed (Bond, 1961).

For practical calculations the sieve size in microns which 80% passes is selected as the criterion of particles size (Bond, 1961). Equation 1.4 shows the Bond theory.

$$W = \frac{10W_i}{\sqrt{L_{p,80\%}}} - \frac{10Wi}{\sqrt{L_{f,80\%}}}$$
 1.4

In Equation 1.4 the diameter of the sieve in microns that 80% of the product passes through is $L_{p,80\%}$ and the diameter of the sieve in micron that 80% of the feed passes through is $L_{f,80\%}$. The work input in kilowatt hours per short ton is W. W_i is the work index. The work index is a parameter that shows the resistance of the material to grinding. It is defined as the kWh per short ton required reducing the material from theoretically infinite feed size to 80% passing 100 microns (Bond, 1961). If the material is homogeneous for size reduction its work index will remain constant through all size reduction stages.

1.5.4 Empirical relations

Mani et al. (2004) studied the grinding performance of four types of agricultural biomass. They were corn stover, switchgrass, wheat straw and barley straw. Corn stover and switchgrass were manually cut to 25-50 mm particles, and then fed into the grinder. Materials were conditioned to two moisture contents of 8% and 12% wet basis. They used a hammer mill with three screens installed. Screen sizes were 3.2, 1.6 and 0.8 mm. They monitored the energy consumption of the grinder and correlate the specific energy consumption of grinder (kWh t⁻¹) as a function of hammer mill screen size in millimeters. The results of their study are summarized in Table 1.7. The first column specifies the biomass tested. The second column shows the correlated equation for the specific energy consumption based on screen size for the four biomasses at 8% moisture content. The correlated equation is linear with the coefficient of determination higher than 0.96. The third column shows the correlated equation for the specific energy consumption based on screen size for the four biomasses at 12% moisture content. In this case the correlated equation is a second order regression model. The coefficient of determination for this case is more than 0.96. It shows that with both moisture contents, the smaller the screen size the higher the specific energy needed for grinding. Corn stover consumes less energy at both moisture contents because of its lower fiber content and because of the presence of larger proportion of spongy vascular tissue in its stem (Mani et al., 2004). Among the four biomasses switchgrass consumed the highest energy to grind in all screen sizes.

| Biomass | Moisture content (wb) | | | | |
|--------------|--|---|--|--|--|
| | 8% | 12% | | | |
| Switchgrass | $E=-16.45S+76.52 (R^2=0.99)^1$ | $E=-9.16S^2+24.22S+43.12$ ($R^2=0.99$) | | | |
| Barley straw | E=-16.30S+65.08 (R ² =0.97) | | | | |
| Wheat straw | E=-16.78S+64.38 (R ² =0.96) | $E=-4.07S^2+7.48S+41.95$ ($R^2=0.98$) | | | |
| Corn stover | E=-6.14S+25.99 (R ² =0.97) | E=5.31S ² -30.86S+55.45 (R ² =0.96) | | | |

Table 1.7 Specific energy requirement for grinding of selected biomass at 12% and
8% (wb) moisture content (Mani et al., 2004)

E: the specific energy requirement (kW h t^{-1}); S: hammer mill screen size (mm)

1.6 Objectives

Review of literature shows that a firm principle for grinding science is not yet well developed. This is mainly due to variations in physical properties and chemical constituents of various biomass. The form and shape of final particles for each of the down steam processes also affects the performance of grinding operation. The review of literature also showed that a good model to describe the throughput of the grinder in producing desired range of particle sizes is not available. The overall aim of this research is to explore further the potential of developing a firm basis to model the performance of biomass grinders.

The specific objectives of this research are as follows:

- 1. Conduct grinding tests on a model fibrous biomass and characterize the size and size distribution of particles after size reduction.
- 2. Develop the population balance method and test its applicability to predict size, size distribution and flow of biomass in a grinder.

Switchgrass is a promising biomass, and cutter mill is the best known grinder for its low energy consumption due to the shearing action. Experimental measurements on switchgrass ground in a grinder are used to verify the theoretical population balance model. The population balance can be used to develop guidelines for selecting the optimum residence time of switchgrass in the grinder to achieve a specific size or size distribution in a mill. The methodology developed in this work can also be easily applied to other potential candidate biomass materials and other types of grinders.

1.7 Scope and organization of the thesis

This thesis is organized into five main chapters. Chapter 1 presents a review of fibrous material characterization, size reduction process and different theories for predicting the power consumption of size reduction process, and the objectives of this research. Chapter 2 introduces the type of experiments, experimental procedures. Chapter 3 shows data analysis methods, results and discussions of first series of tests. Chapter 4 presents the population balance method. The preliminary experiments and their results are explained. The experiments for estimation of main parameters of population balance are described. Chapter 5 draws conclusions and recommendations for future work.

Chapter 2 Experimental Apparatus and Setup

This chapter introduces two sets of experiments carried during the course of this research in order to understand the followings: the characteristics of fibrous particles during size reduction in a grinder; and the energy consumption associated with size reduction of switchgrass.

2.1 Experimental setup

Figure 2.1 shows a diagram of the test apparatus that consists of four main components: a vibratory feeder, a grinder, a set of sieves and an electronic balance. The following sections explain the test equipment.

2.1.1 Vibratory feeder

The vibratory feeder is an ERIEZ model-15A (Eriez Manufacturing Co., Erie, PA) with a narrow flat feeder trough, 406 mm long and 51 mm wide. The full load power consumption is 15 W, and the power supply is 115 V and 60 Hz, single phase. The feeder speed is controlled by varying the applied voltage. The voltage control can be set from 0 to 100%. Maximum vibration when set to 100% is 1000 cycles/s (1 kHz).

In order to investigate the relationship between quantities of switchgrass of varying sizes being fed to the grinder and different vibration rates of feeder, a series of tests were designed and performed. Switchgrass stems were extracted from a mix of harvested switchgrass. The stems were passed through the grinder once with no screen installed inside the grinder. The ground material was analyzed using a set of wire mesh sieves with mesh numbers: $\frac{1}{4}$, $\frac{3}{2}$, 5, 7, 10 and 14. The opening sizes of the sieve's holes were: 6.35, 5.66, 4.00, 2.83, 2.00, and 1.41 mm, respectively.

Roughly 30 g sample of particles which were retained on each sieve was loaded on to the tray of the vibratory feeder. The vibration control was set at 30%, 45%, 60%, 80%, and 100%. The feeding time of the whole sample was measured by a stop watch. The whole
procedure was repeated three times for each sample at a given vibration rate. Figure 2.2 shows the change of feeding rate (g/s) vs. vibration rate (%).



Figure 2.1 Diagram of test apparatus and data logging system.



Figure 2.2 Feed rate versus feeder vibration rate for five narrow size particle samples of switchgrass (100% is 1 kHz).

Figure 2.2 shows that in order to get a constant feeding of biomass established, the minimum vibration had to be set at 30% of its maximum cycle rate (1 kHZ). As a general rule, smaller particles (material on mesh 14) had the smallest feed rate among samples at a given vibration. Excluding mesh 10 material, it appears the feeding rate vs. vibration is parallel. This indicates that the ratio of feeding rate over the vibration yield roughly a constant value.

2.1.2 Grinder

The mill that was used in the experiments is a Retsch grinder SM100 model (Retsch Inc. Newtown, PA). The cutter rotor is powered by a single phase AC motor, with a rated speed of 1690 rpm at 60 Hz with $\cos \phi = 0.93$. The voltage is 120 V and its power is rated at 1.5 kW. Figure 2.3 shows a picture of the grinder. Figure 2.4 shows the inside of the grinder. Screens with different sizes can be installed in the grinder. Screens with circular perforations come in sizes: 0.25, 0.50, 0.75, 1.00, 1.5, and 2.00 mm. Screens with square perforations come in sizes: 2.00, 4.00, 6.00, 8.00, 10.00, and 20.00 mm.



Figure 2.3 Retsch grinder SM100



Figure 2.4 Inside of Retsch grinder SM100

Figure 2.5 shows the two kinds of perforated screens (circular and square perforated) that can be installed in the grinder.



Figure 2.5 Screens that can be installed in grinder SM100

2.1.3 Sieve shaker

The sieve shaker is a Tayler test sieve shaker, RoTap and Coarse Model (W.S. Tayler Canada, St. Catharine, ON). The sieves motion closely resembles hand sieving. It has rotational motion along with a tapping motion that is caused by a hammer. Figure 2.6 shows a picture of the sieve shaker. The shaker oscillated 278 times per minute and taps 150 times per minute. Tray diameter of 200 mm standard sieves with openings ranging from 50 millimetres (2 in) to 20 micrometers are available. The openings are squares made of woven wire. The actual openings are smaller than those corresponding to the mesh numbers because of the thickness of the wires.

One of the most common series of screens is the Tayler standard screen series. The Tayler series identifies sieves by number of openings per inch. The sieves made in USA are most commonly identified by an arbitrary number that does not necessarily represent the number of openings per inch. These sieves also are identified by their opening size in millimetres or microns.

The balance used for weighing the samples has a 1000 g capacity, with a sensitivity of 0.01 g.



Figure 2.6 RoTap sieve shaker Coarse Model.

2.1.4 Data logging system

The data logging system is shown by dashed line in Figure 2.1. The system consists of three main parts:

- A wattmeter, model PCI-118E (Ohio Semitronics Inc., Hilliard, OH);
- A data acquisition card CIO-DAS08 (Techmatron Instruments Inc., Mississuga, ON);
- A desktop computer;

The input of the wattmeter can range from 0-2500 W, 0-25 A, and 0-150 V. The output of the wattmeter varies between 4 and 20 mA. 4 mA means zero power drawn and 20 mA means maximum power drawn, 2500 W. This current is connected to a 250 Ω resistance. The voltage of the resistance is recorded by the data acquisition card. The voltage change based on time is saved in the computer. Test method is explained in Section 2.3.2.

2.2 Test sample

In this study switchgrass was chosen as the biomass. McLaughlin (2005) stated that switchgrass has the potential to be a sustainable supply for bio-energy and bio-product feedstock.

Specifically the crop has the following positive attributes:

1. It has high productivity levels;

2. It is a perennial crop, which reduces the management intensity and energy consumption of its production;

3. It can be grown in areas that are not suitable for other agricultural crop;

4. Farmers are familiar with its harvesting and the machinery required for its production;

Switchgrass (*Panicum Virgatum*, *L*) was collected as a round bale from a farm in Manitoba. The stalks were cut manually to three lengths: 75 mm, 50 mm, and 25 mm. The moisture content of the sample was measured according to the ASAE standard (ASAE S358.2 DEC99). Three samples of 25 g each were dried at 103 °C for 24 hours. The average moisture content is 6.5% on a wet basis.

2.3 Method

Two series of preliminary tests were performed in order to better understand the process of switchgrass size reduction. The objective of the first series was to understand the characteristics of ground switchgrass particles after size reduction in the grinder. The objective of the second series was to monitor the energy consumption of the size reduction process.

2.3.1 Preliminary tests number 1

A sample of 65 g of material was weighed and gradually hand fed to the grinder. No screen was used in the mill. The outlet of the device was open. Therefore all the material immediately left the grinder and was discharged into a container. The grind was subjected to sieve analysis. The sieves, with mesh numbers of $\frac{1}{4}$, $\frac{3}{2}$, 5, 7, 10, 14, 18, 25, 35, and 45, were used. The sieve openings were 6.35, 5.66, 4, 2.83, 2, 1.41, 1, 0.707, and 0.354 mm, respectively. The sieving time was set for 10 minutes (ASAE S319.3). Appendix I summarizes the results. The material on each sieve was collected and weighed. The sieved material was well mixed and re-fed into the grinder for further grinding. This considered as the first cycle of grinding. The procedure of passing through the grinder without screen, sieving, weighting of material and mixing the sieved materials (one cycle of grinding) is repeated six times. It is called cycles of grinding in this thesis. The entire

procedure of six cycles of grinding was repeated using three switchgrass samples each 65 g.

2.3.2 Preliminary tests number 2

In these tests, the power drawn by the grinder while it was idling was measured and recorded for 30 s. This test was repeated three times on three samples of the same size. The power drawn by the grinder with switchgrass inside it was measured. Switchgrass stems were separated from the leaves. Stems were then passed through the grinder once without screen. The collected material was sieved through 7 sieves: 1/4, $3\frac{1}{2}$, 5, 7, 10, 14, 18 plus a pan. The opening sizes of sieves were 6.35, 5.66, 4, 2.83, 2, 1.41, and 1 mm respectively. The material retained on each sieve was collected in individual containers. To measure power, the particles retained on sieve with mesh number 14 (sieve opening of 1.41 mm), was collected. Roughly 30 g of collected material was weighed and put onto the feeder tray. The grinder and data logging system were turned on. The material was then fed into the grinder. The grinder worked for 30 s. The data logging system recorded the energy consumption during grinding.

Chapter 3 Results and Analysis of preliminary tests

This chapter presents the experimental results and discussion for the preliminary tests described in Chapter 2. This chapter also reviews literature pertinent to characterization of particulate material.

3.1 Characterization of particles size

Several parameters are defined for characterizing the size of an irregularly shaped particle. The applicability of one or several of these parameters depends upon the type of particles and the process. Some of these parameters are:

- 1. Volume diameter: diameter of a sphere having the same volume as the particles;
- 2. Surface diameter: diameter of a sphere having the same surface area as the particles;
- 3. Surface volume diameter: diameter of a sphere having the same ratio of surface area to volume as the particles;

In order to study the particle size (or any particle characteristic) of particles the first step is to select a sample of particles that are a representative of all of the particles. Powders may be classified as non-segregating (cohesive) or segregating (free flowing) (Perry et al., 1997). Taking representative samples of cohesive powders is relatively easy when they are well mixed. It is difficult to mix segregating powders. Problems arise in sampling due to non-homogeneities. If the bulk of particles is homogeneous, or can be mixed prior to sampling in order to generate a homogeneous powder, sampling problems do not arise (Allen, 1997). Handling of free flowing particles results in size segregation. To avoid this segregation of particles it is recommended that the two golden rules of sampling be followed. The first rule is that the powder should be sampled when in motion. The second rule is that the entire stream of powder should be sampled for many short increments of time. There are different methods of powder sampling. Among them, the spinning riffler obeys the golden rules of sampling and generates the best representative samples (Perry et al., 1997). In this device a ring of containers rotates under the powder feed. If the powder flows for a long time compared with the period of rotation the sample in each container will be made up of many small portions drawn from all parts of the bulk.

3.1.1 Sieving methods

One of the ways to characterize a sample of particles is by sieving. In sieving, the particles are separated by passing through a set of surfaces containing holes of a specified uniform size. As sieving handles a large quantity of powder, it minimizes sampling problems. Most industrial sieves used for fractionating powders are made from woven wire cloths, woven wires, or punched metals.

A sieving apparatus consists of a set of sieves arranged on top of one another. The stack of sieves shakes in such a way that particles which are small enough to pass through the openings do so. Screens are arranged in flat sieves or in cylindrical sieves. The shakers can operate in one (to and fro motion), two (circular motion) or three dimensional movements (circular and vertical motion). Sieves are often referred to by their mesh size. Mesh size is the number of wires per linear inch (Allen, 1997; and ASTM Standard E 828-81, 2004). ASTM standards range from 635 mesh (20 μ m) to 5 mesh (125 μ m). For sieves No. 4 and smaller sieve sizes, the abbreviation No. shall be used each time a sieve is indicated by a mesh number (ASTM Standard E 828-81, 2004).

Sieving is based on gravimetric force. One problem with the sieving process occurs when long, thin particles pass vertically through sieve holes. These results in particles can be wrongly allocated to a range of size that they do not belong to. For agricultural biofuels that consist of stems of long and thin cylindrical shape, this could be a major concern. The RoTap sieve shaker explained in Section 2.1.3 is an inefficient way for separating switchgrass stems particles according to the length.

To verify the characteristics of particles retained on each sieve a representative sample of particles form each sieve is selected. Length and the maximum diameter of the particles belong to each sample is measured with a calliper. The results are summarized in Appendix II. It is shown that the sieve opening is not a representative of the particles length but it is a representative of the particles maximum diameter. However, this relation is weakened as the particles size increased. For particles retained on sieve with opening

size of 1.4 mm the mean of the maximum diameter is 1.44 mm but for particles retained on sieve with opening size of 5.66 the mean of the maximum diameter is 3.07 mm. Hartmann et al. (2006) studied different methods of size classification of wood chips. He mentioned that screens are common in wood classification. Because of the mentioned problem associated with long and thin particles the application of a dynamic online image analysis can improve its effectiveness. This new classification method can sort particles based on more than one dimension. But it has the problem of particles overlapping. So the most reliable method of characterizing the size of particles is still direct measurement of size by hand using a calliper.

3.1.2 Methods of particle size data analysis

Collection of raw data in a sample is not informative. Statistical description of the collected data helps us to extract useful information from the raw data. One of these statistical descriptions is the mean of the data. The mean is calculated according to Equation 3.1.

$$\frac{1}{x} = \frac{1}{n} \sum_{i=1}^{n} x_i$$
 3.1

In Equation 3.1 \overline{x} is the mean, *n* is the number of times the variable *x* is measured, and x_i is the *i*th measured variable.

Different sets of data may have the same mean. There is a need to define a statistical function that shows how the data are scattered around the mean. The standard deviation is calculated using Equation 3.2.

$$s = \sqrt{\frac{1}{n} \sum_{i=1}^{n} (x_i - \overline{x})^2}$$
 3.2

s is the standard deviation, *n* is the number of times the variable is measured, x_i is the i^{th} measured variable, and \overline{x} is the mean of the data.

Most measurable quantities in engineering and science consist of a continuous range of measured values. Such quantities are therefore modeled by a continuous random variable

X, whose member x can occur anywhere on some continuum of points, called the sample space S (Bury, 1999). The uncertainty of occurrence of a particular value x is measured by probability. It is modeled by a mathematical function f(x) that describes the density of probability for possible values x over its sample space S:

$$\Pr\left(x - \frac{dx}{2} \le X \le x + \frac{dx}{2}\right) = f(x)dx,$$
3.3

The function f is the probability density function (pdf). The cumulative of these probabilities are the cumulative distribution function (cdf). The probability model is associated with one or two values in order to provide flexibility in modeling. The more parameters are defined with a distribution, the more flexible is the model to fit the data (Bury, 1999). There are three types of parameters, and each gives a different kind of flexibility to the model:

- 1. Location parameters locate the model on its measurement axis;
- 2. Scale parameters scale the model on its measurement axis;
- 3. Shape parameters determine the basic shape of the function;

Particle size distribution is an important property of a group of particles. It shows the effectiveness of a grinding system (Pasikatan et al., 1999). It has also been used to control powder packing (Ramkrishnan, 2000). Particle size distribution can show the percentage of fine particles in a group of particles. For some conversion processes this percentage is important and clearly defined. For example, the content of very fine particles (smaller than 100 μ m) should be higher than 10% by weight in order to achieve short ignition times (Esteban et al., 2006). It is important to predict when the need of the conversion process is met.

3.1.2.1 Normal distribution

A fundamental property of the normal distribution function is that differences from the mean are equally distributed (Allen, 1997). This means that the probability of finding particles 10 units larger than the mean is the same as that of finding particles 10 units smaller. The pdf of a set of data that has a normal distribution is a Gaussian bell-shaped

curve. A continuous random variable has a normal distribution if its probability density function has the form:

$$f(x;\mu,\sigma) = \frac{1}{\sigma\sqrt{2\pi}} \exp\left\{-\frac{1}{2}\left(\frac{x-\mu}{\sigma}\right)^2\right\} \qquad \qquad \sigma > 0, \ -\infty < x \qquad \qquad 3.4$$

In Equation 3.4 μ is the mean of the data and σ is the standard deviation of the data. For normal distribution μ is a location parameter and σ is a scale parameter. It is related to the units of measurement for *x*. The corresponding cumulative distribution function of the data is:

$$F(x;\mu,\sigma) = \frac{1}{\sigma\sqrt{2\pi}} \int_{-\infty}^{x} \exp\left\{-\frac{1}{2}\left(\frac{x-\mu}{\sigma}\right)^{2}\right\} dx$$
3.5

It might be expected that this type of distribution would be common; however most distributions are skewed usually to the right.

3.1.2.2 Log-normal distribution

Log-normal distributions have been a popular choice to describe the behaviour of dispersed particles (Hamilton et al., 2003). In log-normal the logarithm of variable x is normally distributed. In order to maintain the bell-shaped curve it is necessary to plot relative occurrence against size in a geometric progression (Allen, 1997). A continuous random variable x has a log-normal distribution if its probability density function has the form:

$$f(x;\mu,\sigma) = \frac{1}{x\sigma\sqrt{2\pi}} \exp\left\{-\frac{1}{2}(\frac{\ln x - \mu}{\sigma})^{2}\right\} \qquad \sigma > 0, \ x > 0, \qquad 3.6$$

Because of the close relationship of normal distribution and log-normal distribution μ and σ have appeared here as well. μ is the mean of the logarithm of data in e basis, and σ is the standard deviation of the logarithm of data in e basis. The corresponding cumulative distribution function of the data is:

$$F(x;\mu,\sigma) = \frac{1}{\sigma\sqrt{2\pi}} \int_{-\infty}^{x} \frac{1}{x} \exp\left\{-\frac{1}{2}\left(\frac{\ln x - \mu}{\sigma}\right)^{2}\right\} dx \qquad 3.7$$

3.1.2.3 Weibull distribution

A continuous random variable x has a Weibull distribution if its probability density function has the form:

$$f(x;\sigma,\lambda) = \frac{\lambda}{\sigma} \left(\frac{x}{\sigma}\right)^{\lambda} \exp\left\{-\left(\frac{x}{\sigma}\right)^{\lambda}\right\} \qquad x \ge 0; \ \sigma,\lambda > 0 \qquad 3.8$$

A Weibull variable *x* has a cumulative distribution function:

$$F(x;\sigma,\lambda) = 1 - \exp\left\{-\left(\frac{x}{\sigma}\right)^{\lambda}\right\}$$
3.9

This model has a scale parameter of, σ , while λ is a shape parameter. Rearranging Equation 3.9 and taking logs twice give:

$$\ln[-\ln(1-F)] = -\lambda \ln(\sigma) + \lambda \ln(x)$$
3.10

Plotting ln[-ln(1-F)] vs ln(x) for the cumulative distribution function gives a line with the slop of λ and intercept of $-\lambda \ln(\sigma)$. Also, the coefficient of determination, R-square, shows how good the fit is.

3.1.3 Standards to characterize the size of particles

There are several standards to characterize the size of particles retained on sieves with known hole size.

3.1.3.1 ASAB Standards

ASBE (2007) describes two standards for analyzing the size and size distribution of ground particles. ASAE S319.3 introduces a method for determining and expressing fineness of feed materials by sieving. The standard is specified for the particles yielding from reduction processes that are primarily spherical or cubical. The sieves for this standard are woven wire-cloth with hole sizes ranging from 4.75 mm down to 53µm. The circular sieves are placed on common-shaking equipment.

ASAE 424 (ASABE 2007) describes a method of determining and expressing particle size of chopped forage materials. The sieve holes for this standard are square with a specified thickness. The thickness is designed to prevent the escape of long particles to the lower sieve. The number of sieves specified for this standard is four with nominal size opening of 19 mm down to 1.17 mm. The corresponding plate thickness ranges from 12.7 mm to 0.64 mm. The shaking device introduces only reciprocating horizontal motion. For both standards S319 and S424 calculation of particle size mean and distribution is based on the assumption that the ground material have a logarithmic-normal distribution. Geometric mean diameter or median size of particles by mass is defined by:

$$d_{gw} = \log^{-1} \left[\frac{\sum_{i=1}^{n} (W_i \log \overline{d}_i)}{\sum_{i=1}^{n} W_i} \right]$$

$$3.11$$

 d_{gw} is geometric mean diameter or median size of particles (mm) by mass. W_i is the mass of particles on i^{th} sieve. Sieves are numbered from 1 to n with the numbering starting from the sieve with the biggest opening and *n* is the pan. \overline{d}_i is the geometric mean diameter or median size of particles on i^{th} sieve (mm).

$$\overline{d_i} = (d_i d_{i-1})^{1/2}$$
3.12

 d_i is the sieve size on which the particles are from. d_i for pan is taken as half of the size of the smallest sieve in the stack. S_{log} is geometric standard deviation of log normal distribution by mass in ten-based logarithm.

$$S_{\log} = \left[\frac{\sum_{i=1}^{n} W_i (\log \overline{d}_i - \log d_{gw})^2}{\sum_{i=1}^{n} W_i}\right]^{1/2}$$
3.13

Standard ASAE S319.3 specifies the calculated S_{log} from Equation 3.13 (geometric standard deviation of log normal distribution by mass). A geometric standard deviation of particle diameter by mass is then estimated from the following equation.

$$S_{gw} \approx \frac{1}{2} d_{gw} [\log^{-1} S_{\log} - (\log^{-1} S_{\log})^{-1}]$$
 3.14

This conversion from log normal standard deviation to linear standard deviation has not been specified in ASAE S424.3. The reason for this omission is not known.

3.1.3.2 ASTM Standard E828-81

ASTM E828-81 (ASTM 2004) outlines a test method for designating the size of RDF-3 from its sieve analysis. RDF (refuse derived fuel) is a shredded fuel derived from processed municipal solid waste (MSW). Two methods of sieving are explained in the standard: hand sieving and machine sieving.

For machine sieving the first priority of apparatus in this standard for RDF-3 50 mm or smaller is horizontal rotating cylindrical screens. This apparatus readily provides the lifting and tumbling action required to bring all materials to the screen surface. Alternatively the screen can be of a rectangular geometry. Trays in this sieve shaker have a 0.46 by 0.66 m clear screen area. The screening motion is vertical. Up to six screen trays can be held in the vibrating unit.

For RDF-3 smaller than 0.01 m, circular sieves of 0.3 m diameter and the RoTap shaker is satisfactory. In order to obtain a complete characterization of the size range of an RDF-3 sample, it is necessary that the number of sieves be such that no more than 25% of the gross sample weight will be retained on any given sieve. The nominal bottom size sieve should allow no more than 15% of the sample weight to pass through it. Standard (ASTM E828-81, 2004) outlines a method for determination of nominal and mean particle sizes based on the Rosin-Rammler graph.

3.1.3.3 ISO 3310-1

ISO 3310-1 (2007) specifies the dimensional accuracy of the test sieve openings for test sieves of metal wire mesh. It recommends methods for visual examination of the sieve openings and for measurement of average opening size and its tolerance. It also recommends that sieve openings be tested after being used for some time to see if the openings are still according to the standards.

3.2 Results

The results of the two sets of preliminary tests (tests number 1 and number 2) are presented. The apparatus, method, and material for these tests are described in Chapter 2.

3.2.1 Preliminary tests number 1

Table 3.1 lists the raw data collected when six grinding cycles were performed. Column 1 of Table 3.1 shows the sieve openings in mm. Columns 2 to 7 show the mean weights (of three duplicated samples) of the material retained on each sieve. The numbers in parentheses are the standard deviations of the three results.

Figure 3.1 shows the weight fraction distribution of the six grinds of 25 mm precut switchgrass particles. The graph shows that as more grinding cycles are performed the peak of the curves shifted to the smaller sieve opening sizes. The cumulative weight fractions of the six grinds are depicted in Figure 3.2.

| Sieve opening, mm | 1 st grind | 2 nd grind | 3 rd grind | 4 th grind | 5 th grind | 6 th grind |
|-------------------|-----------------------|-----------------------|-----------------------|-----------------------|-----------------------|-----------------------|
| 0.000 | 0.39 (0.04) | 0.63 (0.08) | 0.87 (0.13) | 1.10 (0.29) | 1.2 (0.04) | 1.45 (0.19) |
| 0.345 | 0.21 (0.02) | 0.39 (0.01) | 0.53 (0.01) | 0.76 (0.14) | 0.82 (0.07) | 1.06 (0.04) |
| 0.500 | 0.51 (0.09) | 0.89 (0.13) | 1.46 (0.22) | 2.33 (0.04) | 2.70 (0.75) | 3.53 (0.35) |
| 0.707 | 1.56 (0.15) | 4.42 (0.32) | 7.71 (0.35) | 11.76 (1.02) | 12.31 (1.26) | 14.76 (0.25) |
| 1.000 | 7.35 (0.89) | 14.38 (0.31) | 18.33 (0.29) | 19.92 (0.95) | 20.41 (0.26) | 20.94 (0.21) |
| 1.410 | 15.56 (1.08) | 19.07 (0.56) | 18.78 (0.14) | 17.48 (0.60) | 16.51 (0.72) | 14.98 (0.53) |
| 2.000 | 17.41 (0.43) | 12.79 (0.35) | 9.74 (0.12) | 6.87 (0.87) | 6.53 (1.29) | 4.66 (0.17) |
| 2.830 | 16.51 (1.39) | 9.94 (0.71) | 6.1 (0.23) | 3.23 (0.77) | 2.90 (0.64) | 1.8 (0.12) |
| 4.000 | 4.4 1 (1.07) | 1.78 (0.63) | 0.76 (0.18) | 0.30 (0.12) | 0.17 (0.15) | 0.05 (0.03) |
| 5.660 | 0.61 (0.31) | 0.19 (0.08) | 0.05 (0.02) | 0.01 (0.02) | 0 | 0 |
| 6.350 | 0.30 (0.23) | 0.11 (0.04) | 0.06 (0.01) | 0 | 0 | 0 |

Table 3.1 Weight of material retained on each sieve for 6 grinds of 65 g of 25 mm precut samples. Number in parentheses shows the standard deviation for three duplicates.



Figure 3.1 The change of weight fraction versus sieve opening (mm) for the six grinds of switchgrass

Log-normal distribution is a preferred data reduction technique to describe the behaviour of dispersed particles (Hamilton et al., 2003). The log-normal distribution was tested for

the data of six grinds. Cumulative weight fractions are given vs. logarithm of the size of the sieves. Correlations of the data with linear functions are calculated. The results of correlations are shown in Table 3.2, with the first column showing the number of grinds. The second column lists the mean (location parameter) of the distribution and the third column lists the standard deviation (scale parameter) of the distribution. The fourth column is the coefficient of determination.

Table 3.2 Mean (location parameter), standard deviation (scale parameter) and coefficient of determination for correlation of six grinds of 25 mm precut switchgrass with log normal distribution

| Grind | μ | σ | R-square |
|--------|--------|--------|----------|
| First | 0.8001 | 0.4845 | 0.9312 |
| Second | 0.5733 | 0.5307 | 0.9271 |
| Third | 0.4160 | 0.4627 | 0.9023 |
| Fourth | 0.3146 | 0.4658 | 0.876 |
| Fifth | 0.2244 | 0.2228 | 0.8516 |
| Sixth | 0.1721 | 0.2280 | 0.8349 |

When more grinding cycles are performed, the correlation coefficient moves farther from 1, starting from 0.93 for the first grind and decreasing to 0.83 for the sixth grind.

The Weibull distribution is tested on the sieving data (see Section 3.1.2.3). Plotting ln[-ln(1-F)] vs. ln(x) for the cumulative distribution function gives a line with the slope of λ and an intercept of $-\lambda \ln(\sigma)$ (Equation 3.10). The coefficient of determination (also known as R-square) shows how good the fits are. The results of regression with six grinds of 25 mm precut switchgrass are summarized in Table 3.3. The first column shows the number of the grind. The second column shows the coefficient of determination (R-square) of the fits. The R-squares change from 0.977 for the first grind to 0.954 for the sixth grind indicating that the data fit is better for the Weibull function than the lognormal distribution. The shape parameter, λ , ranges from 2.067 to 2.474; the scale parameter, σ , ranges from 1.303 to 1.646.

Cumulative size distributions versus sieve opening sizes are shown in Figure 3.2. Weibull distributions for each grind are also depicted in Figure 3.2. It is observed that Weibul distribution shows a good fit for all six cycles of grinding of switchgrass.



Figure 3.2 Cumulative weight fraction versus sieve opening (mm) of six grind of 25 mm precut switchgrass

| Table 3.3 R-square, shape parameter |
|---|
| and scale parameter for the six grinds of |
| 25 mm precut switchgrass |

| Grind | R-square | λ | σ |
|--------|----------|-------|----------|
| First | 0.9773 | 2.474 | 1.646058 |
| Second | 0.9605 | 2.300 | 1.528859 |
| Third | 0.9565 | 2.155 | 1.466566 |
| Fourth | 0.9662 | 2.362 | 1.319740 |
| Fifth | 0.9565 | 2.155 | 1.466566 |
| Sixth | 0.9541 | 2.067 | 1.303207 |

Although particles used in the tests are cylindrical shape with relative large aspect ratio (length over diameter is greater than 1), the ASAE S319 was applied to the six grind cycles. The results are summarized in Table 3.4.

Table 3.4 d_{gw} , S_{log} and S_{gw} calculated for six grinds of 25 mm precut switchgrass based on ASAE S319.3 (2006)

| Grind | | First trial | | | Second tria | ıl | Т | Third trial | |
|--------|----------|-------------|----------|----------|-------------|----------|----------|-------------|----------|
| | d_{gw} | S_{log} | S_{gw} | d_{gw} | S_{log} | S_{gw} | d_{gw} | S_{log} | S_{gw} |
| First | 2.188 | 0.220 | 1.158 | 2.135 | 0.217 | 1.116 | 2.302 | 0.223 | 1.239 |
| Second | 1.742 | 0.231 | 0.974 | 1.744 | 0.227 | 0.956 | 1.775 | 0.239 | 1.027 |
| Third | 1.486 | 0.237 | 0.853 | 1.487 | 0.228 | 0.820 | 1.500 | 0.236 | 0.857 |
| Fourth | 1.343 | 0.227 | 0.735 | 1.319 | 0.229 | 0.729 | 1.235 | 0.235 | 0.703 |
| Fifth | 1.225 | 0.230 | 0.681 | 1.231 | 0.229 | 0.680 | 1.327 | 0.237 | 0.763 |
| Sixth | 1.162 | 0.230 | 0.646 | 1.154 | 0.230 | 0.640 | 1.210 | 0.266 | 0.418 |

The data shows that particle sizes decreased with increase in the number of cycles. In all three trials, the size of particles decreased almost to the half of the original size. The standard deviations (S_{gw}) also decreased with increase in grinding cycles. This indicates that as grinding progresses the particles being ground become more uniform in size. It is also interesting to note that S_{log} does not reflect in this change in standard deviation while S_{gw} does. This indicates that Standard S424 needs to be updated to include estimation of S_{gw} from S_{log} .

A breakage ratio is defined as the ratio of the particle size after each grinding cycle over its initial particle size prior to grinding. Data of Table 3.4 was used to calculate the breakage ratios and fit an exponential curve and a power curve to the data. Figure 3.3 is a plot of breakage ratio vs. grinding cycles. As Table 3.4 shows the maximum experimental grinding cycles were 6. The curves are extrapolated to show the trends. Figure 3.3 shows that a power curve fits the data better than an exponential curve. As is expected a larger breakage ratio happens during early cycles than during the subsequent cycles. Obviously this curve is specific to the type of grinder and material. It is interesting to find out the form of these curves and formula for other types of grinders.



Figure 3.3 Exponential and power curves fitted to the data from the first trial of grinding cycles in Table 3.3.

3.2.2 Preliminary tests number 2

The objective of the experiments was to measure the energy consumption of size reduction of switchgrass. The raw data collected are shown in Figure 3.4. The data logging system is described in detail in Section 2.1.4. Output of the system is voltage vs. time. One hundred data were recorded every second. The x-axis in Figure 3.4 is time in one hundredth of second. The y-axis is voltage.



Figure 3.4 Raw data from data logging system

When grinder was off the voltage was 1 V and when grinder was idling the voltage was 1.85 V. The method is described in Section 2.3.2. The power required to run the grinder without feeding was measured which allowed for the determination of the net power required to grind the material. The specific energy required for grinding the material was determined from the subtraction of the power drawn when the grinder was grinding the material from the power drawn when the grinder was idling over 30 s grinding period. The feed samples were particles that passed through the sieve with a mesh number 10 (sieve opening 2 mm) and retained on the sieve with mesh number 14 (sieve opening 1.41 mm). The ground particles were collected after grinding and analysed for their size by sieving. Test was repeated three times. Figure 3.5 shows the graph of cumulative undersize particles vs sieve size on a logarithmic probability paper for three ground samples. From the graph the sieve size that 80 percent of particles passed through were specified.

Results of the power drawn by the grinder when it is grinding switchgrass are summarised in Table 3.5. The first column shows the test number. The second column lists the feed size before grinding. The third column lists the size of the sieve that 80% of the particles passed through (Figure 3.5) for each sample. The last column of Table 3.5 lists the energy required to perform each test. In the first test the minimum size reduction was obtained because feed particles of 2 mm in size reduced to a product of 1.9 mm in



size. The energy required for this test was the least. The same trend continues in the second and third tests.

Figure 3.5 Cumulative undersize distribution by mass for three samples of ground switchgrass

| Test no. | Feed | Ground | Energy required |
|----------|------|--------|-----------------|
| | (mm) | (mm) | (kWh/t) |
| 1 | 2 | 1.9 | 131.62 |
| 2 | 2 | 1.6 | 150.51 |
| 3 | 2 | 1.7 | 143.60 |

Table 3.5 Feed and product particle size, and energy required for grinding switchgrass for three grinding tests

Mani et al. (2004) performed a similar test. The only difference was that they installed three screens with a different sized opening in the grinder. They reported a specific energy requirement of 62.55 kWh/t for grinding switchgrass. In their study feed particles had 25-30 mm chop size and the installed screen had a mesh with 0.8 mm openings (minimum screen size opening of the installed screen). The moisture content of switchgrass was 8 % wt wet basis. The difference between the results is because of two reasons:

- 1. In this research the output of the grinder was blocked and material stayed in the grinder during grinding.
- 2. The friction between the particles and the rotating blades causes energy consumption during grinding.

Chapter 4 Modeling of Size Reduction

In this chapter the population balance model is introduced. The population balance equations are derived, with a focus on size reduction. The method of measuring and estimating the two parameters for the population balance equation is introduced and described. The parameters are calculated for grinding switchgrass. A set of population balance equations is then solved for batch grinding to predict the change of particle size in the grinder as a function of grinding time.

4.1 An overview of population balance equation

In all chemical and physical processes the two governing laws are the laws of conservation of mass and energy. These two laws are the basis for design and control of any process. When a process involves dispersed phases or particulate materials such as colloids, polymers, powders or emulsions, population balance is commonly used to characterize the changes in particle numbers and sizes (Lin et al., 2002). Hill et al. (1995) and Scarlett (2002) defined population balance as a modeling tool to describe the change in particulate system in a controlled manner. Population balance can help to optimize and design a control system for the process and analyze the effect of change of operating conditions on the efficiency of the process.

A general form of the population balance equation for grinding is as follows (Hill et al., 1995; Randolph et al., 1988; Mishra, 2000; Scarlett, 2002):

$$\frac{dn(d,t)}{dt} = [Inflow] - [Outflow] + [Birth] - [Death]$$
4.1

In Equation 4.1, n(d,t) refers to the number concentration of particles between size d and d+dd during the time interval dt. Number concentration is the ratio of number of particles of a specific size divided by the total number of particles in the system. The birth and death terms represent an increase or decrease in the number of particles due to agglomeration, attrition, breakage, or nucleation. The *inflow* means the addition or

entering of particles to the system. The *outflow* means the removal or *leaving* of particles from the system.

Randolph et al. (1988) formulated number based population balance equation in studying crystallization. Vanni (1999) and Mishra (2000) introduced the population balance similar to Equation 4.1 but defined the corresponding parameters in a mass basis. Scarlett (2002) considered the population balance equation as number based, but stated that the word "balance" can be misleading; the objective is to study changes in particles population. Austin and colleagues have published a number of papers on size reduction of mieral particles (Austin et al., 1964; Klimple et al., 1970; Austin, 1971; Austin et al., 1971; Gardner et al., 1975; Jindal et al., 1976; Austin et al., 1976, and Austin, 1999). The basis of their work is also the mass-based population balance. They explained that their technique consists of describing the grinding of material in a mill as a rate process in a manner similar to that used to describe chemical reaction in a reactor (Austin et al., 1976). In their preliminary work they considered the grinding process as a first order reaction (Klimpel et al., 1971). Later they found that the breakage of large particles in a ball mill did not follow the first order kinetic law whereas the breakage of smaller particles did follow this law (Gardner et al., 1975). In a recent study, Bilgili (2007) shows experimentally that the first order rate is only correct for some special cases and for other cases higher order rate equations are applicable.

4.2 Derivation of population balance equation

Figure 4.1 shows the mass balance over a specified size category of particles in a grinder. In batch grinding inflow and outflow are equal to zero. Only two terms of Equation 4.1 are required. They are birth and death terms. The birth term represents the breakage of large particles to form particles having a size of the specified size category. The death term represents the breakage of particles in the specified size category to smaller particles.



Figure 4.1 One specified size category in a grinder and the corresponding population balance equation terms

Population balance equation can be formulated in two ways: mass-based equation and number-based equation. In both forms a density function defines: n(d,t) (number density or the number of particles that have a size of *d* to (d+dd)) or M(d,t) (mass density of the mass of particles that have a size of d to (d+dd)). Equation 4.2 represents the mass based continuous population balance equation.

$$\frac{d}{dt}M(d,t) = \int_{d}^{\infty} S_M(e)b_M(d,e)M(e,t)de - S_M(d)M(d,t)$$
4.2

In Equation 4.2 M(d,t) is the mass of particles that have the size of *d* at time *t*. $S_M(e)$ is the mass fraction of particles with size *e* that are ground in time. $b_M(d,e)$ is the mass fraction of ground particles with size *e* that goes to size *d* or generate particles with size *d*.

Although modern instruments are available for continuous particle size measurements but these instruments are expensive, needs expertise and using them is time consuming. Instead a sieve analysis for limited size intervals is easier to perform. In this case always the question is if the selected intervals are enough for the analysis. Hill et al. (1995) suggested that first one starts from a large interval and progressively reduce the size intervals until predicted particle size distribution remains approximately the same.

Equation 4.3 represents the discrete form of mass balance of particles on sieve size *i* at time *t*:

$$\frac{dM(i,t)}{dt} = \sum_{j=1}^{i-1} M(j,t) S_M(j) b_M(i,j) - S_M(i) M(i,t)$$
4.3

M(i,t) is the mass of particles (g) of size interval *i* at time *t*. $S_M(i)$ (s⁻¹) is the rate of grinding. $b_M(i,j)$ (dimensionless) is a mass-based breakage distribution function. Equation 4.3 implies that the change of the material in each size interval occurs due to two processes:

1. It decreases because of size reduction and is defined by rate of grinding $S_M(i)$. For each size interval *i*, it represents the rate of disappearance of the material due to size reduction.

2. It increases because of the addition of particles from the breakage of bigger particles creates particles having the size of the size interval. The breakage distribution function, $b_M(i,j)$ refers to the material that leaves the size interval *j* and goes to the size interval *i*.

Figure 4.2 shows an example of the application of Equation 4.3 to three size intervals.



Figure 4.2 The simplified 3 steps of size intervals and the correspondent population balance parameter.

Starting from one size particles that belongs to size interval 1 (N=1), the only term that change the amount of particles is the rate of size reduction S(1). Applying Equation 4.3 to particles in interval N=1 simplifies the equation to become:

$$\frac{dM(1,t)}{dt} = -S(1)M(1,t)$$
 4.4

Where M is the mass of particles belong to the interval 1 and S is the rate of disappearance of particles as the materials grind.

As the grinding continues particles that belong to size interval 2 (N=2) are created and their population changes. The change in the population of particles depends on two terms. The particles population disappears by rate S(2) but increases because of the breakage of the previous size (size interval one). Equation 4.3 takes the following form.

$$\frac{dM(2,t)}{dt} = -S(2)M(2,t) + [M(1,t)S(1)b(2,1)]$$
4.5

This process of disappearances and additions of particles can be extended to third size interval as in Equation 4.6:

$$\frac{dM(3,t)}{dt} = -S(3)M(3,t) + [M(1,t)b(3,1) + M(2,t)S(2)b(3,2)]$$
4.6

4.3 Measurement of grinding rate, *S*, and breakage distribution function *b*

Experimental setup, method for the preparation of the samples and method to measure grinding rate and breakage distribution function are described below.

4.3.1 Experimental setup, sample preparation and test procedure

Equipments were the same as those shown in Figure 2.1 and described in Section 2.1 except the vibratory feeder that was not available when these experiments were performed. The procedure followed here is based on those developed by Klimple (1970) and Austin et al. (1976).

The stems of switchgrass were hand cut to pieces and fed to the grinder once without installing any of the screens around the cutter rotor. The collected materials were sieved through 7 Tayler sieves of the following mesh numbers: 1/4, $3\frac{1}{2}$, 5, 7,10,14,18, plus pan. Table 4.1 lists the opening size for each sieve mesh number. The material left on each sieve was collected into a container. In total eight fractions (including pan) were obtained.

Samples 1 and 8 were excluded because they contain a wide size range of particles. Sample 1 contains all the particles bigger than those retained on sieve with mesh number 1/4; sample 8 contains all the particles smaller than mesh number 18. Particles of fractions 2 to 7 (six samples) were used to determine values of *S* and *b*.

Table 4.1 specification of eight fractions of ground switchgrass particles

| Sample number, <i>i</i> | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |
|-------------------------|------|------|------|------|------|------|------|-----|
| Sieve mesh number | 1⁄4 | 31/2 | 5 | 7 | 10 | 14 | 18 | Pan |
| Sieve opening, mm | 6.35 | 5.66 | 4.00 | 2.83 | 2.00 | 1.41 | 1.00 | |

For estimating S_i , the outlet of the grinder was blocked with a curved sheet metal having the same curvature as for a screen. This converts the grinder to a batch grinder. Starting from sample 2 (*i*=2) (material left on sieve mesh number $3\frac{1}{2}$), 30 g of material were weighed and was denoted W_{1i} . The sample was ground for 30 s (t). The ground material was collected and weighed. The ground material was sieved for 10 min on the same set of sieves as those in Table 4.1. The materials left on the sieve mesh number $3\frac{1}{2}$ were weighed and was denoted W_{2i} . Here indices 1 and 2 indicate weight before and weight after grinding on the same size sieve. Index *i* represents the sieve number 2 to 7. For each sieve *i*, S_i was calculated as follows,

$$S_{i} = \frac{\frac{W_{1i} - W_{2i}}{W_{1i}}}{t}$$
4.7

where *t* is the grinding time that was set at 30 s.

For estimating b(i,j), the outlet of the grinder was left open by not installing any of the screens. This simulated a one cycle grinding action in order to minimize interaction of the particles. Starting from sample 2 (material left on sieve $3\frac{1}{2}$), 65 g of material were weighed and was denoted as W_{1j} . The sample was fed to the grinder while the knife rotor was rotating. The ground biomass were recovered and weighed. The material was sieved for 10 min on a stack of sieves $3\frac{1}{2}$, 5, 7, 10, 14, and pan. Materials retained on each sieve were weighed and denoted W(i,2), where i= 2, 3, 4, 5, 6, and pan. b(i,j) was then estimated using the following equation.

$$b(i,j) = \frac{W(i,j)}{W_{1j} - W(j,j)}$$
4.8

In equation 4.8, the denominator is the difference between the mass on sieve *j* before grinding, W_{1j} , and the mass left on the sieve after grinding W(j,j). The numerator is the mass of the particles on sieve *i*.

4.4 Raw data

Table 4.2 shows the three trials of collected data for calculation of *S* (grinding rate). The first row in the table shows the mesh number of the sieve the feed coming from. For each trial the first row shows the weight of the samples before grinding, W_{Ii} . The second row shows the weight of samples after grinding. The difference between the first row and second row signifies the loss of material during grinding. The percent losses are listed in the fifth row for each trial. The sixth row of Table 4.2 lists the mass of material that was not ground and was left on the sieve *i*, W_{2i} .

| Feed mesh number | 31/2 | 5 | 7 | 10 | 14 | 18 |
|--|-------|-------|-------|-------|-------|-------|
| First trial | | | | | | |
| Weight of sample before grinding, | 30.00 | 30.01 | 30.00 | 30.01 | 30.01 | 30.00 |
| W_{1i} , g | | | | | | |
| Weight of sample after grinding, g | 29.11 | 30.08 | 29.92 | 29.89 | 29.49 | 29.93 |
| Grinding duration, <i>t</i> , s | 30.00 | 36.00 | 30.00 | 34.47 | 30.31 | 32.61 |
| Sieving duration, min | 10.00 | 10.00 | 10.00 | 10.00 | 10.00 | 10.00 |
| Lost material, % | 2.97 | -0.23 | 0.27 | 0.40 | 1.73 | 0.23 |
| Weight of un-ground material, W_{2i} , g | 1.80 | 3.60 | 4.40 | 6.18 | 5.84 | 10.05 |
| Second trial | | | | | | |
| Weight of sample before grinding, | 30 | 30.01 | 30.04 | 30.02 | 30.03 | 30 |
| W_{1i}, \mathbf{g} | | | | | | |
| Weight of sample after grinding, g | 29.79 | 29.87 | 29.89 | 29.76 | 29.64 | 29.6 |
| Grinding duration, , <i>t</i> , s | 30 | 30 | 30 | 30.08 | 31 | 33.12 |
| Sieving duration, min | 10 | 10 | 10 | 10 | 10 | 10 |
| Lost material, % | 0.7 | 0.5 | 0.5 | 0.8 | 1.3 | 1.33 |
| Weight of un-ground material, W_{2i} , g | 1.5 | 3.55 | 3.9 | 5.76 | 6 | 8.5 |
| Third trial | | | | | | |
| Weight of sample before grinding, | 30 | 30.01 | 30 | 30.01 | 30.02 | 30 |
| W_{1i}, g | | | | | | |
| Weight of sample after grinding, g | 29.9 | 29.88 | 29.9 | 29.77 | 20.8 | 29.97 |
| Grinding duration, <i>t</i> , s | 30 | 30 | 31 | 30 | 32 | 33.13 |
| Sieving duration, min | 10 | 10 | 10 | 10 | 10 | 10 |
| Lost material, % | 0.33 | 0.43 | 0.33 | 0.80 | 0.73 | 0.10 |
| Weight of un-ground material, W_{2i} , g | 2.6 | 4 | 4.52 | 5.6 | 5.4 | 11 |

Table 4.2 Raw data for the calculation of S (grinding rate) values for three trials.

Table 4.3 lists the weight of the ground particles recovered from various sieves. For each sample collected form a specific size sieve, 65 g was ground. The ground material was analysed on a set of five sieves smaller than the specified sieves and pan. The material retained on each sieve weighted and listed in the correspondent column of Table 4.3. This procedure repeated for all the specified sieves and three duplicated samples from each specified sieve. Appendix III lists the results of the other two duplicated samples.

| | Origin sieve number, <i>j</i> | | | | | | |
|------------------------------------|-------------------------------|-------|-------|-------|-------|-------|--|
| Destination sieve number, <i>i</i> | 2 | 3 | 4 | 5 | 6 | 7 | |
| 2 | 5.82 | | | | | | |
| 3 | 9.38 | 10.06 | | | | | |
| 4 | 16.03 | 19.02 | 19.82 | | | | |
| 5 | 14.03 | 15.45 | 20.71 | 29.25 | | | |
| 6 | 12.26 | 12.62 | 15.33 | 22.45 | 40.39 | | |
| 7 | | 5.26 | 6.56 | 10.00 | 18.16 | 46.11 | |
| 8 | | | 1.31 | 1.96 | 4.03 | 17.34 | |
| 9 | | | | 0.46 | 0.59 | 0.76 | |
| 10 | | | | | 0.29 | 0.25 | |
| 11 | | | | | | 0.13 | |
| Pan | 7.44 | 2.21 | 0.98 | 0.52 | 0.38 | 0.2 | |
| Total ground particles | 59.14 | 54.56 | 44.89 | 35.39 | 23.45 | 18.68 | |
| Total | 64.96 | 64.62 | 64.71 | 64.64 | 63.84 | 64.79 | |

Table 4.3 Weight (g) of the ground particles from specified sieves for the first set of samples

4.5 Analysis

Equation 4.7 was used to calculate S values with input data from Table 4.2. A typical calculation for sieve No. $3\frac{1}{2}$ is as follows:

$$S_2 = \frac{\frac{W_{12} - W_{22}}{W_{12}}}{t}$$
 4.9

Substituting symbols with the corresponding data from Table 4.2 yields:

$$S_2 = \frac{\frac{30.0 - 1.80}{30.0}}{30} = 0.031 \tag{4.10}$$

Calculations were repeated for each of the fractions in Table 4.2. Table 4.4 lists the calculated *S* values.

| Sample number, i | 2 | 3 | 4 | 5 | 6 | 7 |
|----------------------------|-------|-------|-------|-------|-------|-------|
| Sieve mesh number | 31/2 | 5 | 7 | 10 | 14 | 18 |
| Sieve Opening, mm | 5.66 | 4 | 2.83 | 2 | 1.41 | 1 |
| $S(s^{-1})$ (first trial) | 0.031 | 0.024 | 0.028 | 0.023 | 0.027 | 0.020 |
| $S(s^{-1})$ (second trial) | 0.032 | 0.029 | 0.029 | 0.027 | 0.026 | 0.022 |
| $S(s^{-1})$ (third trial) | 0.030 | 0.029 | 0.027 | 0.027 | 0.026 | 0.019 |
| Average S | 0.031 | 0.027 | 0.028 | 0.026 | 0.026 | 0.020 |
| Std. dev. | 0.001 | 0.003 | 0.001 | 0.002 | 0.001 | 0.002 |

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Table 4.4 Results of calculation of *S* values from batch grinding for 3 trials.

In Table 4.4, the fourth row lists the *S* values calculated for each sample based on data from Table 4.2 and Equation 4.7. Similar measurement and calculation were performed for the second and third trials from Table 4.2. The results are summarized in the fifth and sixth row of Table 4.4. The average *S* values indicate that it appears the *S* values appear to decrease as the size of the sieve becomes smaller. This slowing rate of grinding is expected with the smaller size particles.

The question came up whether a batch process represents the grinding rate S for continuous grinding process. A second set of S values were estimated using the grinding and sieving data in Table 4.3 and the duplicated results from Appendix III. We assume that the residence time for material in grinder was 10 seconds. Using this time and weights from Table 4.3, a new set of S values were calculated and listed in Table 4.5.

| Sample number, i | 2 | 3 | 4 | 5 | 6 | 7 |
|----------------------------|-------|-------|-------|-------|-------|-------|
| Sieve mesh number | 31/2 | 5 | 7 | 10 | 14 | 18 |
| Sieve Opening, mm | 5.66 | 4 | 2.83 | 2 | 1.41 | 1 |
| $S(s^{-1})$ (first trial) | 0.091 | 0.084 | 0.069 | 0.055 | 0.037 | 0.029 |
| $S(s^{-1})$ (second trial) | 0.093 | 0.083 | 0.07 | 0.053 | 0.037 | 0.030 |
| $S(s^{-1})$ (third trial) | 0.092 | 0.085 | 0.069 | 0.051 | 0.038 | 0.030 |
| Average S | 0.092 | 0.084 | 0.069 | 0.053 | 0.037 | 0.030 |
| Std. dev. | 0.001 | 0.001 | 0.001 | 0.002 | 0.001 | 0.001 |

Table 4.5 Results of calculation of S values from first cycle of grinding for 3 trials.Residence time is assumed to be 10 s.

The average *S* values in Table 4.5 are larger than the *S* values in Table 4.4. These values would have been even larger if the assumed grinding residence time was less than 10 seconds. Similar to Table 4.4, the *S* values in Table 4.5 decreases with smaller sieves.

b(i,j) or breakage distribution function is the percentage of ground material originally from sample *j* that goes to narrow size category of *i*. For example for material originally from sieve 2 (*j*=2), in order to calculate b value for destination sieve 3 (*i*=3), Equation 4.8 becomes:

$$b(3,2) = \frac{W(3,2)}{W_{12} - W(2,2)}$$

$$4.11$$

Substituting with data from Table 4.3:

$$b(3,2) = \frac{9.38}{65 - 5.82} = 0.1586$$
4.12

Table 4.6 lists b(i,j) values calculated from data in Table 4.3.

| | Origin sieve number, <i>j</i> | | | | | | |
|------------------------------------|-------------------------------|--------|--------|--------|--------|--------|--|
| Destination sieve number, <i>i</i> | 2 | 3 | 4 | 5 | 6 | 7 | |
| 2 | 0. | | | | | | |
| 3 | 0.1586 | 0. | | | | | |
| 4 | 0.2710 | 0.3486 | 0. | | | | |
| 5 | 0.2372 | 0.2832 | 0.4613 | 0. | | | |
| 6 | 0.2073 | 0.2313 | 0.3415 | 0.6343 | 0. | | |
| 7 | | 0.0964 | 0.1461 | 0.2825 | 0.7744 | 0. | |
| 8 | | | 0.0292 | 0.0553 | 0.1718 | 0.9283 | |
| 9 | | | | 0.0130 | 0.0251 | 0.0407 | |
| 10 | | | | | 0.0124 | 0.0133 | |
| 11 | | | | | | 0.0069 | |
| Pan | 0.1258 | 0.0405 | 0.0218 | 0.0146 | 0.0162 | 0.0107 | |
| Total | 0.9999 | 1.0000 | 0.9999 | 0.9997 | 0.9999 | 0.9999 | |

Table 4.6 Calculated breakage distribution functions, *b* values, dimensionless values for the ground particles from specified sieves for the first set of samples

Each b value is calculated as the percentage of the mass of particles coming to size divided by the total particles left the size due to grinding.

The objective is to study the distribution of the ground material to five sieves (sieves with mesh numbers: $3\frac{1}{2}$, 5,7,10 14) plus pan. All the sieves with bigger mesh numbers (smaller opening size) are eliminated. In other words it is assumed that the particles that go through these sieves are accumulated in the pan. Calculated new *b* values are summarized in Table 4.7.

Table 4.7 Results of calculated b(i,j) values for samples.

| Mesh No. | Sieve opening, mm | ìj | 2 | 3 | 4 | 5 | 6 | 7 |
|----------|-------------------|----|--------|--------|--------|--------|--------|--------|
| 31/2 | 5.66 | 2 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| 5 | 4.00 | 3 | 0.1586 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| 7 | 2.83 | 4 | 0.2710 | 0.3486 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| 10 | 2.00 | 5 | 0.2372 | 0.2831 | 0.4613 | 0.0000 | 0.0000 | 0.0000 |
| 14 | 1.41 | 6 | 0.2073 | 0.2313 | 0.3415 | 0.6343 | 0.0000 | 0.0000 |
| Pan | 0.00 | 7 | 0.1258 | 0.1136 | 0.1971 | 0.3656 | 1.0000 | 0.0000 |
Equation 4.3 with the estimated S and b values can be solved numerically to predict the particle size changes in time (McCabe et al., 1985).

The explicit form of Equation 4.3 is:

$$M(i,t+\Delta t) = [-S(i)M(i,t) + \sum_{j=1}^{i-1} M(j,t)S(j)b(i,j)]\Delta t + M(i,t)$$
4.13

For clarity M (mass based) indexes in Equation 4.13 are omitted. A set of 7 equations are solved simultaneously to predict the change of mass of material on each sieve based on time. Equation 4.13 was converted to MATLAB code (see Appendix IV).

Figure 4.3 shows the change of the mass of material retained on each sieve as a function of time. Grinding rates, *S*, are taken from batch grinding results in Table 4.4. The amount of material used in the simulation was 30 g of particles from the particles that passed through the sieve with a mesh number $\frac{1}{4}$ (opening 6.35 mm) and retained on the sieve with mesh number $\frac{3}{2}$ (opening 5.66 mm).



Figure 4.3 The change of mass of material as a function of time for grinding 30 g of material that passed through the sieve with a mesh number ¹/₄ and retained on the sieve with a mesh number 3¹/₂. Grinding rates, *S* values, are from data of batch grinding. (Table 4.4)

Figure 4.4 is similar to Figure 4.3 except the grinding rates, *S*, are calculated based on data from Table 4.5.



Figure 4.4 The change of mass of material based on time when feed is 30 g of material that passed through sieve with mesh number ¹/₄ and retained on sieve with mesh number 3¹/₂. Grinding rates, *S* values, are from data of first cycle of grinding.

4.5.1 Discussion

The tests include size reduction and particles fractionation, with the two processes discussed separately below.

4.5.1.1 Size reduction

An assumption that is made to solve the population balance equation is that particles belonging to each size category are ground independently. In solving the equations the interaction of the particles of different sizes is ignored. However in a batch grinder all of the particles from different size categories are in the grinder together and they may interfere each other during grinding.

It is expected that the composition of particles can be predicted at any time after the start of the grinding process. Actual measured weights of grinding 30 g of particles retained on

sieve mesh number $3\frac{1}{2}$ for 30 s and distributed to sieves $3\frac{1}{2}$, 5, 7, 10, 14 and pan, are extracted from Figure 4.3. These data are then compared with the measured weights of ground particles resulting from batch grind of materials on the sieve with mesh number $3\frac{1}{2}$ for 30 s grinding. Table 4.8 lists this comparison.

| Mesh No. | Actual weight | Calculated weight | Difference |
|----------|---------------|-------------------|-------------|
| | (g) | (g) | |
| 3 1/2 | 1.8 | 10.92 | 9.12 |
| 5 | 1.22 | 2.115 | 0.895 |
| 7 | 1.39 | 3.661 | 2.271 |
| 10 | 1.54 | 4.261 | 2.721 |
| 14 | 2.69 | 4.261 | 1.571 |
| Pan | 20.82 | 5.054 | -15.766 |
| Total | 29.46 | 30.272 | RSSRE=0.956 |

Table 4.8 Weight distribution of batch grind material of mesh number 3 ½ sample.

In Table 4.8, the second column and third column list the actual measured and calculated weights of material retained on each sieve after batch grinding for 30 s, respectively. Calculated weights are extracted from Figure 4.3 as follow. There are 6 curves in Figure 4.3. Each curve shows the change of retained material on the specified sieve as a function of time. On each curve the weight of material after 30 s of grinding is read and listed in the third column of Table 4.8. A comparison is made for the weights on the second and third column. Root square sum of relative errors (RSSRE) is calculated from Equation 4.14.

$$RSSRE = \frac{\sqrt{\sum_{i=1}^{n} \left(\frac{x_{mes,i} - x_{cal,i}}{x_{mes,i}}\right)^2}}{n}$$

$$4.14$$

In Equation 4.14, $x_{mes,i}$ is the i^{th} measured value. $x_{cal,i}$ is the i^{th} calculated value and n is the number of variables.

RSSRE calculated for measured and calculated data in Table 4.8 is 0.956. This means that the calculated data poorly matched the experimental ones. As seen from the data in columns two and three, the measured weights showed a much better grinding performance than what calculated by the model. The performance of the grinder is thus underestimated by the population balance model with *S* value determined from batch grinding.

The simulated results from the population balance equations using S values estimated from first cycle of grinding (Table 4.5) are shown in Figure 4.4. The actual measured data for 30 s of batch grinding is listed in the second column of Table 4.9. The calculated weights after 30 seconds of grinding were read from Figure 4.4 and summarized in the third column of Table 4.9.

| Mesh No. | Actual weight | Calculated weight | Difference |
|----------|---------------|-------------------|------------|
| | (g) | (g) | |
| 3 1/2 | 1.8 | 0.7339 | -1.066 |
| 5 | 1.22 | 0.7339 | -0.486 |
| 7 | 1.39 | 2.369 | 0.979 |
| 10 | 1.54 | 4.5113 | 2.971 |
| 14 | 2.69 | 8.164 | 5.474 |
| Pan | 20.82 | 13.6 | -7.22 |
| Total | 29.46 | 30.272 | RSSRE=1.49 |

Table 4.9 Results using S values obtained from first cycle of
grinding and residence time of 10 s

Last column in Table 4.9 lists the difference between actual measured and calculated weights. The root square sum of relative errors is (*RSSRE*): 1.49.

A comparison between grinding rates calculated based on batch grinding and first cycle of grinding shows that grinding rates from batch grinding are much smaller than grinding rates from first cycle of grinding (see Tables 4.4 and 4.5). One of the possible reasons for the difference between batch and first cycle of grinding is that feeding process in batch

grinding was not instantaneous. It took about 20 s to feed 30 g of material to the grinder. Therefore, not all of the 30 g material spent the same time in the grinder during the grinding. Figure 4.5 depicts a diagram that shows the residence time distribution of the feed material.



Figure 4.5 Residence time distribution of feed material in batch grinding

In Figure 4.5 the horizontal axis shows the time, and vertical axis shows the amount of material fed to the grinder. It shows that the residence time of the particles inside the grinder was not the same. The particles fed into the grinder at the beginning had a residence time of 30 s, and the particles fed into the grinder at the end of feeding only had a residence time of 10 s. This made the *S* values measured from batch grinding being smaller than the *S* values measured from first cycle of grinding.

A sensitivity analysis is performed in Section 4.6 to discuss the effect of the residence time on one cycle of grinding.

4.5.1.2 Particles fractionation based on size

Switchgrass is a heterogeneous fibrous material with a special structure which can be represented by a long cylinder with a small diameter. It has a special structure that along the stem some parts are hollow and some parts (nodes) are dense and solid. Figure 4.6 shows one typical stem of switchgrass.



Figure 4.6 A single switchgrass stem

During the process of grinding the hollow parts break easily and solid parts are more resistance to breakage. RoTap sieve shaker that is used for fractionating the different size of particles has a combined vertical and horizontal shaking movement. These two motions and the special shape of the particles affect the fractionating process. The sieves are made of woven wires with small thickness. If the material initially rests on the sieves in a way that its length is parallel to the surface of sieve, and the motion is only horizontal, none of the particles would pass the openings. RoTap sieve shaker has horizontal and vertical motion together, the vertical motion makes the particles change their position, and pass through the holes when their cross section becomes parallel to the surface of the sieves. This phenomenon makes the fractionating process in a way that the opening size is not a representative of particle size. In order to modify this process a new set of sieves have been designed. These sieves are made of thick aluminium sheets with circular holes. The new sieve shaker only provides horizontal shaking that prevents the particles from repositioning and passing through the holes by their diameter. More description and the results of preliminary tests with the new sieves and sieve shaker are explained in Section 5.2.1.

4.6 Sensitivity analysis

When the first cycle of grinding for measuring the b values was performed (Section 4.3.4), time was not recorded because the particles were fed into the grinder and discharged out instantly. For calculating S values from cycles of grinding the residence time of particles during the first cycle of grinding was assumed to be 10 s (Section 4.5).

In Table 4.9 a comparison was made between the actual measured weights from 30 s batch grinding and calculated weights after 30 s from Figure 4.4 (based on first cycle of grinding and 10 s residence time). In order to improve the agreement both actual measured and calculated data were revised. For actual measured data the ground particles collected from the first cycle of grinding of material originally from sieve mesh number $3\frac{1}{2}$ was chosen. The composition of feed particles size is listed in the second column of Table 4.3. The particles are fed to the grinder for the second cycle of grinding with no screen installed. The product particles were collected and analyzed according to their size by sieving with sieves with mesh numbers $3\frac{1}{2}$, 5, 7, 10, 14, and pan. The weights of particles retained on each sieve, actual weights, are summarized in the second column of Table 4.10.

| Mesh No. | Actual weight | S | Calculated weight | Diff. |
|----------|---------------|------------|-------------------|-------|
| | (g) | (s^{-1}) | (g) | |
| 31/2 | 2.67 | 0.182 | 0.52 | 2.15 |
| 5 | 4.08 | 0.169 | 2.30 | 1.78 |
| 7 | 9.97 | 0.139 | 9.11 | 0.86 |
| 10 | 11.93 | 0.109 | 14.98 | -3.05 |
| 14 | 16.62 | 0.073 | 14.49 | 2.13 |
| Pan | 17.86 | 0.058 | 18.68 | -0.83 |
| RSSRE | | | | 0.16 |

Table 4.10 Results using *S* values obtained from first cycle of grinding and a residence time of 5 s

It is assumed that the residence time for the first cycle of grinding is 5 s. A new series of S values were then calculated based on the results from the first cycle of grinding with a residence time of 5 s. These new S values are listed in the third column of Table 4.10. The composition of particles fed into the grinder for second cycle of grinding is known from Table 4.3. The particles retained on each sieve are treated independently and using the correspondent S and b values, the new weights are calculated and listed in the fourth

column of Table 4.10. The root square sum of relative errors (*RSSRE*) is calculated to be 0.16 based on Equation 4.13, which shows that the population balance model can give a fairly good agreement with the experimental data when the *S* and *b* values are estimated accurately and consistently. The results in Table 4.10 also shows that the *S* and *b* values estimated from individual particles size interval without the consideration of the interaction between particles of different sizes in the grinder can give a reasonable prediction for the first cycle of grinding when there is minimum interaction among particles of different sizes. On the other hand, there is a very strong interaction among particles of different size in the batch grinding when all particles stayed in the grinder over a certain period of grinding time. Therefore, population balance model with the *S* and *b* values estimated with the particle-particle interactions neglected fails to give a good prediction of the results.

Chapter 5 Conclusions and Future Work

5.1 Conclusions

Cycles of grindings were performed for size reduction of switchgrass with a grinder. Six cycles of grinding were performed for three uniform sizes of precut stems of switchgrass. Different probability distribution functions were investigated to define which one fits best to the data. Particle size distribution is an important property for a group of particles. It also shows the effectiveness of the grinding system (Pasikatan et al., 1999). Log-normal and Weibull distribution functions were tested to examine which one describes the results best. It was shown that Weibull distribution is more flexible and it fits to the data very well. Once the method to test different probability distribution functions is established it can be applied for different biomass and grinders. When the probability distribution function function for each set of biomass and grinders is defined the optimum one for each conversion process can be chosen.

Population balance was applied to biomass grinding. Two grinding parameters S (grinding rate, s⁻¹) and b (breakage distribution function, dimensionless) were defined and obtained experimentally from batch and first cycle of grinding using switchgrass as the material. Size fractionation was performed by a Ro-Tap sieve shaker.

Population balance equations with experimentally determined S and b values were applied for the prediction of grinding feed particles with a narrow size distribution. These feed particles were selected based on the sieving process. Particles retained on each sieve are considered as particles having a narrow size distribution between upper sieve size and size of the sieve that they are retained on. The equations were solved using the Euler method to predict the accumulation and depletion of particles size with time.

The predicted weight distribution of ground particles at different sieve sizes for the first cycle of grinding was found to be in good agreement with the experimental data from the second cycle of grinding, when both *S* and *b* values obtained from the cycles of grinding data were used in the simulation. However, the model prediction using *S* values obtained form batch grinding severely underestimated the grinding performance in the batch grinding process, likely due to the non-uniform residence time distribution of grinding materials in the grinder and the strong interaction among particles of different sizes.

5.2 Proposed future research

Following future researches are suggested to improve the modeling of the grinding process and to optimize the process of size reduction and fractionation of biomass for biofuel production.

5.2.1 Improved sieving method

As the particles of switchgrass have a long cylindrical shape, sieve shakers that are designed for spherical or cubical particles are not efficient for size fractionation of these particles. For this reason a new sieve shaker was designed based on ASAE S424 (2001). The proposed screens and sieve shaker in ASAE S424 (2001) was intended for determining the particle size distribution of chopped forage materials. The new horizontal shaker consists of a set of square-hole screens having widths of 406 mm and lengths of 565 mm with different thicknesses. The shaker oscillates the screen stack in a horizontal plane. The new design is based on the same idea but is intended for the laboratory use. It has a set of thick aluminium screens of 30 cm in diameter with circular holes. The specifications of screens are summarized in Table 5.1.

| Screen no. | Hole diameter | Screen thickness | Open area |
|------------|---------------|------------------|-----------|
| | cm | cm | % |
| 1 | 4.8 | 2.6 | 35.98 |
| 2 | 3.2 | 2.0 | 33.00 |
| 3 | 1.6 | 0.953 | 35.65 |
| 4 | 0.8 | 0.635 | 32.45 |
| 5 | 0.4 | 0.3 | 32.77 |
| Pan | | | |

Table 5.1 The specifications of the screens in a horizontal sieve shaker

In Table 5.1 the first column lists the screen numbers. The second column is the diameter of the holes. The screen thicknesses are listed in the third column. The percentage of open area in each screen is summarized in the fourth column. The shaker is a Retech

model AS 400. The base plate performs horizontal circular motions with a radius of 15 mm. The speed of 50 to 300 rpm can be electronically controlled. The base plate is driven by a 125 Watt motor.

In an attempt to perform a preliminary test, switchgrass (collected as round bale from a farm in Manitoba) was passed through the grinder once. Since there was no screen installed in the grinder, the ground material left the grinder immediately and collected into a container. All the ground material was put in a big tray, mixed thoroughly and divided into four samples. One of these representative samples was weighed (64 g), and was put on the upper most sieve of the sieve shaker. The speed of the horizontal shaking was initially set at 130 rpm. No motion of particles was observed. Shaking rate was increased in 10 rpm intervals. When the shaking speed was set to 160 rpm particles started to move. Time of shaking was set at 5 minutes. Material retained on each sieve was weighed with the results summarized in Table 5.2.

| Screen no. | Hole diameter | Weight of particles on |
|------------|---------------|------------------------|
| | cm | each sieve, g |
| 1 | 4.8 | 13.15 |
| 2 | 3.2 | 2.98 |
| 3 | 1.6 | 15.12 |
| 4 | 0.8 | 15.95 |
| 5 | 0.4 | 11.48 |
| Pan | | 3.52 |

 Table 5.2 Preliminary results of sieving switchgrass using new sieve shaker.

The particles retained on each sieve were checked by visual observation. No separation of particles by their lengths was achieved. In order to evaluate the performance of sieving with this new sieving device two series of tests are suggested:

 160 rpm was the first speed of shaking at which particles started to move. Preliminary results were collected at this speed of shaking. Sieving in speed from 160 to 300 rpm is suggested. Too high a rate causes mixing and dynamic movement of particles. There is an optimum range of shaking rates that should be identified by numerous tests and comparing of results. 2. Time of sieving was set at 5 minutes for the preliminary tests. Different sieving times should be tested and an optimum time of sieving should be set.

5.2.2 Improvement of the method of feeding

For the first tests the material was fed into the grinder by hand. It raised problems in both series of tests:

- 1. During the grinding process the grinder worked alternately empty then full of particles. As a result all the particles were not uniformly ground.
- 2. In order to measure an accurate grinding rate (*S*) it is important that all the particles have uniform residence time in the grinder.

The vibratory feeder was installed to improve the feeding process, with the intention that all particles are fed uniformly into the grinder. The feeder is worked properly for particles smaller than those retained on the sieve with a mesh no. 14. However the larger particles tend to tangle together, and dispersed at the point where they left the feeder tray to enter the grinder. There is thus a need to change the hopper that is attached to the grinder. Changing the standard hopper to a long stock hopper that can direct every size of particles to the grinder chamber is suggested.

The feeder tray is narrow and can only handle 45 g of particles of a size retained on mesh no. 14 (the maximum particle size that can be fed into the grinder without problems during the test). It is suggested that a different feeding system be installed to ensure the uniform feeding of particles into the vibratory feeder tray.

5.2.3 Correlation of grinding rates (S), breakage distribution functions (b) and Weibull constants with particle size

Grinding rate (*S*), breakage distribution function and two constants of the Weibull distribution are characteristics of particles. They depend on particle size, biomass species and size reduction method. In this research the dependence of grinding rate, calculated with assumed 5 s of residence time, on the sieve opening size has been investigated. The results are summarized in Figure 5.1. The correlated logarithm equation is also shown in the Figure 5.1. The R^2 or coefficient of determination for the correlation of the grinding rate with sieve size opening is equal to 0.98.



Figure 5.1 Measured grinding rate versus sieve opening size data and a simple logarithmic correlation.

5.2.4 Optimizing the method for monitoring energy consumption

Switchgrass particles have a very low bulk density. The amount of material fed into the grinder for energy monitoring using the existing feeding system was not sufficient to show significant changes on the energy consumption graph. Modification of the feeding system that was proposed in Section 5.2.3 would help to feed material more uniformly into the grinder and ensure all the material has the same residence time in the grinder.

5.2.5 Simulating the process of size reduction with rate of reaction

Simulating the process of size reduction with rate of reaction approach is suggested. Rate of reaction is defined as an equation which is a combination of change of concentration of reactants and reaction activation energy. Size reduction can be considered as a reaction in which particles with different sizes are reactants and energy consumption of size reduction is similar to the activation energy.

5.2.6 Performance of the grinder installed with screens

All the methods studied in this research were conducted in a grinder either without screen or with a blocked outlet (batch grinding). When screens with different sized openings are installed in the grinder the process is similar to a chemical process with a combined reactor and separator, which retains the oversized particles and rejects the undersized particles. In this case the oversized particles stay in the grinder until they are ground to sufficiently small size to pass the screen. Modeling of a grinder with screens based on this idea should be considered for future study.

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Appendix I

AI.1 The effect of time of sieving on the material retained on each sieve

In order to choose an optimum time for sieving, a series of tests are performed during the second cycle of grinding for 25 mm precut particles. The time of sieving is set at 5, 10, 15, and 20 min. The particles retained on each sieve is weighed and recorded. The weight fractions are calculated with results are depicted in Figure AII.1.



Figure AII .1 The change of retained particles on each sieve versus time of sieving

In Figure AII.1 each curve shows the change of weight fraction of particles on each sieve. It is shown that the change of particles on each sieve is not significant when the time of sieving increased from 10 minutes. 10 minutes sieving is thus chosen as the optimum time for sieving.

Appendix II

AII.1 Length and maximum diameter of the particles retained on various sieves

A representative sample of ground switchgrass was taken from each sieve. The sampling method was as follow. All the particles that retained on one sieve were spread on a tray and mixed together. The particles were divided to four or six groups and one group was chosen as a representative sample. Length and maximum diameter of the particles of each sample was measured by a calliper. The selected sieves had the openings of 1.41, 2, 2.83, 4, and 5.66 mm (sieves with mesh numbers $3\frac{1}{2}$, 5, 7, 10, and 14).

The raw data for length measurements is summarized in Table AI.1. In Table AI.1, the first column lists the range of length (mm), the second to fifth columns show the number of particles belongs to each length range for particles retained on sieve with specified opening. Mean and standard deviation of the length of the particles are also listed in the last two rows of Table AI.1. The frequency of the particles belong to each length category is depicted in Figure AI.1. The particles retained on sieve with opening of 1.41 mm, have a mean length of 53 mm. This means that the sieve opening is not a representative of the particles length.



Figure AI.1 Distribution of the length of the particles retained on sieve with specified opening

| | | Si | ieve opening, m | m | |
|--|-------|-------|-----------------|-------|-------|
| Range of length, mm | 1.41 | 2 | 2.83 | 4 | 5.66 |
| ≤ 5 | 12 | 1 | | | |
| 5 <l≤10< td=""><td>22</td><td>5</td><td></td><td></td><td></td></l≤10<> | 22 | 5 | | | |
| 10 <l≤15< td=""><td>21</td><td>16</td><td>3</td><td>1</td><td></td></l≤15<> | 21 | 16 | 3 | 1 | |
| 15 <l≤20< td=""><td>17</td><td>20</td><td>5</td><td>3</td><td></td></l≤20<> | 17 | 20 | 5 | 3 | |
| 20 <l≤25< td=""><td>6</td><td>17</td><td>12</td><td>5</td><td>1</td></l≤25<> | 6 | 17 | 12 | 5 | 1 |
| 25 <l≤30< td=""><td>3</td><td>17</td><td>15</td><td>9</td><td>0</td></l≤30<> | 3 | 17 | 15 | 9 | 0 |
| 30 <l≤35< td=""><td>2</td><td>13</td><td>10</td><td>7</td><td>3</td></l≤35<> | 2 | 13 | 10 | 7 | 3 |
| 35 <l≤40< td=""><td>1</td><td>12</td><td>9</td><td>10</td><td>6</td></l≤40<> | 1 | 12 | 9 | 10 | 6 |
| 40 <l≤45< td=""><td>1</td><td>2</td><td>4</td><td>7</td><td>8</td></l≤45<> | 1 | 2 | 4 | 7 | 8 |
| 45 <l≤50< td=""><td></td><td>2</td><td>6</td><td>12</td><td>7</td></l≤50<> | | 2 | 6 | 12 | 7 |
| 50 <l≤55< td=""><td></td><td>0</td><td>3</td><td>10</td><td>2</td></l≤55<> | | 0 | 3 | 10 | 2 |
| 55 <l≤60< td=""><td></td><td>1</td><td>3</td><td>12</td><td>12</td></l≤60<> | | 1 | 3 | 12 | 12 |
| 60 <l≤65< td=""><td></td><td>1</td><td>2</td><td>7</td><td>8</td></l≤65<> | | 1 | 2 | 7 | 8 |
| 65 <l≤70< td=""><td></td><td>0</td><td>0</td><td>4</td><td>4</td></l≤70<> | | 0 | 0 | 4 | 4 |
| 70 <l≤75< td=""><td></td><td>1</td><td>2</td><td>3</td><td>1</td></l≤75<> | | 1 | 2 | 3 | 1 |
| 75 <l≤80< td=""><td></td><td></td><td></td><td>1</td><td>2</td></l≤80<> | | | | 1 | 2 |
| 80 <l≤85< td=""><td></td><td></td><td></td><td>4</td><td>0</td></l≤85<> | | | | 4 | 0 |
| 85 <l≤90< td=""><td></td><td></td><td></td><td>0</td><td>0</td></l≤90<> | | | | 0 | 0 |
| 90 <l≤95< td=""><td></td><td></td><td></td><td>0</td><td>2</td></l≤95<> | | | | 0 | 2 |
| 95 <l≤100< td=""><td></td><td></td><td></td><td>0</td><td></td></l≤100<> | | | | 0 | |
| 100 <l≤105< td=""><td></td><td></td><td></td><td>1</td><td></td></l≤105<> | | | | 1 | |
| Mean of the length | 13.61 | 24.81 | 34.38 | 49.29 | 53.57 |
| Standard deviation | 7.95 | 11.88 | 13.50 | 20.60 | 13.83 |

Table AI.1 Length distribution of particles on sieves with different openings

The raw data for maximum diameter measurements is summarized in Table AI.2. In Table AI.2, the first column lists the range of maximum diameter, the second to fifth columns show the number of particles belongs to each maximum diameter range for particles retained on sieve with specified opening. Mean and standard deviation of the maximum diameter of the particles are also summarized in the last two rows of Table AI.1. The frequency of the particles belong to each maximum diameter range is depicted

| | Sieve openin | ıg, mm | | | |
|---|--------------|--------|------|------|------|
| Range of Maximum Diameter mm | 1.41 | 2 | 2.83 | 4 | 5.66 |
| ≤ 0.2 | 2 | | | | |
| 0.2 <md≤0.4< td=""><td>0</td><td>1</td><td></td><td></td><td></td></md≤0.4<> | 0 | 1 | | | |
| 0.4 <md≤0.6< td=""><td>3</td><td>5</td><td></td><td></td><td></td></md≤0.6<> | 3 | 5 | | | |
| 0.6 <md≤0.8< td=""><td>0</td><td>4</td><td></td><td>3</td><td></td></md≤0.8<> | 0 | 4 | | 3 | |
| 0.8 <md≤1< td=""><td>7</td><td>15</td><td></td><td>2</td><td></td></md≤1<> | 7 | 15 | | 2 | |
| 1 <md≤1.2< td=""><td>8</td><td>14</td><td></td><td>1</td><td></td></md≤1.2<> | 8 | 14 | | 1 | |
| 1.2 <md≤1.4< td=""><td>25</td><td>9</td><td>4</td><td>6</td><td></td></md≤1.4<> | 25 | 9 | 4 | 6 | |
| 1.4 <md≤1.6< td=""><td>12</td><td>11</td><td>1</td><td>8</td><td>2</td></md≤1.6<> | 12 | 11 | 1 | 8 | 2 |
| 1.6 <md≤1.8< td=""><td>10</td><td>9</td><td>2</td><td>3</td><td>1</td></md≤1.8<> | 10 | 9 | 2 | 3 | 1 |
| 1.8 <md≤2< td=""><td>8</td><td>12</td><td>8</td><td>4</td><td>1</td></md≤2<> | 8 | 12 | 8 | 4 | 1 |
| 2 <md≤2.2< td=""><td>6</td><td>7</td><td>10</td><td>8</td><td>1</td></md≤2.2<> | 6 | 7 | 10 | 8 | 1 |
| 2.2 <md≤2.4< td=""><td>2</td><td>13</td><td>7</td><td>7</td><td>6</td></md≤2.4<> | 2 | 13 | 7 | 7 | 6 |
| 2.4 <md≤2.6< td=""><td></td><td>2</td><td>4</td><td>6</td><td>4</td></md≤2.6<> | | 2 | 4 | 6 | 4 |
| 2.6 <md≤2.8< td=""><td></td><td>4</td><td>8</td><td>7</td><td>5</td></md≤2.8<> | | 4 | 8 | 7 | 5 |
| 2.8 <md≤3< td=""><td></td><td></td><td>12</td><td>9</td><td>6</td></md≤3<> | | | 12 | 9 | 6 |
| 3 <md≤3.2< td=""><td></td><td></td><td>4</td><td>6</td><td>7</td></md≤3.2<> | | | 4 | 6 | 7 |
| 3.2 <md≤3.4< td=""><td></td><td></td><td>6</td><td>4</td><td>6</td></md≤3.4<> | | | 6 | 4 | 6 |
| 3.4 <md≤3.6< td=""><td></td><td></td><td>2</td><td>4</td><td>3</td></md≤3.6<> | | | 2 | 4 | 3 |
| 3.6 <md≤3.8< td=""><td></td><td></td><td>2</td><td>4</td><td>6</td></md≤3.8<> | | | 2 | 4 | 6 |
| 3.8 <md≤4.< td=""><td></td><td></td><td>2</td><td>2</td><td>2</td></md≤4.<> | | | 2 | 2 | 2 |
| 4 <md≤4.2< td=""><td></td><td></td><td></td><td>3</td><td>1</td></md≤4.2<> | | | | 3 | 1 |
| 4.2 <md<u>≤4.4</md<u> | | | | 5 | 3 |
| Mean of the MD | 1.44 | 1.56 | 2.62 | 2.69 | 3.07 |
| Standard deviation of MD | 0.48 | 0.63 | 0.71 | 1.13 | 0.75 |

Table AI.2 Maximum diameter distribution of particles on sieves with different openings

in Figure AI.2. The particles retained on sieve with opening of 1.41 mm, has a mean maximum diameter of 1.44 mm comparing with the sieve opening of 1.41 means that the sieve opening is a good representative of the particles maximum diameter. The relation weakened as the sieve opening increases. For the sieve opening of 5.66 mm the mean maximum diameter of the particles is 3.07 mm.



Figure AI.2 Distribution of the maximum diameter of the particles retained on sieve with specified opening

Appendix III

AIII.1 The data from two duplicates of samples for the measurement of *b* values

Table AIII.1 lists the collected data from the second set of samples.

| Weight of material, g | | | | | | | |
|--------------------------|---------------------|-------|-------|-------|-------|-------|--|
| | Origin sieve number | | | | | | |
| Destination sieve number | 2 | 3 | 4 | 5 | 6 | 7 | |
| 2 | 6.01 | | | | | | |
| 3 | 9.30 | 9.82 | | | | | |
| 4 | 15.83 | 18.98 | 19.70 | | | | |
| 5 | 14.10 | 15.52 | 20.67 | 29.30 | | | |
| 6 | 12.50 | 12.65 | 15.40 | 22.20 | 40.30 | | |
| 7 | | 5.30 | 6.33 | 9.90 | 18.21 | 46.50 | |
| 8 | | | 1.27 | 2.10 | 4.12 | 17.30 | |
| 9 | | | | 0.45 | 0.65 | 0.70 | |
| 10 | | | | | 0.33 | 0.24 | |
| 11 | | | | | | 0.13 | |
| Pan | 7.10 | 2.29 | 1.3 | 0.45 | 0.45 | 0.15 | |

Table AIII.1 Weight distribution of the ground particles from specified sieves for the second set of samples

In this table the third row shows the sample numbers that the feed material is coming from, and the first column shows the sample number of the distributed ground material after sieving. The feed samples were ground and fractionated to five sieves plus pan. Table AIII.2 lists the collected data from the third set of samples.

| Weight of material, g | | | | | | | |
|--------------------------|---------------------|-------|-------|-------|-------|-------|--|
| | Origin sieve number | | | | | | |
| Destination sieve number | 2 | 3 | 4 | 5 | 6 | 7 | |
| 2 | 6.05 | | | | | | |
| 3 | 9.25 | 10.40 | | | | | |
| 4 | 15.90 | 18.85 | 20.02 | | | | |
| 5 | 13.90 | 15.65 | 20.50 | 29.27 | | | |
| 6 | 12.34 | 12.45 | 15.70 | 22.16 | 40.45 | | |
| 7 | | 5.40 | 6.09 | 9.50 | 18.10 | 46.30 | |
| 8 | | | 1.20 | 2.11 | 4.10 | 17.20 | |
| 9 | | | | 0.43 | 0.57 | 0.72 | |
| 10 | | | | | 0.22 | 0.20 | |
| 11 | | | | | | 0.12 | |
| Pan | 7.40 | 2.11 | 1.10 | 0.60 | 0.35 | 0.12 | |

Table AIII.2 Weight distribution of the ground particles from specified sieves for the second set of samples

Appendix IV

MATLAB CODES

Matlab codes for solving the set of explicit form of population balance, Equation (4.13):

```
%Input the initial values
s=[0.063 0.049 0.057 0.046 0.053 0.0];
x1(1,1)=6.00; x1(1,2)=0; x1(1,3)=0; x1(1,4)=0; x1(1,5)=0;
x1(1,6)=0;
deltabi=[0 ,0.158607,0.271052,0.237234,0.20730,0.125803;
                    ,0.3486 ,0.283174,0.231305,0.11369 ;
         0,0
                    ,0
                             ,0.46135,0.341501 ,0.1971 ;
         0,0
         0,0
                    ,0
                             ,0
                                     ,0.63436 ,0.3656 ;
                             ,0
         0,0
                    ,0
                                     ,0
                                                ,1
                                                          ];
%Initialization for calcuting the x matrix
deltat=5;
t=0;1=0;
%Calculating the x matrix for the time intervals of 30 second and till
500
%intervals
for timecounter=2:200
    for n=1:6
        a=0;
        for sigmacounter=1:n-1
            a=a+x1(timecounter-
1, sigmacounter)*s(sigmacounter)*deltabi(sigmacounter,n);
        end
        x1(timecounter,n)=x1(timecounter-1,n)*(1-s(n)*deltat)+deltat*a;
        b=x1(timecounter,n);
    end
end
t(1)=0;
for n=2:50
    t(n)=t(n-1)+5;
end
%Ploting the variation of the amount of material in each fraction based
%to time
for i=1:6
    for j=1:50
       y5(j)=x1(j,i);
    end
   plot(t,y5);
    %text(t(100+10*i),y1(100),'\leftarrow x');
   hold on
end
xlabel('Time(s)');
ylabel('Mass(g)');
```