Moisture and Storage Time Effects on Grinding Characteristics of Loblolly Pine Woodchips

by

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Abstract

In the processing of biomass to bio-products, grinding is unavoidable. Grinding is used to produce particulate materials and enhance handling, storage, and conversion processes. Performance of grinding operation is usually evaluated based on specific grinding energy requirements and physical properties of resulting grinds. The objective of this work was to quantify the effects of moisture and storage time on the specific grinding energy requirement and some physical properties of grinds. Loblolly pine woodchips were ground using a hammer mill with two screen sizes (3.175 mm and 6.350 mm). Moisture content of woodchips was adjusted to 12%, 20%, 30%, 40%, and 50% for storage times of 0 month, 2 month, and 4 month. To further investigate the effect of moisture on loblolly pine wood, the specific toughness and bending stress of loblolly wood was measured using three point bending test. Also, the bulk density, aspect ratio, flow index, particle size distribution, specific grinding energy requirements, and minimum drying energy requirements were measured and analyzed for different drying-grinding sequences. Results indicated that specific grinding energy ranged from 54.341 kJ/kg d.b. to 818.818 kJ/kg d.b.. Storage time did not have significant effect on grinding difficulty. However, for both screen sizes, grinding difficulty increased with increase in moisture content to a threshold of 30%. Further increase in moisture content resulted in decrease in grinding difficulty for screen size 6.350 mm. Although this was not obvious for screen size 3.175 mm, a reduction in slope was observed above the threshold moisture content. Bond model produced the best fit (r^2 = 0.984) when the three grinding equations were fitted. Geometric mean diameter of grinds initially increased with increase in moisture content of woodchips from 12% to 20%, then reduced with further increase to 50%. It was also significantly affected ($p < 0.05$) by storage time and hammer mill screen size. The mean oven-dried bulk density decreased with increase in moisture content of feedstock and with increase in screen size. However, an interaction was observed between the moisture content of feedstock and hammer mill screen size. The three point bending test showed that jaggedness in the force-displacement curve disappeared with increase in moisture content. The toughness of loblolly pine wood increased with increasing moisture as well as with increasing tree radius and with decreasing tree height. Although, bending stress followed the same trend with tree height and radius, it reduced as moisture content increased. For the drying-grinding study, the highest oven-dried bulk density of grinds was 267.083 kg/m^3 and lowest was 97.947 kg/m^3. Generally, high aspect ratio, with mean values ranging from 4.30 to 6.36, was recorded. The specific grinding energy requirement varied from 211.639 kJ/kg d.b. to 818.162 kJ/kg d.b. With the consideration of moisture loss during grinding, there was a trade-off between specific grinding energy requirement and minimum drying energy requirement. Two-phase grinding had lower cumulative energy consumption compared to one-phase grinding.

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Chapter 1: Introduction

The finite nature of fossil fuel, the impact of fossil fuel utilization on the environment, and oil price fluctuations have necessitated the need to replace some of the fossil fuel and other fossil-based products consumed in the world (Cherubini and Stromman, 2011). Alternative and renewable energy sources, such as bioenergy, solar energy, wind energy, geothermal energy, and hydro-power meet this need. Bioenergy is of special interest because it is the only renewable resource that can directly produce carbon-based fuels, chemicals and products that are currently obtained from crude oil (Soetaert and Vandamme, 2009). Other benefits associated with bioenergy include regional energy independence, environmental friendliness, and employment opportunities (Thomas, 2007; Pacheco, 2006).

Bioenergy are derived from biological materials (often called biomass) such as agricultural residues and wastes, energy crops, forest resources, and algae. An important biomass source is loblolly pine (*Pinus taeda*) which is a common forest tree grown in the United States and indigenous to the southeastern United States. According to Taylor et al. (2012), loblolly pine grows on about 35 million acres of land in the southeastern region of the United States. Rapid growth, high yield, and high response to silvicultural management practices make loblolly pine a suitable bioenergy resource (Baker and Langdon, 2008).

Several unit operations are needed to preprocess and prepare biomass before they are fed into bioenergy conversion plants. These unit operations include transportation, storage, drying, particle size reduction, and densification (Kokko et al., 2012; Tabil et al., 2011). It is essential to note that the importance and combination of these processes are predicated on the target products. For instance, drying of biomass is not necessary in the production of bio-ethanol through fermentation while it is mandatory in the production of bio-oil through pyrolysis. Nevertheless, particle size reduction of biomass is required for all biomass conversion processes (Sokhansanj et al., 2006; Naimi et al., 2013).

Size reduction of biomass produces smaller particle sizes, thus affecting the physical characteristics of biomass (such as surface area to volume ratio, bulk density, energy density, pore size and number of particle to particle contact point of biomass—Miao et al., 2011; Ghorbani et al., 2010) and post-grinding unit operations (such as handling, storage, transportation, and conversion—Tabil et al., 2011). Size reduction of biomass is necessary to maximize biomass conversion efficiency (Mani et al., 2006; Igathinathane et al., 2009). However, the optimum particle size required for the various conversion processes are different, as shown in Table 1.1. Examples of size reduction operations include chipping, cutting, crumbling, chopping, milling, or grinding depending on the extent of size reduction achieved (Fellows, 2000). Grinding involves the production of particulate or powder like materials that are usually less than 3 mm in size.

Grinding is energy intensive, with grinding energy constituting a significant percentage of the total energy consumed in the production of bioenergy. For example, according to the US Department of Energy, the energy consumed during biomass comminution constitutes about one-third of the total energy requirement in the production of bioethanol (Probst et al., 2013, 1993; Bitra et al., 2008; Cadoche and Lopez, 1989). To ensure sustainability and cost effectiveness of biomass conversion processes, it is imperative to minimize the energy dissipated during the creation of biomass grinds.

Table 1.1. Examples of particle sizes of biomass used in biomass conversion processes.

Process/Operation	Particle Size Required (mm)	Reference
Gasification of corn stover	< 6.35	Kumar et al., 2009
Pyrolysis of loblolly pine	< 0.30	Mahadevan et al., 2015
Hydropyrolysis of rapeseed	< 0.22	Onay et al., 2006
Pyrolysis of hardwood	0.10 – 1.00	Dobele et al., 2007
Fermentation of redcedar	0.5	Ramachandriya et al., 2014
Fermentation of corn stover	2.00 – 10.00	Ohgren et al., 2006
Circulating fluidized bed pyrolysis	< 6.00	Bridgwater et al., 1999
Entrained flow pyrolysis	< 2.00	Bridgwater et al., 1999
Hydrolysis	0.20 – 2.00	Sun and Cheng, 2002

Hammer mill is one of the most commonly used grinding equipment because it is relatively cheap, versatile, low maintenance, and has large throughput. Hammer mill reduces the particle size of biomass by impact force. Particles size distribution of grinds produced by hammer mill have wide spread which makes them suitable for pelletizing, cubing, and briquetting (Mani et al., 2003). Other important grinding equipment are knife mill and ball mill.

Particle size distribution (geometric mean diameter and geometric standard deviation) of resulting grinds is one of the methods used to quantify the effectiveness of the grinding operation. Particle size distribution also influences the efficiency of post-grinding unit operations (Adapa et al., 2011; Mani et al., 2004). Factors that affect the particle size distribution of grinds include moisture content, grinding equipment operating variables, and size reduction ratio (Armstrong et al., 2007; Ghorbani et al., 2010). Several studies have documented that storage duration changes the properties of biomass (Nurmi, 1999; Brand et al., 2011; Thornqvist, 1985), but no study has investigated the relationship between storage time and grinding effectiveness of biomass.

The overall goal of this study was to understand the contributions of moisture content, storage time, and hammer mill screen size to loblolly pine woodchips grinding effectiveness. To achieve this goal, the following specific objectives were carried out:

i. Investigate the effect of storage time on volatile matter, energy, and ash contents of loblolly pine woodchips.
ii. Understand the contribution of moisture content, storage time, and hammer mill screen size to grinding moisture loss, bulk density of grinds, particle size distribution of grinds, and specific grinding energy requirement.
iii. Quantify the effect of moisture content, tree height, and tree radius on the toughness and bending stress of wood chips through three point bending test.
iv. Investigate the influence of drying and grinding sequences on specific grinding energy and physical properties of resulting grinds.

Chapter 2: Literature Review

2.1 Biomass versus Fossil Products

Crude oil is a finite resource because the rate of its consumption evidently far surpasses the rate of its production from the decomposition of the remains of plants and animals that existed millions of years ago. The prediction is that at the current consumption rate, crude oil will run out by the end of this century (Jolley, 2006; Li et al., 2009). Discoveries of new fossil fuel deposits have nevertheless continue diminish the importance of this prediction. On an average, 12 billion barrels of fossil fuel are discovered yearly across the world. In 2011, 10 different sedimentary basins were discovered in Brazil, Iran, Mozambique, Cyprus, Azerbaïdjian, United States, and Iraq. Among other significant discoveries are the giant oil field Tupi (Lula, Brazil) in 2006; the Levant Basin, Mediterranean in 2010; and Rovuma Basin, East Africa in 2011 (IFPEN, 2013).

While the future availability of crude oil remains uncertain and controversial, the continuous utilization of fossil-based products (such as fuel, chemicals, and polymers) produces carbon dioxide, the primary greenhouse gas responsible for warming up the earth. According to IEA (2008), the transportation sector alone consumes about 60% of the world's total fossil fuel and contributes about 23% of the global carbon dioxide emission. Global warming possess an immense threat to the future of our fragile environment. In addition, the price of fossil-based fuels remains unstable (McKendry, 2002a; Frombo et al., 2009). These clearly necessitate the inevitability of the need for a fossil fuel free future.

Bio-based products is favored as a replacement for fossil-based products because of their following attributes: environmental friendliness, foreign crude oil independence, power sufficiency, the requirement of little or no modification to existing energy systems, and employment opportunities (Maghrour, 2009; Thomas, 2007; Pacheco, 2006). In 2012, about 7.1% of the total fuel consumed (13.8 billion gallons) in the United States transport sector was obtained from biofuels (mainly ethanol and biodiesel) (USDA, 2015).

2.2 Biomass Conversion

The processes used to convert biomass to fuels, power, chemicals, and polymers from biomass typically follows one of two routes: thermochemical and biochemical (Figure 2.1). The thermochemical routes utilize temperature and/or pressure to breakdown the biomass while the biochemical routes focus on using biological processes and microbes in fermenting biomass, usually into fuels such as ethanol and butanol (NREL, 2009).

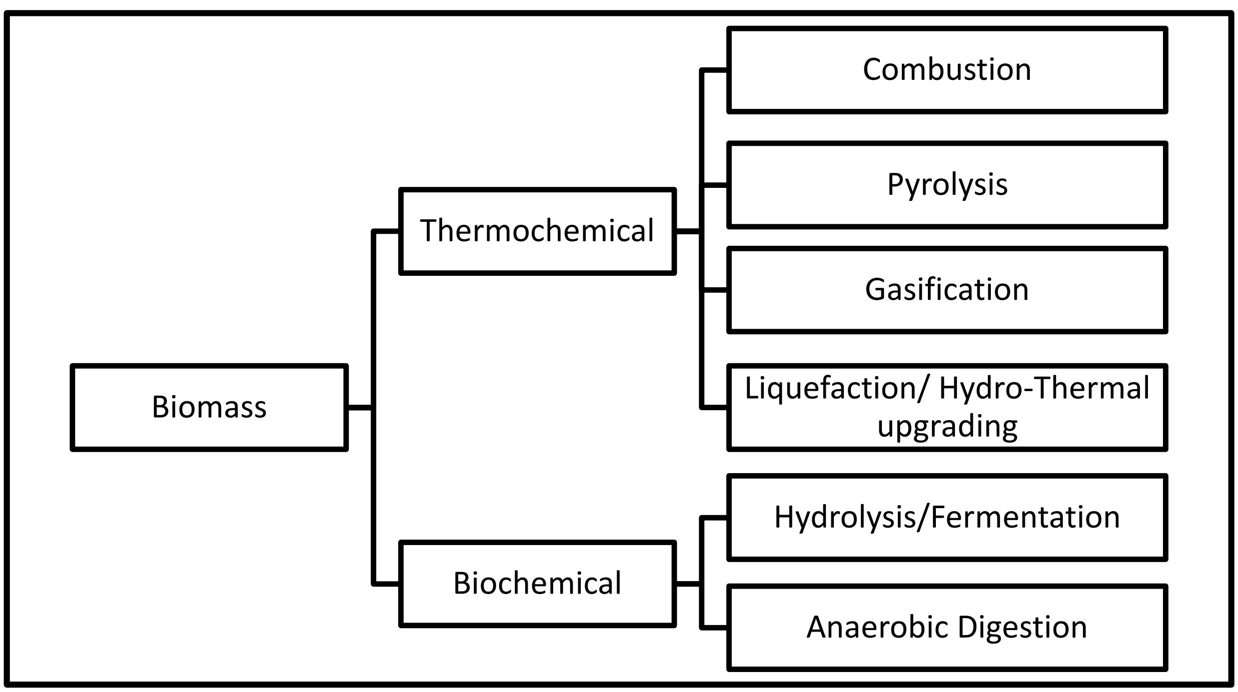

Figure 2.1. Biomass conversion routes.

The choice of biomass conversion route is dependent on the desired end products, biomass type, and biomass properties. The direct combustion of biomass in the presence of abundant oxygen supply is the oldest thermochemical process, and it is utilized in the production of heat and power (Demirbas, 2004). Pyrolysis is aimed at the production of char (bio-char), and liquid (bio-oil) from lignocellulosic biomass by heating the biomass in the absence of oxygen while gasification is predominantly focused on the production of synthesis gas (carbon monoxide, carbon dioxide, hydrogen, methane, and nitrogen). Biomass is thermally broken down in the presence of limited oxygen supply during gasification (McKendry, 2002c). The synthesis gases (syngas) produced from gasification process may be reacted to form transportation fuels through Fischer-Tropsch process. Liquefaction is usually used to convert wet biomass to liquid hydrocarbon at low temperature and high pressure (McKendry, 2002b).

Sugars present in lignocellulosic biomass are converted into liquid products as a result of the action of microbes (mostly yeast) in a fermentation process. This process is usually preceded by the hydrolysis of biomass into fermentable sugar. Bio-ethanol and bio-butanol are common biofuel obtained from the fermentation process (Binod et al., 2011). Methane and carbon dioxide are produced from the anaerobic digestion of biomass by bacteria in the absence of free oxygen (Lemmer et al., 2015).

2.3 Biomass Logistics

Biomass logistics encompasses all the operations that are used to prepare harvested biomass before they enter the throat of conversion plants. Transportation, handling, densification, size reduction, drying, and storage fit within this classification (Mafakheri and Nasiri, 2014). The goals of biomass logistics process is to provide feedstock that meets the requirements of end-users and to ensure that feedstock are provided in a reliable and sustainable manner. Cost and quality are therefore essential to the biomass logistics process. Biomass feedstock quality is determined by the properties of biomass, and the value of these properties are dependent on the end-use and desired conversion process.

One of the major problems with biomass is its low bulk density. This results in high transportation cost because transportation rate is typically based on per unit volume (Tabil et al., 2011). Likewise, a large storage volume or space is required to store biomass because of its low density. Consequently, biomass are densified to increase their bulk density. Common densification processes are pelleting, briquetting, and cubing. According to Fasina (2008), the bulk density of peanut hull increased from 151 kg/m^3 for raw materials to about 600 kg/m^3 for pellets. Colley et al. (2006) reported that pelleting increased the bulk density of switchgrass to 708.0 kg/m^3 from 169.5 kg/m^3. Several studies have also shown that densification improves the flow and storage properties of biomass (Kaliyan and Morey, 2006).

The moisture content of woody biomass at harvest is approximately 50% for woody biomass (Cutshall et al., 2011) while the moisture content of grassy biomass at harvest varies between 18% and 22% (Kemmerer and Liu, 2014). Drying of biomass is therefore necessary to meet the needs of thermochemical based bio-refinery, as some thermochemical conversion processes require low moisture content feedstock. For example, 8% - 10% moisture content is recommended for fast pyrolysis (Dobele et al., 2007). Drying also help to reduce the transportation cost because less water is transported after drying.

The size of individual unit (such as logs, leaves, and straws) of biomass after harvest is typically several orders of magnitude larger than the size requirement for bioenergy conversion plants. Particle size reduction is used to prepare biomass into a form that can be transported, handled, stored, and converted effectively by these bioenergy conversion plants. The size reduction process mechanically breaks larger biomass feedstocks into smaller sizes, resulting in increased total surface area, number of inter particle bonding, reactivity, and digestibility (Mani et al., 2004; Igathinathane et al., 2009). Particle size reduction has been given different names such as milling, grinding, chopping, cutting, dicing, slicing, and chipping. In many cases this nomenclature describes the size reduction ratio—the ratio of the average particle size of the feedstock to the average particle size of grinds. Particle size reduction of liquid is referred to as emulsification, homogenization or atomization (Fellows, 2000). The reduction of tree logs into chips (particle size of 3.15 mm to 45.00 mm—Pochi et al., 2015) is commonly referred to as chipping while the fracturing of solid materials into particulate/granular matters (typically < 3 mm particle size—Tumuluru et al., 2014) is called grinding.

This study focuses on the grinding of biomass, and discussions on grinding theory, grinding energy requirement and grinding equipment, grinding equations, particle size distribution, and moisture loss during grinding are therefore presented in sections 2.4, 2.5, 2.6, 2.7, and 2.8, respectively.

2.4 Grinding Theory

Three major forces are involved in the grinding process. They are impact force, compression force and shear force. Although in every grinding equipment, all these forces exist, only one is usually found to be prominent. Impact force is dominant in hammer mills, shear force in attrition mills and compression force in roller mills (Fellows, 2000). See section 2.5 on the details of these grinding equipment.

The ideal stress-strain response of biological materials to applied force is shown in Figure 2.2. When grinding forces are applied to biological material, these forces will be initially absorbed by the biological materials and cause the deformation of the tissues of the biological material (O – E in Figure 2.2). If the forces are withdrawn before the strain in the material reaches the elastic stress limit (E) the material tissues return to their original form before deformation and the energy absorbed is released as heat. For strains beyond the elastic stress limit, the material is said to be permanently deformed because it has lost its ability to restore to its initial form. Further application of force increases the strain in the material until the yield point is reached. Above the yield point (Y), the material begins to flow until it reaches the breaking point (B) where fission is achieved along the lines of fissure (Figura and Teixeira, 2007; Brennan 2006). Some materials do not exhibit all the forms and points described above before breaking (Figure 2.2).

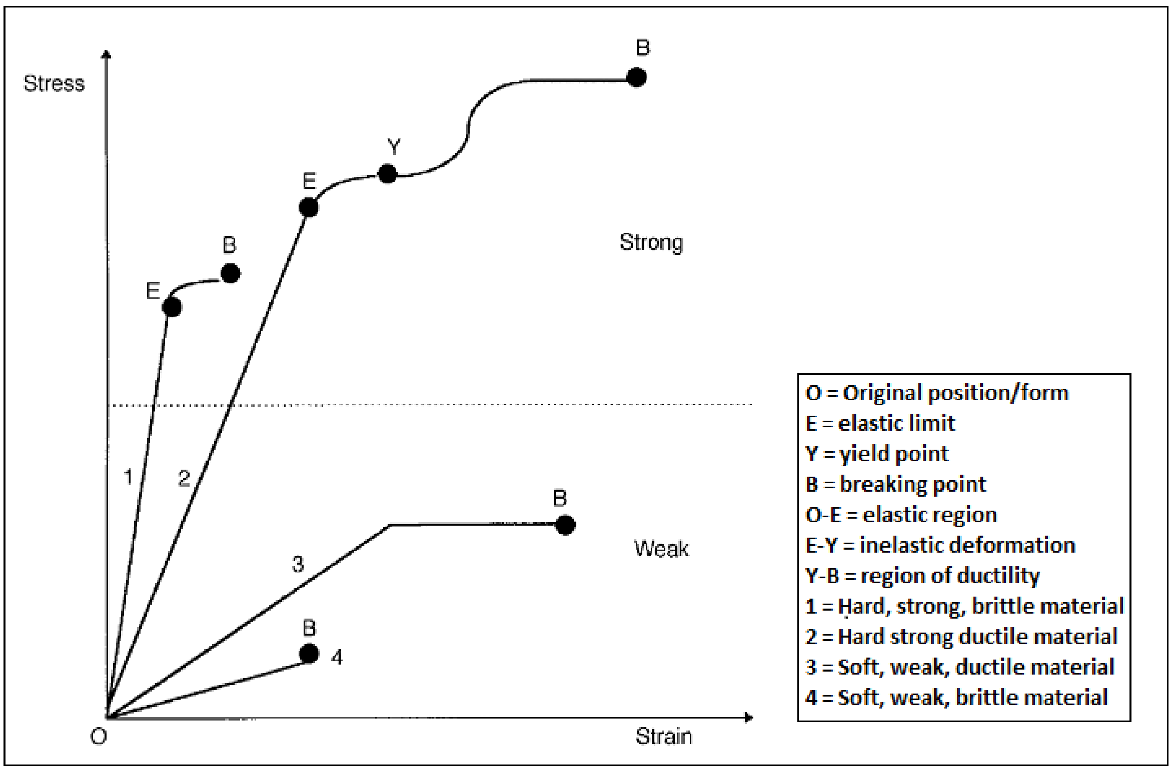

Figure 2.2. Ideal stress-strain response of biological materials (Brennan, 2006).

At fission, energy stored in the material is released in form of heat and sound while some are used to create new surfaces. It is important to note that the number of fissure lines or cracks in particles reduces with particle size. Therefore, new cracks have to be created on particles that do not have existing cracks in order to further reduce the particles. This results in more energy consumption. Hence, the smaller the particle size, the higher the grinding energy consumption (Fellows, 2000; Loncin and Merson, 1979).

2.5 Biomass Grinding and Energy Requirement

Grinding is a highly energy inefficient and energy intensive process. Only about 0.06% - 1% of the total energy consumed in the process is associated with new surface creation (Mohsenin, 1986). A great percentage of the energy consumed during grinding process is used up/lost by inefficiency of motor and transmission, friction between particles, friction between particles and machine elements, windage losses, noise, heat, vibration, hysteresis losses of unfractured material, strain energy of unfractured material, energy of transport of material within the equipment, and energy of fracture itself (Igathinathanea et al., 2009; Galanty and Miller, 1963). Biomass occurs in different forms and structure, and it has varying chemical, physical, and mechanical properties. Therefore, energy required to grind the various biomass feedstock differ (Naimi et al., 2012; Mani et al., 2004). Several studies have revealed that the main factors influencing the energy requirement during grinding are as follows:

i. Grinding equipment
ii. Grinding equipment operating parameters
iii. Biomass type
iv. Biomass moisture content
v. Size reduction ratio
vi. Biomass pretreatment

2.5.1 Grinding Equipment

Grinding efficiency depends on the size reduction technology used for grinding (Mohsenin, 1986; Ghorbani, et al., 2010). Miao et al. (2011) measured the specific grinding energy requirement for hammer mill and knife mill, and they reported that for the same screen size, knife mill consumed higher energy but produced smaller particle size than hammer mill. The specific grinding energy requirement for miscanthus ground through 1 mm screen size was about 200 kJ/kg d.b. for hammer mill and about 700 kJ/kg d.b. for knife mill. Examples of grinding equipment used for grinding biological materials are described below.

i. Hammer mill

Hammer mill is the most commonly used equipment for comminuting biological materials because it is versatile, relatively cheap, operates on a simple mechanism, and has high throughput. Figure 2.3 shows an annotated diagram of a typical hammer mill. Hammers, sometimes referred to as beaters, are arranged on a shaft rotating at a high velocity and powered by a prime mover. Materials are fed into the machine by gravity, and particles are ground by the impact forces from the fast moving beaters in the grinding chamber. Ground materials with size smaller than the screen at the base of the chamber fall through the screen onto the collection container. Materials larger than the screen size remain in the chamber and are continuously recirculated and hammered until they are smaller than the screen opening (Manlu et al., 2003). It is practically impossible to predetermine the particle distribution of the ground product, as it is highly influenced by the breakage characteristics of feedstock, particle orientation during impact with the hammers, and several hammer mill operating parameters (Probst et al., 2013; Bitra et al., 2008).

Ground materials from hammer milling have a large range of particle size (high coefficient of variation) but is desirable in densification processes such as pelleting and cubing (Mani et al., 2004). Ghorbani, et al. (2010) reported that the coefficient of variation of hammer milled alfalfa grinds ranged from 48.89% to 106.25%. Some other disadvantages of hammer milling include high dust production, high energy inefficiency, noise pollution, and high length to width ratio (Dooley et al., 2013).

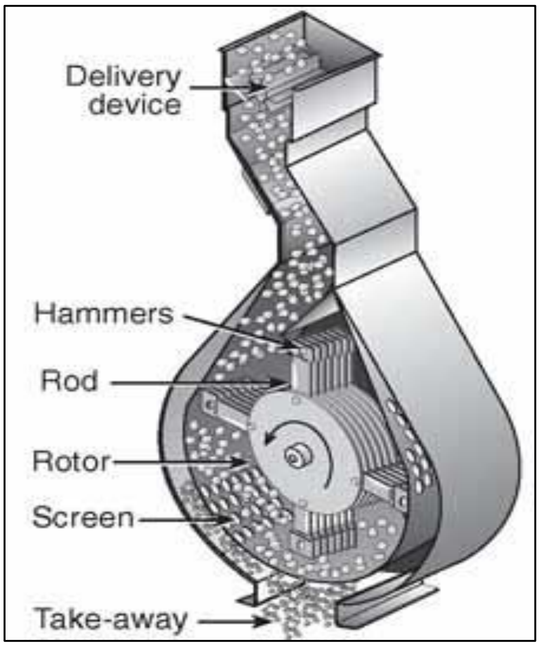

Figure 2.3. A typical hammer mill (FeedMachinery, 2013).

ii. Knife mill

In a knife mill, shear force is applied to materials by sharp blades/knives rotating at a high velocity (Figure 2.4). The materials are continuously sheared and reduced to smaller sizes until they are small enough to pass through the screen at the base of the grinding chamber (Wennerstrum et al., 2002). The blade bevel angle of the knife mill is an important parameter that influences the efficiency of this grinding equipment (Miu et al., 2006). Womac et al. (2005) studied the shearing characteristics of some biomass stems and reported that the average cutting energy of corn stover, hickory wood, and switchgrass stems at were 27.94 kJ/m^2, 121.99 kJ/m^2, and 78.00 kJ/m^2, respectively for 30^o knife bevel angle and 34.19 kJ/m^2, 160.07 kJ/m^2, and 95.20 kJ/m^2, respectively for 45^o knife bevel angle.

Grinds produced from knife mills contain less fines than those produced from hammer mills. According to Himmel et al. (1985), hammer milled aspen produced higher proportion of fines (< 0.25 mm) than knife milled aspen. Paulrud et al. (2002) ground woodchips (mixture of pine and spruce) using a knife mill and hammer mill fitted with 1.5 mm screen size, and they reported that about 70% of the particles from hammer mill was less than 500 μm while about 15% of the particle from knife mill was less than 500 μm. The authors also reported that knife mill produced shorter and broader particles (i.e. smaller particle length-to-width ratio). Knife mills are liable to damage from small rocks and metals which may be picked up during prior unit operations. Himmel et al. (1985) suggested that knife mills should be equipped with feeding mechanism (such as pneumatic) that can remove dense contaminants from biomass, especially straws and corn stover, before entering into the grinding chamber.

Figure 2.4. A typical knife mill (Naimi, 2008).

iii. *Attrition mill*

In the attrition mill, materials are rubbed between two rough surface plates to cause shearing. The distance between the two plates and their relative speed determines the efficiency of this mill. Attrition mills are suitable for grinding dry and wet materials (Brennan, 2006). One of the plates can be held stationary while the other rubs the material against it to shear the material. The other option is to counter rotate the plates at high velocity. Frequent replacement of grinding plate is a major problem associated with this type of mill (Fellows, 2000). According to Himmel et al. (1985), the comminution of aspen woodchips using attrition mill required more energy (about 792 kJ/kg) than hammer milling (about 504 kJ/kg) because attrition mills are designed to grind brittle materials such as coal. Furthermore, excessive heat is generated during attrition milling and may damage biomass (Naimi, 2008).

iv. *Ball mill*

Ball mills are mostly used to produce fine particles. Steel balls (2.5 cm – 15.0 cm in diameter) are utilized in reducing the size of materials in the ball mill. These balls are introduced into a rotating cylinder containing the material to be ground. At low speed, grinding is done by shear force while at a high speed of rotation, impact force from lifting and dropping the balls is used in grinding. Beyond a critical speed, grinding stops because the balls are just rotated round the wall of the cylinder by the high centrifugal force (Fellows, 2000). Equation 2.1 gives an expression for the critical speed of operation. In practice, operation speed of ball mills is usually restricted to 65% – 80% of the critical speeds (Brennan, 2006).

$$N = \frac{42.3}{\sqrt{D}} \tag{2.1}$$

where

N = Critical speed (rpm)

D = Diameter of the cylinder (m)

Ball milling have been reported to cause reduction in the crystallinity of biomass cellulose which improves digestibility during hydrolysis and fermentation. Sidiras and Koukios (1989) observed a negative correlation between the crystallinity index of the barley straw cellulose and ball milling time. Chang and Holtzapple (2000) measured the crystallinity index before and after ball milling of poplar wood and reported that crystallinity index decreased from 54% to 10%.

v. Crumbler

Crumbler is a relatively new technology developed for biomass size reduction to address the limitations of conventional grinders (hammer mill and knife mill). It was originally designed for woody biomass size reduction but has been tested for grassy biomass, such as switchgrass and corn stover (Dooley et al., 2013). The laboratory crumbler (Figure 2.5) was developed by Forest Concepts, LLC, Auburn, Washington to accommodate wood materials that are not more than 16 mm thick, 130 mm wide, and 1 m long. Woody biomass is processed into veneer using rotary veneer lathe or chips using conventional chippers before crumbling. The particle size of grinds (crumbles) is controlled by the thickness of the wood veneer and the crumbler cutter width which ranges from 1.5 mm to 6 mm (Lanning et al., 2012).

According to Lanning et al. (2012), the crumbles have narrower particle size distribution compared to grinds obtained from hammer milling which improves their flow properties. The specific grinding energy consumed by the crumbler is also lower when compared to the specific grinding energy consumed during hammer milling. For example, the specific grinding energy requirement for poplar woodchips was 240 kJ/kg d.b. and 690 kJ/kg d.b. for crumbler and hammer mill, respectively. In their study on simultaneous saccharification and fermentation of redcedar, Ramachandriya et al. (2014) reported that 2.5 mm crumbles and 0.5 mm hammer mill grinds produced comparable wood glucan-to-ethanol yield.

Figure 2.5. Forest Concepts laboratory crumbler (Dooley et al., 2013).

The grinding energy requirements for some biomass species and grinding equipment as reported in some studies are shown in Table 2.1.

Table 2.1. Grinding energy for different biomass and grinding equipment.

Biomass Type	Equipment	Screen Size (mm)	Grinding Energy (kJ/kg)	Reference
Douglas fir	Knife mill	N/A	131.70 – 161.50	Niami et al., 2012
Pine	Knife mill	N/A	200.80 – 263.50	Niami et al., 2012
Aspen	Knife mill	N/A	197.40 – 232.00	Niami et al., 2012
Hybrid poplar	Knife mill	N/A	252.60 – 297.30	Niami et al., 2012
Hardwood chips	N/A	0.60 - 0.20	72.00 – 144.00	Datta, 1981
Hardwood chips	N/A	0.30 - 0.15	360.00 – 720.00	Datta, 1981
Pine chips	Knife mill	0.71	853.20	Phanphanich and Mani, 2011
Torrefied Pine	Knife mill	0.13	86.04	Phanphanich and Mani, 2011
Logging residues	Knife mill	0.74	852.12	Phanphanich and Mani, 2011
Alfalfa	Hammer mill	1.96 – 0.32	30.51	Ghorbani et al., 2010
Alfalfa	Hammer mill	1.53 – 0.42	5.65	Ghorbani et al., 2010
Switchgrass	Hammer mill	2.00	115.79	Miao et al., 2011
Switchgrass	Knife mill	2.00	263.16	Miao et al., 2011
Switchgrass	Hammer mill	5.60	161.64	Samson et al., 2000
Corn stover	Hammer mill	N/A	57.60	Ehlers et al., 2013
Corn stover	Flail Shredder	N/A	13.50	Ehlers et al., 2013
Canola straw	Hammer mill	19.05	10.48	Tumuluru et al., 2014
Wheat straw	Hammer mill	19.05	12.67	Tumuluru et al., 2014

2.5.2 Grinding Equipment Operating Parameters

The efficiency of grinding equipment is dependent on several operating parameters. The major parameters are screen size, feed rate, and rotor speed. The contribution of screen size to grinding efficiency and grinding energy requirement is discussed in section 2.5.5 because of its direct impact on size reduction ratio.

i. Feed rate

Feed rate is the rate of injecting materials into grinding machine. Feed rate has significant influence on grinding efficiency because the specific energy required for grinding materials is ultimately a function of the grinding machine capacity (O'Dogherty, 1982; Lopo, 2002). Niami et al. (2013) reported that about 21 kJ/kg increase in specific grinding energy occurred due to reduction in feed rate from 7.56 kg/h to 4.50 kg/h. Mani et al. (2004) also ascribed feed rate as one of the potential causes for about 100% inflation in the energy required to grind switchgrass when they compared results from their study to the data reported by Jannasch et al. (2002). However, both authors did not specify the feed rate considered in their work. Dey et al. (2013) studied the hammer milling of coal and iron ore, and they reported that specific grinding energy consumption initially increased (from 3.10 kJ/kg to 3.25 kJ/kg for coal and from 4.50 kJ/kg to 4.97 kJ/kg for iron ore) as feed rate increased from 100 kg/h to 125 kg/h, then decreased to 2.97 kJ/kg for coal and 4.28 kJ/kg for iron ore when feed rate was further increased to 150 kg/h. The authors attributed this to a corresponding changes in size reduction ratio as a result of feed rate.

Throughout rates (grinding capacity)—the rate at which ground particles flow out of the grinding equipment—is proportional to feed rate. Therefore, the higher the feed rate, the higher the griniding capacity. High feed rate may however result in oveloading and clogging of grinding equipment.

ii. Rotor speed

Similar to feed rate, the speed of the grinding equipment rotor influences the efficiency and energy consumption during grinding. Depending on the grinding equipment used, energy consumption could increase or decrease when the equipment rotor speed is increased. Hassan (1994) documented that grinding energy reduced by 59.1% and 67.9% when the hammer mill rotor speed was changed from 1460 to 2930 rpm and from 1460 to 3910 rpm, respectively.

Bitra et al. (2009b) measured the specific grinding energy of switchgrass, wheat straw, and corn stover to be 114.4 kJ/kg, 125.1 kJ/kg, and 103.7 kJ/kg, respectively at hammer mill rotor speed of 2000 rpm. When the hammer mill rotor speed was increased to 3600 rpm, the authors reported increase in specific grinding energy of 37% for switchgrass, 30% for wheat straw, and 45% for corn stover. Dey et al. (2013) reported similar trend for hammer mill grinding of coal and iron ore. Bitra et al. (2009c) also reported that the average specific energy consumed during the knife milling of corn stover and wheat straw increased from 15.66 kJ/kg to 23.18 kJ/kg, and 18.43 kJ/kg to 20.27 kJ/kg, respectively when rotor speed was increased from 250 rpm to 500 rpm. However, they reported that the average specific grinding energy for switchgrass decreased by 14% when rotor speed was increased from 250 rpm to 500 rpm.

2.5.3 Biomass Type

The differences in the physical and chemical properties of biomass contribute to the variability in the energy required to grind these feedstocks. Tumuluru et al. (2014) reported that the specific energy consumption for hammer mill grinding (19.05 mm screen size) was 11.63 kJ/kg, 10.48

kJ/kg, 28.98 kJ/kg, and 12.67 kJ/kg for barley, canola, oat, and **w**heat, respectively. Naimi et al, (2012) attributed the difference in specific grinding energy requirement for four biomass (Douglas fir, 131.7 kJ/kg – 161.5 kJ/kg; pine, 200.8 kJ/kg – 263.5 kJ/kg; aspen, 197.4 kJ/kg – 232.0 kJ/kg; hybrid poplar, 252.6 kJ/kg – 297.3 kJ/kg) to the difference in the lignin content and ash content. Lignin content was found to negatively correlate with grinding energy requirement while higher ash content resulted in higher grinding energy requirement. Mani et al. (2004) studied the hammer mill grinding of wheat straw, barley straws, corn stover and switchgrass. The authors reported that switchgrass had the highest specific grinding energy because of its fibrous nature. In addition, biomass mechanical properties, such as ductility and strength are important properties that influence grinding energy requirement of biomass. According to Fellows (2000), friability—the hardness of a biological material and its tendency to crack is influenced by the structure of the biological materials and directly proportional to the energy required to grind.

2.5.4 Biomass Moisture Content

Moisture content is perhaps the most important property of biological materials because several of the other physical properties are impacted by the moisture content of biological materials (Mathlouthi, 2001). Freshly harvested woody biomass has a high moisture content close to 50% (Cutshall et al., 2011) while grassy biomass are harvested at about 20% moisture content (Kemmerer and Liu, 2014). Several studies have shown that the moisture content of biomass is directly proportional to energy consumed during grinding.

Mani et al. (2004) reported that energy requirement for grinding corn stover increased from 79.45 kJ/kg to 123.48 kJ/kg when moisture content increased from 6.2% to 12.0% (wet basis). Similarly, the energy required to grind switchgrass increased from 186.34 kJ/kg to 210.49 kJ/kg as moisture content increased from 8.0% to 12.0% (wet basis). Soucek et al. (2003) observed that as the moisture content of knot weed increased from 9.18% to 19.66%, specific grinding energy increased from 190.80 kJ/kg to 835.20 kJ/kg. Hehar (2013) reported the increase in specific grinding energy for loblolly pine woodchips from 124.62 kJ/kg to 360.00 kJ/kg for hammer mill screen size 1.20 mm and from 82.10 kJ/kg to 236.70 kJ/kg for hammer mill screen size 3.18 mm when moisture content increased from 4.7% to 23.6%. Zhang (2014) studied the knife milling of poplar woodchips and reported that specific grinding energy increased from 4968.00 kJ/kg (for 1.2% moisture content) to 5741.05 kJ/kg (for 10% moisture content). The increase in grinding energy with moisture content can be attributed to increase in ductility as a result of the plasticizing effect of water (Łysiak, 2007).

Little attention has been given to wet grinding of biomass. There is therefore limited information available on the grinding performance and specific grinding energy requirement of biomass above 30%. However, Forest Concepts (2012) studied the wet grinding of biomass and reported that specific grinding energy was 240 kJ/kg d.b. and 730 kJ/kg d.b. for poplar chips (45% moisture content) and corn stalks (46% moisture content), respectively using hammer mill. Miao et al. (2011) also reported that the grinding energy requirement for energy cane at 40% was approximately 1400 kJ/kg d.b. when ground through 8 mm screen size.

Armstrong et al. (2007) observed that the time required to grind corn increases with moisture content. The time required to grind increased from about 20 s to 29 s as moisture content increased from 9.25% to 15.41%. This is because ground particles at higher moisture content agglomerates to form larger mass which in turn clog the opening on the grinding equipment screen and prevents materials from flowing out of the grinding chamber (Fellows, 2000). Dust production/pollution during grinding is higher at lower moisture contents. Dust production causes material loss, health hazards to workers, and increases the risk of fire explosion in processing plants (Kent, 1983;

Fellows, 2000). Hehar et al. (2014) reported that dust production increased from 5.43% to 21.70% (for hammer mill screen size 1.2 mm), 4.25% to 10.11% (for hammer mill screen size 3.18 mm), and 5.29% to 14.11% (for hammer mill screen size 6.35 mm) when the moisture content of loblolly pine woodchips was decreased from 23.6% to 4.7%.

2.5.5 Size Reduction Ratio

The ratio of the average particle size of feedstock to the average particle size of grinds is referred to as the size reduction ratio. This parameter is controlled by the aperture opening of the screen of the grinding equipment and also depends on the initial size of the biomass and the required size for post-grinding processes. The specific grinding energy requirement for biomass grinding increases with increase in the size reduction ratio or decrease in grinding equipment screen size (Brennan, 2006). Adapa et al. (2011) reported that the specific energy consumption during grinding of barley, canola, oat, and wheat straws using a screen size of 6.4 mm was 29.74 kJ/kg, 44.57 kJ/kg, 58.43 kJ/kg, and 44.14 kJ/kg, respectively. When the screen size was reduced to 1.6 mm, specific grinding energy increased to 90.36 kJ/kg, 128.52 kJ/kg, 149.47 kJ/kg, and 153.25 kJ/kg (for barley, canola, oat, and wheat straws, respectively). Mani et al. (2004) also reported an inverse proportionality between screen size and energy consumption during grinding: Average specific energy consumed during grinding of wheat straw of initial mean chop size of 7.67 mm were 185.58 kJ/kg, 133.24 kJ/kg, and 40.90 kJ/kg for screen sizes 0.8 mm, 1.6 mm, and 3.2 mm. Cadoche and Lopez (1989) observed that specific grinding energy requirement increased from 27.0 kJ/kg to 28.8 kJ/kg for agricultural wastes straws and from 10.08 kJ/kg to 468.00 kJ/kg for hardwoods when size reduction ratio was increased from 2 to 14.

Particles split into smaller units along cracks on the particles during grinding, and as the particle size reduces, the number of these cracks reduces. Grinding energy requirement therefore rapidly increases as particle size decreases because more energy is consumed in the production of new cracks in smaller particles before they are fractured (Fellows, 2000). Ghorbani et al. (2010) reported that the specific grinding energy consumed in the hammer milling of alfalfa chops through a size reduction ratio of 4.8 increased from 12.36 kJ/kg to 20.61 kJ/kg as the initial particle size of chops increased from 1.53 mm to 1.96 mm.

2.5.6 Biomass Pretreatment

Torrefaction is the most widely known pretreatment method that has been demonstrated to reduce biomass grinding energy consumption. Torrefaction is a heat treatment process that removes the hemicellulose component of biomass and changes the structure of the biomass. Torrefied biomass are hydrophobic and have increased brittleness. These changes and the disintegration of lignin and hemicellulose during the torrefaction process are believed to be responsible for the improved grindability and higher energy density of torrefied biomass (Arias et al, 2008). Phanphanich and Mani (2011) reported that the energy required to grind torrefied pine was 10 times less than that required to grind untreated pine. Similary, Repellin et al. (2010) obtained about 90% decrease in energy required to grind natural beech when beech was torrefied at 280 °C. The authors also reported that torrefaction decreased the grinding energy of spruce from 2700 kJ/kg to less than 360 kJ/kg, when spruce was torrefied at 300°C.

Although energy consumed during grinding torrefied biomass is drastically reduced, the process of torrefaction also consumes energy (Bergman and Kiel, 2005; Repellin et al., 2010). According to Carter et al. (2013), the energy required for biomass torrefaction was found to be directly proportional to moisture content. It increased from 310.0 kJ/kg to 509.8 kJ/kg for pine, 277.3 kJ/kg to 574.7 kJ/kg for sweet gum, and 259.1 kJ/kg to 609.9 kJ/kg for switchgrass as moisture content increased from ~ 4% to ~ 19%. There is therefore a trade-off between grinding energy reduction and energy utilized in biomass torrefaction.

Other biomass pretreatment that can influence grindability and grinding energy requirements are: hydrothermal carbonization and drying. Hydrothermal carbonization increases hydrophobicity of biomass and drying reduces the moisture content (section 2.5.4 describes the effect of moisture content on biomass grinding energy requirements).

2.6 Grinding Equations

Grinding equations are mathematical models that are commonly used to predict the energy consumed during grinding process as a function of the particle sizes before and after grinding, and a characteristic grindability coefficient. The general grinding equation assumes that grinding energy is directly proportional to the differential change in particle size and inversely proportional to the nth power of the size of the material (Equation 2.2).

$$\delta E = -K\frac{\delta L}{L^n} \tag{2.2}$$

where

δE = specific grinding energy requirement (kJ/kg)

δL = differential change in particle size (mm)

L = particle size (mm)

K = grinding difficulty (kJ/kg mm^n)

n = constant (dimensionless)

The commonly used grinding equations (Kick, Rittinger, and Bond equations) assign values to the constant (n) in the general equation (Equation 2.2) (Stamboliadis, 2004). These three equations have been used to estimate the energy required to grind biological materials (Djantou et al., 2007; Naimi et al., 2013) and are described below:

i. Kick's equation

Kick's equation was formulated on the premise that n in Equation 2.2 is equal to 1, based on the assumption that the energy required for grinding is proportional to the ratio of the average particle size of the feedstock to the average particle size of the grinds which is also called the size reduction ratio (Djantou et al., 2007; Naimi et al., 2013). The integration of Equation 2.2 when n = 1 gives:

$$\Delta E = K_k \ln \frac{L_f}{L_p} \tag{2.3}$$

where

ΔE = specific grinding energy requirement (kJ/kg)

K_k = Kick constant (kJ/kg)

L_p = average particle size of grinds (mm)

L_f = average particle size of feedstock (mm)

ii. Rittinger's equation

Equation 2.4 expresses the Rittinger equation, and it assumes that all the energy consumed in grinding is used to create new surface area through shearing. Therefore, the Rittinger's equation assigns the value of 2 to n in Equation 2.2 (Djantou et al., 2007; Naimi et al., 2013). Equation 2.4 is derived from the integrating of Equation 2.2 when n = 2.

$$\Delta E = K_R \left(\frac{1}{L_p} - \frac{1}{L_f} \right) \tag{2.4}$$

where

ΔE = specific grinding energy requirement (kJ/kg)

K_R = Rittinger constant (kJ/kg mm)

L_p = average particle size of grinds (mm)

L_f = average particle size of feedstock (mm)

iii. Bond's equation

The Bond's equation (Equation 2.5) takes into consideration the creation of new cracks tip length in particles before fracturing. Hence, n in Equation 2.2 was given the value 1.5 (Djantou et al., 2007; Naimi et al., 2013).

$$\Delta E = K_b \left(\frac{1}{L_p^{0.5}} - \frac{1}{L_f^{0.5}} \right) \qquad (2.5)$$

where

ΔE = specific grinding energy requirement (kJ/kg)

K_b = Bond constant (kJ/kg $mm^{0.5}$)

L_p = average particle size of grinds (mm)

L_f = average particle size of feedstock (mm)

These three equations have been found to be adequate for different size ranges of grinds. Rittinger's equation adapts well for fine grinding while Kick's equation adapts well for coarse grinding. The Bond's equation adequately predicts the energy requirement in coarse, intermediate, and fine grinding (Brennan, 2006; Fellows, 2000; Lee et al., 2013). Size reduction ratio below 8:1 can be classified as coarse grinding while the size reduction ratio for fine grinding can exceed 100:1 (Brennan et al., 1990). Several studies have fitted all three equations to biomass grinding. Ghorbani et al. (2010) reported that Rittinger equation gave the best fit for the hammer milling of alfalfa chops while Naimi et al. (2013) reported that Kick's equation had the best fit for the knife milling of hybrid willow and Douglas fir composite.

According to Galanty and Miller (1963), "literature on size reduction is plentiful and several useful laws are available, but a single generally acceptable theory has not been formulated. The framework is complex and promises to be more complex in the future." This viewpoint still holds today because of the vast differences in chemical composition and physical properties of biological materials (Gent et al., 2012; Sharma et al., 2008; Chakkaravarthi et al., 1993) and underscores the need for investigation into grinding energy requirement for biological materials.

In their study on the grinding of miscanthus, switchgrass, willow, and energy cane, Miao et al, (2011), modelled the specific grinding energy requirements as a power function of screen size, size reduction ratio, and geometric mean diameter. Soucek et al. (2003) studied the grinding of spruce bark, willow, cypress, apple tree, and poplar, and they also reported a power relationship between the energy required to grind and the screen size of the grinding equipment. Mani et al. (2004) established a linear relationship between the specific grinding energy requirements and hammer mill screen size for biomass (switchgrass, barley straw, wheat straw, and corn stover) at 8% moisture content. The authors however observed a polynomial relationship between the specific grinding energy requirements and hammer mill screen size for biomass at 12% moisture content.

2.7 Particle Size and Particle Size Distribution

Apart from grinding energy consumption, grinding performance is evaluated by particle size and particle size distribution of both feedstock and ground materials (Brandt et al., 2012). Particle size distribution of biological materials is a measure of the spread of the size population of particles, and it is usually expressed as a frequency distribution curve. The size used in particle size distribution is a function of the measurement method used or the process the particles will be subjected to. Examples of commonly used sizes are: martin diameter, ferret diameter, stokes diameter, drag diameter, and surface area diameter (Fıratlıgil-Durmus et al., 2010).

The most common equivalent particle size used for biological materials is the geometric mean diameter (Phanphanich and Mani, 2011; Bernhart and Fasina, 2009). It is the median diameter for a log-transformed particle size distribution. Particle size distribution of biological materials are mostly log-transformed to remove their inherent skewness thereby imposing normality on the distribution (log-normal distribution). Geometric standard deviation is used to quantify the spread/range of particle size around the geometric mean diameter (Temmerman et al., 2013).

Sieve analysis, image analysis, laser diffraction method, sedimentation approach, air elutriation analysis, and microscopy method are measurement techniques used for measuring particle size of biological materials (Allen, 2003). Sieve analysis is mostly used because it is cheaper and versatile, even though it is laborious. It utilizes a combination of standard sieves, arranged in descending order of their opening sizes, and a mechanical shaker (ASABE Standards, 2008). According to Igathinathane et al. (2012), sieve analysis produced lower geometric mean diameter when compared to image analysis. This was attributed to the slipping down of long particles through the sieves.

The particle size of biomass grinds is influenced and controlled by the screen size of grinding equipment. Womac et al. (2007) observed a positive correlation between knife mill screen size and geometric mean diameter. The geometric diameter of grinds increased for corn stover (from 3.62 mm to 14.01 mm), for switchgrass (from 3.05 mm to 13.04 mm), and for wheat straw (from 3.46 mm to 10.87 mm) as knife mill screen size increased from 12.7 mm to 50.8 mm. Tumuluru et al. (2014) similarly observed that when hammer mill screen size increased from 19.05 mm to 31.75

mm, the geometric mean diameter of oat straws, canola straws, wheat straws, and barley straws increased from 1.50 mm to 2.18 mm, 0.98 mm to 1.21 mm, 1.17 mm to 1.68 mm, and 1.13 mm to 1.63 mm, respectively.

Probst et al. (2013) reported that the moisture content of corncobs influenced the particle size distribution of the resulting grinds. The geometric mean diameter of corncobs initially increased from 0.84 mm to 0.90 mm as moisture content increased from 10.04% to 14.65%, then decreased to 0.73 mm as moisture content increased to 20.13%. Naimi et al. (2012) studied the knife mill grinding of woody biomass using a 2 mm screen size and reported that the particle size distribution of grinds was affected by the biomass type. Approximately 50% of the particles of aspen and hybrid poplar grinds were smaller than 0.42 mm while approximately 35% of the particles of Douglas fir grinds were smaller than 0.42 mm. Less than 30% of the particles of loblolly pine grinds were smaller than 0.42 mm Adapa et al. (2010) also reported that the geometric mean diameter of grinds was biomass type specific. The authors reported that geometric mean diameter of grinds was 0.463 mm (for barley straw), 0.521 mm (for canola straw), 0.566 mm (for oat straw), and 0.719 mm (for wheat straw) when grinding was carried out with hammer mill screen size of 3.2 mm. The differences in the particle size distribution of biomass due to moisture and biomass type can be ascribed to the difference in the mechanical properties of biomass feedstock.

In their study on the comminution of switchgrass, wheat straw, and corn stover using hammer mill, Bitra et al. (2009b) established that the geometric mean diameter of grinds was a polynomial function of the hammer mill rotor speed (Table 2.2). In another study, Bitra et al. (2009a) worked on the knife milling of switchgrass, and they reported that the particle size distribution of knife milled switchgrass was affected by feed rate and knife mill rotor speed, but no specific trend was observed. The authors attributed this to the unpredictability in the breakage characteristics of switchgrass during grinding.

Table 2.2. Relationship between geometric mean diameter and hammer mill speed for switchgrass, wheat straw, and corn stover (Bitra et al., 2009b).

Biomass	**Regression Model****	**r^2**
Switchgrass	$0.0937 + 5.1100E\text{-}04N – 1.1579E\text{-}07N^2$	0.9795
Wheat Straw	$0.7348 + 5.3327E\text{-}05N – 2.0176E\text{-}08N^2$	0.9458
Corn Stover	$0.5362 + 8.3919E\text{-}05N – 2.3900E\text{-}08N^2$	0.5677

*N = Hammer mill rotor speed.

2.8 Moisture Loss during Grinding

Most biological materials are viscoelastic. The viscoelasticity of biomass causes the conversion of applied grinding mechanical energy to heat energy (Havimo and Hari, 2010). Therefore, there is the generation of heat during biomass grinding. Heat produced during grinding increases the temperature of the biomass and its water content, and result in their release as water vapor. Researches on grinding have reported a difference between the mass of feedstock and the grinds, and this has been attributed to dust lost, unground sample retained in the grinder, and moisture loss. Fiber content of biomass and size reduction ratio have been reported to influence this particular phenomenon of moisture loss. Higher fiber content is believed to cause lower moisture loss, whereas, higher size reduction ratio causes higher moisture loss. In a study on the grinding of alfalfa chops by hammer mill, 4.1% reduction in moisture was observed for average ground particle size of 1.96 mm. 9% of the initial moisture was loss for average particle size of 1.68 mm and 9.37% for 1.53 mm (Ghorbani et al., 2010). Probst et al. (2013) measured the moisture content of corn and corncobs before and after hammer mill grinding. They observed that the moisture content of corn samples reduced from 19.64% (before grinding) to 18.01% (after grinding) and the moisture content of corncobs samples reduced from 20.13% (before grinding) to 12.93% (after grinding).

2.9 Biomass Storage

Although the entire processes of harvesting, pretreating, transporting and converting woody biomass to produce bio-products can be carried out within a short time, storage is needed provide supply buffer for the period of the year when demand exceeds supply. Storage is therefore used to ensure continuous and dependable supply of feedstock (Nurmi, 1999). Biomass is usually stored in bundles, piles (covered and uncovered), silos or bins, loose slash (Scottish Building Standards Agency, 2006; Forest Bioenergy, 2007; Ashton et al., 2007; Afzal et al., 2010).

Similar to other biological materials, biomass are living materials. They therefore respire and experience metabolic and microbial activities during storage which can change biomass properties, composition, and structural configuration (Jirjis, 2005). Brand et al. (2011) highlighted a number of changes in the characteristics of biomass in storage, namely: alteration of extractives, increase in moisture content, decrease in calorific value, and loss of dry matter. These changes are attributed to auto-oxidative, enzymatic processes, and heat development. Nevertheless, several studies have shown that storage of uncomminuted biomass is less susceptible to heat development and microbial actions, and it could also improve the heating quality of the biomass (Jirjis 2005; Brand et al., 2011, Afzal et al., 2010). The storage of comminuted biomass is however common because handling cost for uncomminuted biomass is higher (Barontini et al., 2014).

Although, there are no documented reports on the direct relationship between the method or duration of storage and the energy consumed during grinding of biomass, all the aforementioned alterations in properties of biomass during storage suggests possible changes in the specific grinding energy consumption and the properties of the ground material.

Chapter 3: Effects of Moisture and Storage Time on Specific Energy Required to Grind and Some Physical Properties of Loblolly Pine Ground

3.1 Abstract

Specific grinding energy and particle size distribution of grinds are used to assess the efficiency of grinding equipment and material grindability. The objective of this study was to quantify the effects of moisture content and storage time on the grindability and physical properties of loblolly pine woodchips. Samples were ground in a hammer mill fitted with 3.175 mm and 6.350 mm screen sizes. The specific grinding energy, moisture loss during grinding, particle size distribution and bulk density of the grinds were measured. Woodchips stored for 6 months reduced in volatile matter content from 86.74% to 85.59% and energy content from 20.30 MJ/kg to 20.11 MJ/kg. There was a complimentary increase in ash content during the same storage time from 0.31% to 0.46%. Moisture loss during grinding increased with increase in moisture content of loblolly pine woodchips and decreased with increase in screen size. Bulk density of grinds reduced from 273.64 kg/m^3 to 106.03 kg/m^3 with 3.175 mm screen and from 251.14 kg/m^3 to 131.40 kg/m^3 with 6.350 mm screen when moisture content of pine chips increased from 12% to 50%. The storage time did not significantly affect the specific grinding energy ($p < 0.05$). However, the specific grinding energy increased with increase in moisture content at a decreasing rate. For samples ground through 6.350 mm screen size, a specific grinding energy peak was recorded at 30% moisture before a reduction with further increase in moisture content. Kick, Bond, and Rittinger were fitted, and Bond model was best fitted to the grinding energy data.

3.2 Introduction

Loblolly pine (Pinus taeda), a member of the pine family, is a significant forest resource that can be used for the production of bio-based fuel, power, chemicals, and polymers (Celikbag et al., 2014; Cambero and Sowlati, 2014). Loblolly pine is native to the southeastern United States and currently grows on about 35 million acres of land in the region (Taylor et al., 2012). The selection of loblolly pine for the production of bio-based products is favored by its rapid growth, high yield, and high response to silvicultural management practices (Baker and Langdon, 2008). In the United States, production of bio-based products is aimed at reducing dependence on imported fossil fuel, creation of rural economic opportunities, and environmental sustainability (Maghrour, 2009). According to the Energy Independence and Security Act of 2007, the United States goal is to produce 36 billion gallons of biofuels by 2022 (US Environmental Protection Agency, 2014).

A number of operations are required to prepare biomass before they are converted to fuels, chemicals, and products. These operations include transportation, drying, size reduction, storage and handling (Mafakheri and Nasiri, 2014). Size reduction is one of the most important step because of its impact on the effectiveness and efficiency of downstream processes. Size reduction involves the breaking of biomass particles into smaller fractions and may be classified as chipping, cutting, crumbling, chopping, milling, or grinding depending on the extent of size reduction (Fellows, 2000). For instance, the reduction of tree logs into chips (3.15 mm to 45.00 mm— Pochi et al., 2015) is commonly referred to as chipping while grinding involves the productions of particulate or powder like material (typically < 3 mm—Tumuluru et al., 2014).

The energy consumed during grinding and particle size distribution of resulting grinds are used to determine the effectiveness of grinding operation (Adapa et al., 2011). According to Mohsenin (1986), grinding operation is highly energy intensive and inefficient, as over 99% of the energy consumed during grinding process is lost in the form of heat and sound energy. However, grinding remains an unavoidable operation in the production of bio-based products because in many instances biomass feedstock cannot be converted at the harvest particle size (Tabil et al., 2011). Grinding is also used to improve physical, handling, and storage properties (such as surface area, bulk density, energy density, porosity, and flowability) of biomass feedstock (Mani et al., 2004).

Specific grinding energy (defined as grinding energy consumed per unit mass of material) is influenced by factors such as moisture content, biomass species, initial particle size, size reduction ratio, and type and size of grinding equipment (Lopo, 2002; Ghorbani et al., 2010). Size reduction ratio is the ratio of the average particle size of feedstock to the average particle size of grinds. Mani et al. (2002) hammer milled wheat straw, barley straw, corn stover, and switchgrass and reported that specific grinding energy was biomass specific. Switchgrass consumed the highest specific grinding energy of 225.43 kJ/kg while corn stover consumed the least specific grinding energy of 79.45 kJ/kg for hammer mill screen size of 0.8 mm. Similar result was reported for hammer mill screen sizes 1.6 mm and 3.2 mm by the authors. Adapa et al. (2011) also reported that specific grinding energy of barley increased from 90.36 kJ/kg to 29.74 kJ/kg when hammer mill screen size was reduced from 6.4 mm to 1.6 mm.

Naimi et al (2012) studied the grinding performance of two softwoods (Douglas-fir and Pine) and two hardwoods (aspen and hybrid poplar). They reported that the energy required to grind the hardwood samples was higher than that required to grind the softwood samples. However, the authors also reported that the hardwoods produced smaller particle size. The difference in grinding energy therefore could have been as a result of the differences in particle size or due to differences in properties of the feedstocks. In a similar study on the comminution of Miscanthus, switchgrass, willow, and energy cane, Miao et al. (2011) concluded that hammer mill consumed lower energy, but produced bigger particles, than knife mill. This contradicts the results from Himmel et al. (1985) study on the comminution of hardwood, straw, corn cobs, and corn stover that reported that hammer mill consumed more energy than knife mill. Himmel et al. (1985) also reported in the same study that hammer mill produced higher fines (0.25 mm and smaller). Consequently, it is important to report size reduction ratio (or particle size of resulting grinds) alongside specific grinding energy requirements. The grinding equations that have been used to relate energy consumption during grinding to the particle size of grinds and feedstock are given in Equations 3.1 – 3.3 (Earle and Earle, 1983). The constants of these equations are independent of particle size of resulting grinds and can be used to compare the grinding energy demands for different biomass.

$$\text{Kick:} \quad E = -K_K \ln \frac{L_f}{L_p} \tag{3.1}$$

$$\text{Rittinger:} \quad E = -K_R \left(\frac{1}{L_p} - \frac{1}{L_f} \right) \tag{3.2}$$

$$\text{Bond:} \quad E = -K_b \left(\frac{1}{L_p^{0.5}} - \frac{1}{L_f^{0.5}} \right) \tag{3.3}$$

where

E = energy requirement (kJ/kg)

K_K, K_R, K_b = Kick's, Rittinger's, and Bond's constant respectively

L_p = particle size of grinds (mm)

L_f = particle size of feedstock (mm)

Particle size distribution is affected by machine speed, feed rate, and moisture content of feedstock (Bitra et al., 2009; Mani et al., 2004) and important in assessing the degree of grinding achieved. In spite of its importance, describing particle size distribution of biological materials is problematic. Geometric mean diameter and geometric standard deviation are often used to represent the particle size distribution of biological materials (ASABE Standard S319.4 – ASABE Standards, 2008) because of the ability to take into account the skewness in the particle size distribution. Other properties of grinds such as moisture content and bulk density are important to the performances of subsequent downstream operations. Bulk density is needed in sizing of biomass handling structures, storage containers, and transportation vessels (Kaliyan and Morey, 2006).

Ultimately, the properties of grinds are dependent on the type of equipment used for grinding. One of the most common grinding equipment used for material size reduction is the hammer mill because of its large throughput, flexibility, and durability. However, it produces excessive dust which result in loss of materials (Dooley et al, 2013). Dust also pose health hazards to workers and increase the risk of fire and explosion in processing plants (Amyotte and Eckhoff, 2010). The spread of the distribution of particles produced by hammer mill is wide, thus making hammer milled samples have higher bulk density and suitable for pelletizing, cubing, and briquetting (Mani et al., 2003). Other important grinding equipment are knife mill, and ball mill.

Biomass supply and demand imbalances resulting from major holidays, adverse weather conditions, and energy prices (Rupar and Sanati, 2005) may cause unreliability in the supply of biomass feedstock. To provide supply buffer during these imbalances, biomass is therefore usually stored before or after comminution. Common storage methods are pile (covered and uncovered), bins, silos, and bundles (Ashton et al., 2007). Several literature have reported changes in properties of biomass during storage. Autoxidation and moisture exchange with the environment are among the many processes that occur in the course of storage (Brand et al, 2011; Nurmi, 1999). Afzal et al. (2010), in their study of white birch storage, documented that as storage time increased, there was reduction in energy content and dry matter, and an increase in ash content and carbon content.

However, there have been no documented study on the effect of biomass storage duration on grinding energy and the properties of grinds.

The objectives of this study were to: 1) quantify the effect of storage time on the volatile matter content, ash content, and energy content of loblolly pine woodchips, and 2) quantify the effects of storage time, moisture content, and hammer mill screen size on grinding characteristics of loblolly pine woodchips. The parameters measured to evaluate grinding characteristics were specific grinding energy, particle size distribution of grinds, bulk density of grinds and moisture loss during grinding.

3.3 Materials and Methods

Clean loblolly pine woodchips were obtained from West Fraser Mill, Opelika, AL. Chips were received at 52.23±1.74% moisture content (wet basis) and were air dried in an open space until 12.07±0.21% moisture content (wet basis) was achieved. Moisture content determination was carried out according to ASABE Standard S358.3 (ASABE Standards, 2012) by oven drying about 10 g samples at 103°C for 24 hours. Particle size distribution of the woodchips was determined using TMI chips classifier (model 36852 – 07, Testing Machines, Inc., New Castle, Del.) (Figure 3.1). The screen set used comprised of 45.00 mm, 19.05 mm, 13.00 mm, 7.00 mm, 3.00 mm, and pan (screen aperture were circular). The geometric mean diameter of woodchips was calculated to be 11.616 mm, and geometric standard deviation of woodchips was 6.976 mm.

Figure 3.1. TMI woodchips classifier used for woodchips particle size distribution measurement.

3.3.1 Storage Time Effect on Chemical Properties

3.3.1.1 Sample Preparation

The chemical property tests were carried out on woodchips stored for 0 month, 2 months, 4 months, and 6 months. The loblolly pine woodchips were stored indoor in a plastic bag during the course of the experiment. At the end of each storage month, woodchips were ground with a hammer mill (model 10HBLPK, C.S. Bell Co., Tiffin, Ohio) through 3.175 mm screen, then ground with a Wiley mill through 1 mm screen. Before the chemical properties tests were carried out, the moisture content of the resulting grind was measured according to the ASABE Standard S358.3 (ASABE Standards, 2012) by oven drying about 10 g samples at 103°C for 24 hours. The measured moisture content ranged from 9-11% (wet basis).

3.3.1.2 Volatile Matter Content

The volatile matter content of samples was measured using a furnace (model VMF 10/6/3216P, Carbolite, Hope Valley, England) and according to the ISO 562 Standard (ISO, 2010). Sample of about 1 g was placed in a crucible (with closely fitting cover). The covered crucible and its content were placed in the furnace at 900°C for 7 minutes. After this time period, the crucible was removed and placed in a desiccator to cool to room temperature. Then, the crucible and its content were weighed to the nearest 0.1 mg. The volatile matter content (dry basis) was calculated by applying Equation 3.4 to the data recorded.

$$VC = \left[\left(\frac{M_i - M_f}{M_i - M_c}\right)100\ \right]\left[\frac{100}{100 - M_w}\right] \tag{3.4}$$

where

VC = volatile matter content on dry basis (%, dry basis)

M_c = mass of crucible and lid (g)

M_i = initial mass of crucible, lid and sample dry matter (g)

M_f = final mass of crucible, lid and devolatilized sample (g)

M_w = moisture content of biomass (%, wet basis)

3.3.1.3 Ash Content

Ash content of samples was measured according to the NREL Laboratory Analytical Procedure (NREL, 2005). Before sample preparation, crucibles were placed in a muffle furnace (model F6020C, Thermoscientific, Dubue, Iowa) at 575°C for 4 hours. Crucibles were then cooled to room temperature in a desiccator. Approximately 1 g of the samples that were ground through 1 mm screen size of the Wiley mill were placed in the crucibles. The crucible was subsequently placed in the furnace which was ramped according to this protocol: 1) ramped from room temperature to

105°C at 20°C/minute, 2) held for 12 mins at 105°C, 3) ramped to 250°C at 10°C/minute, 4) held at 250°C for 30 minutes, 5) ramped to 575˚C at 20˚C/minute, 6) held at 575˚C for 180 minutes, and 7) cooled to 105°C. The crucibles and ash were then transferred into a desiccator where they were kept for 1 hour to cool to room temperature before the mass of the crucible and ash were measured to the nearest 0.1 mg. Ash content on dry basis for the sample was computed as follows:

$$AC = \left(\frac{M_{ca} - M_c}{M_{cs} - M_c}\right) \times \left(\frac{100}{100 - M_w}\right) \times 100\% \tag{3.5}$$

where,

AC = percentage of ash in biomass (%, dry basis)

M_{ca} = mass of crucible and ash (g)

M_c = mass of crucible (g)

M_{cs} = mass of crucible and biomass (g)

M_w = moisture content of biomass (%, wet basis)

3.3.1.4 Energy Content

A bomb calorimeter (model C200, IKA Works Inc., Wilmington, N.C.) was used to measure the energy content of samples. About 1 g of sample was weighed and compressed into pellet form with the aid of a press (Model No.-C21, IKA Works Inc., Wilmington, N.C.). The pellet was then weighed and placed in the calorimeter crucible. The crucible was positioned in a vessel which was then pressurized with oxygen gas. After preparation, the equipment burned the sample in the presence of abundant oxygen. The energy content value for each sample was calculated by the calorimeter based upon the difference in the temperature of the surrounding water as a result of the biomass combustion.

3.3.2 Moisture, Storage Time, and Hammer mill Screen Size Effects on Grinding Characteristics

3.3.2.1 Sample Preparation

Three storage times were considered for this study. After each storage time (0 month, 2 months, or 4 months), woodchips were adjusted to five moisture levels (12%, 20%, 30%, 40%, and 50% wet basis). For fresh woodchips (0 month storage time), moisture adjustment was carried out by air-drying woodchips to these moisture content levels. For stored woodchips (2 month and 4 month storage times), the moisture adjustment was carried out by re-wetting. A predetermined quantity of water was added to and thoroughly mixed with the woodchips. The samples thereafter were enclosed in a labeled polythene bag, and kept in the refrigerator (temperature of 4±1°C) for a minimum of 10 days. This allowed for better uniformity of the moisture within the sample (Al-Mahasneh and Rababah 2007; Balasubramanian and Viswanathan, 2010). The moisture content of the samples were confirmed after equilibration period according to ASABE Standard S358.3 (ASABE Standards, 2012). The woodchips were then ground and analyzed.

3.3.2.2 Grinding Operation and Specific Grinding Energy

The hammer mill (model 10HBLPK, C.S. Bell Co., Tiffin, Ohio) (Figure 3.2) used in this study has 12 swinging beaters attached to its high speed shaft. The mill was powered by a 3 hp, 3-phase alternating current motor, with a rated speed of 1760 R.P.M. at 60 HZ (model C182T17FB29D, LEESON Electric Corporation, Grafton, Wisc.). Power was transmitted through belt and pulley arrangement, and hammer mill speed was maintained at 3450±50 rpm when loaded and unloaded. During grinding, the hammer mill was fitted with either a 3.175 mm screen or a 6.350 mm screen. Each experimental run was conducted with 3 kg of biomass that was fed into the mill within a 9.5±0.5 minutes time frame.

A wattmeter (model No.-A3314/02, EZ meter, Santa Ynez, Cal.) (Figure 3.3) was used to measure the electrical energy consumed during grinding. The energy required to run the hammer mill empty was initially measured and was referred to as parasitic energy. Then energy required to grind the 3 kg biomass samples at the various treatment combinations was determined as the change in wattmeter reading before and after grinding. The specific grinding energy was calculated as expressed in Equation 3.6 (Miao et al., 2011).

Figure 3.2. C.S. Bell Co. hammer mill used for grinding of woodchips.

Figure 3.3. Wattmeter used for grinding energy measurement.

$$E = \frac{E_t - E_p}{M_{dry}} \quad (3.6)$$

where

E = specific grinding energy (kJ/kg)

E_t = total energy consumed (kJ)

E_p = parasitic energy (kJ)

M_{dry} = dry matter mass of biomass (kg)

3.3.2.3 Moisture Loss during Grinding

Before and after grinding, the moisture content of the samples (woodchips and grinds) was determined according to ASABE Standard S358.3 (ASABE Standards, 2012) by drying about 10 g of samples in an oven for 24 hours. Moisture loss during grinding was calculated as shown in Equation 3.7.

$$MC_{loss} = \frac{(MC_i - MC_f)}{100 - (MC_i - MC_f)} \quad (3.7)$$

where

MC_{loss} = moisture loss during grinding (%, wet basis)

MC_i = moisture content of woodchips (%, dry basis)

MC_f = moisture content of grinds (%, dry basis)

3.3.2.4 Bulk Density

Bulk density of grinds was measured as received from hammer mill (wet bulk density) and after oven drying (oven-dried bulk density). Since samples lost moisture during grinding, the bulk density of oven-dried samples was carried out to nullify the effect of moisture content on bulk density. Bulk density measurement was carried out according to ASTM E 873 – 82 Standard (ASTM, 2006). A bulk density measurement apparatus (Burrows Co., Evanston, Ill.) was used. The apparatus consist of a 1137 mm^3 metal container that was filled with free falling sample through a funnel positioned at a 0.6 m height above the top edge of the container. The container was carefully leveled after filling. The mass of the sample (M_s) that filled the container was determined and used in calculating the bulk density (ρ_b) of the grinds, as expressed in Equation 3.8 (Fasina, 2007). Figure 3.4 show the bulk density apparatus used for grinds.

$$\rho_b = \frac{M_s}{V_c} \tag{3.8}$$

where

ρ_b = bulk density of grinds (kg/m^3)

M_s = mass of grinds that filled the container (kg)

V_c = volume of container (1137 m^3)

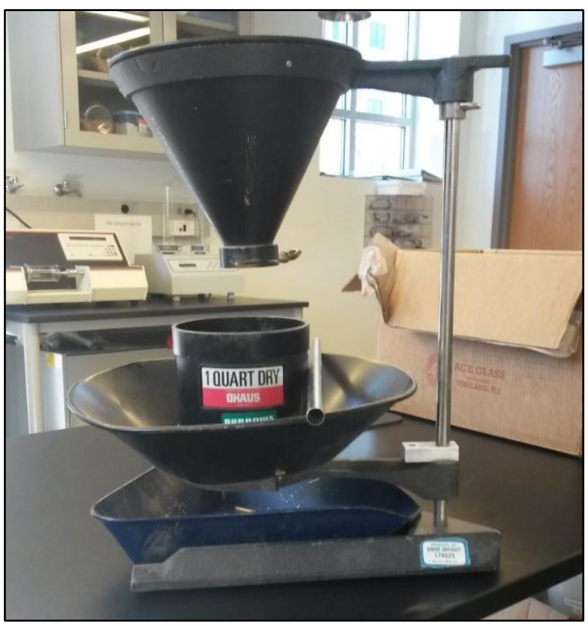

Figure 3.4. Apparatus used for bulk density measurement.

3.3.2.5 Particle Size Distribution

Sieve analysis was used to determine the particle size distribution of grinds according to ASABE Standard S319.4 (ASABE Standards, 2008). Approximately 100 g of sample was screened for 15 minutes with a Ro-Tap test sieve shaker (model RX – 29, W.S. Tyler, Mentor, Ohio) and 10 ISO sieve set (Figure 3.5). The screen set used for sieve analysis is shown in Table 3.1. These screens sets were selected to ensure that more than 95% of the sample passed through the topmost screen and less than 5% was retained on the pan.

The mass retained on the screens was plotted against the nominal diameter of the screens to form the particle size distribution. Geometric mean diameter, geometric standard deviation, and coefficient of variation were calculated from the mass retained on each screen according to Equations 3.9 – 3.13 (ASABE Standards, 2003).

Figure 3.5. W.S. Tyler Ro-Tap sieve shaker used for particle size distribution measurement.

Table 3.1. Hammer mill screen size and sieves used for particle size distribution analysis.

Hammer mill Screen Size (mm)	Sieve Mesh Number												
3.175*	N/U	N/U	8	12	18	25	40	60	120	N/U	170	270	Pan
6.35*	4	5	8	12	18	25	40	60	N/U	140	N/U	N/U	Pan
Equivalent Size (mm) of Sieve	4.76	4.00	2.38	1.68	1.00	0.71	0.42	0.25	0.13	0.11	0.09	0.05	---

*N/U means sieve was not used.

$$\bar{d}_i = \sqrt{(d_i \times d_{i+1})} \tag{3.9}$$

$$d_{gw} = log^{-1}\left[\frac{\sum_{i=1}^{n} W_i \ \log \bar{d}_i}{\sum_{i=1}^{n} W_i}\right] \tag{3.10}$$

$$S_{log} = \left[\frac{\sum_{i=1}^{n} W_i \left(\log \bar{d}_i - \log d_{gw}\right)^2}{\sum_{i=1}^{n} W_i}\right]^{1/2} \tag{3.11}$$

$$S_{gw} = \frac{1}{2}\, d_{gw}\left[log^{-1} S_{log} - \left(log^{-1} S_{log}\right)^{-1}\right] \tag{3.12}$$

$$CV = 50\,\frac{(d_{84} - d_{16})}{d_{50}} \tag{3.13}$$

where

d_{gw} = geometric mean diameter of particles by mass (mm)

W_i = mass on i^{th} screen (g)

$\bar{d}_i$ = geometric mean diameter of particles on i^{th} screen (mm)

d_i = nominal screen aperture size of the i^{th} screen (mm)

d_{i+1} = nominal screen aperture size in next larger than i^{th} screen, just above in a set (mm)

S_{gw} = geometric standard deviation of particle diameter by mass (mm)

S_{log} = geometric standard deviation of log-normal distribution by mass in ten-based logarithm (dimensionless)

d_{84} = particle diameter at 84% probability (mm)

d_{50} = particle diameter at 50% probability, d_{gw} (mm)

d_{16} = particle diameter at 16% probability (mm)

CV = coefficient of variation

3.3.3 Experiment Design and Data Analysis

A split plot experimental design was adopted. Figure 3.6 shows the experimental setup for each storage time. Microsoft Excel 2013 (Microsoft Excel 2013, Microsoft Corporation, Redmond, Wash.) was used for data processing and formatting while SAS software (SAS 9.3, SAS Institute Inc., Cary, N.C.) was utilized to perform statistical analysis of data. All statistical significance testing were carried out at a confidence level of 95%. SAS procedures used in this study includes PROC MEANS and PROC CORR. PROC NLIN was used to develop a non-linear model for predicting specific grinding energy using storage time, moisture content, and particle size distribution. PROC REG was used to develop regression models. Furthermore, PROC TTEST was used to check for significance of the difference between wet and oven-dried bulk densities.

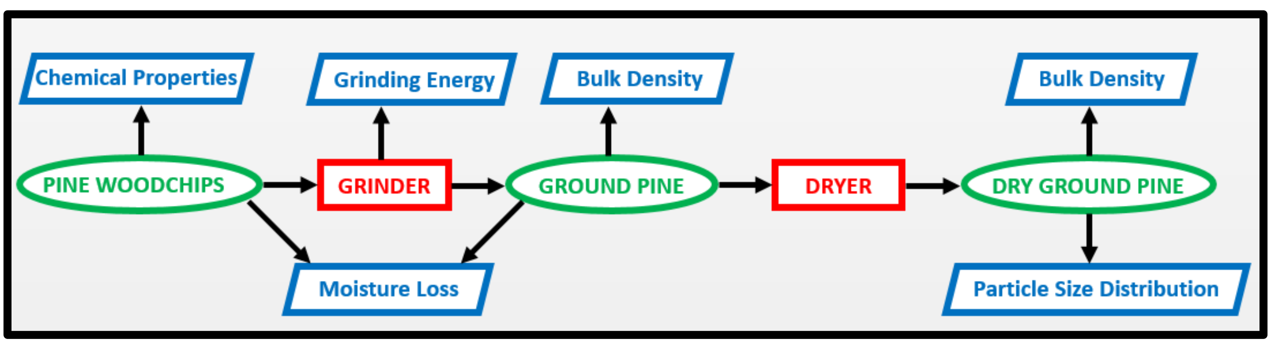

Figure 3.6. Experimental setup.

3.4 Results and Discussion

3.4.1 Storage Time Effect on Chemical Properties

3.4.1.1 Volatile Matter Content

There was a significant decrease ($p < 0.05$) in volatile matter content of woodchips with increase in storage time (Figure 3.7). Mean percentage volatile matter content decreased from 86.74% to

85.47% for a storage time of 6 months. Equation 3.14 shows that the volatile matter content of loblolly pine woodchips linearly reduced with storage time. This trend is in agreement with the work of Casal et al. (2010) on the storage of pine woodchips where volatile matter content (initially at 85.00%) was reduced by about 3% during 12 months of storage. Also, the values of volatile matter content measured in this study are similar to the values reported for loblolly pine by Wadkins et al. (2013) (85.40%) and Hehar et al. (2014) (82.36%).

This reduction in volatile matter content with storage time has also been reported for biomass pellets—often referred to as off-gassing or out-gassing (Yazdanpanah et al., 2014; He et al., 2012). Chemical and biological degradation of biomass occur during storage resulting in emission of carbon monoxide, carbon dioxide, and methane. The rate of release of these gases accelerates with increase in temperature (Levitt et al., 1995). In addition, Svedberg et al. (2004) suggests that carbon monoxide emission during off-gassing might be as a result of autoxidation of fats and fatty acids. They also reported the formation of hexanaldehyde — a major straight chain alkanal group in volatile matter.

The evidence of volatile matter release during storage of loblolly pine woodchips suggests the need to ensure that adequate safety measures are taken during storage of loblolly pine, especially in enclosed facilities, as emitted volatiles may contain CH_4—a main components of natural gas that can potentially cause fire and explosion. Emitted volatiles also pose health and occupational hazards to storage facility workers (Svedberg et al., 2004).

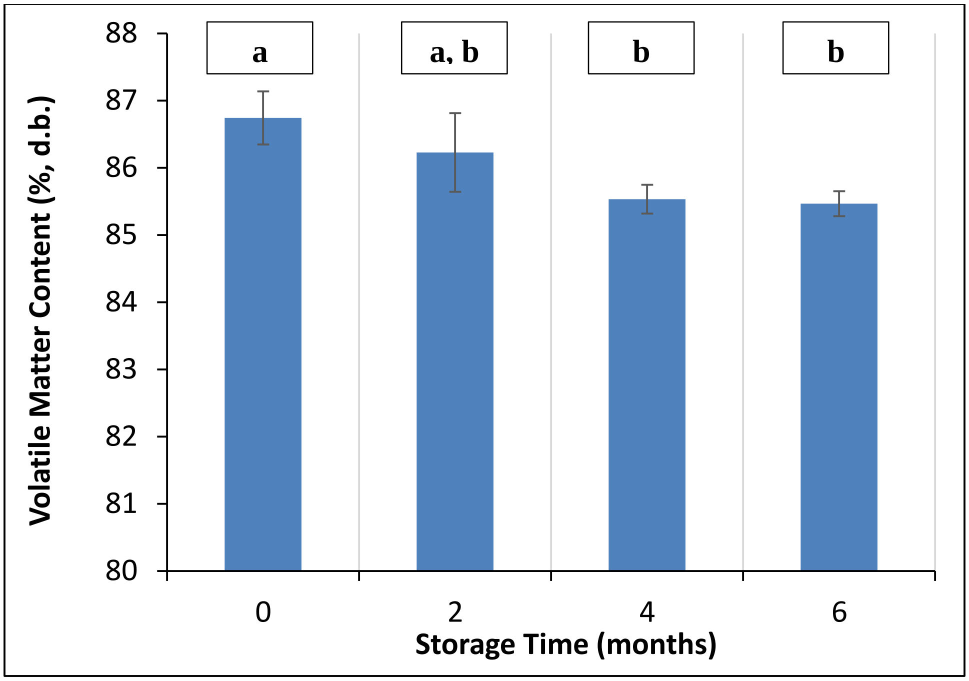

Figure 3.7. Effect of storage time on volatile matter content of loblolly pine (means with different letters are significantly different at 0.05 significance level using Tukey's multiple comparison).

$$y = 86.671 - 0.226x \qquad [r^2 = 0.685] \qquad (3.14)$$

where

y = volatile matter content (%, dry basis)

x = storage time (months)

3.4.1.2 Ash Content

Ash content of the loblolly pine woodchips in this study varied between 0.31% and 0.46% during the storage period. This is comparable to the values reported by Naimi et al. (2012) for loblolly pine (0.45%) and Douglas fir (0.43%) but lower than the value reported by Mani et al. (2004) for herbaceous biomass (wheat straw, 8.32%; barley straw, 10.72%; corn stover, 7.46%; and switchgrass, 5.49%). Lower ash content increases energy content (Fasina, 2006) and reduces fouling and slagging during conversion (Rizvi et al., 2015).

Figure 3.8 illustrates that a significant increase ($p < 0.05$) in ash content occurred as storage time increased from 0 to 6 months. We do not believe that the increase is a result of the formation of ash. This increase is probably due to compositional percentile changes because of the decrease in volatile matter content (Figure 3.7). Similar relationships were reported by Casal et al. (2010) and Afzal et al. (2010). While Casal et al. (2010) reported an increase of about 1.4% in ash content of pine woodchips during a storage period of 12 months, Afzal et al. (2010) reported an increase from 0.43% to 1.06% and 1.09% for covered and uncovered white birch woodchips pile. Equation 3.15 shows that the relationship between average ash content and storage time for loblolly pine woodchips was linear.

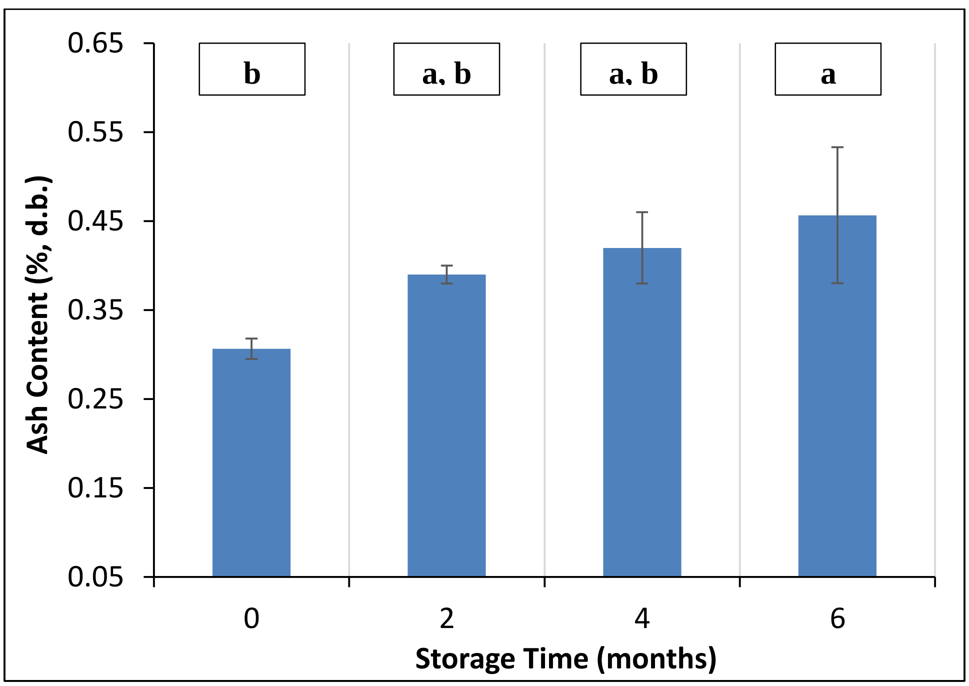

Figure 3.8. Effect of storage time on ash content of loblolly pine (means with different letters are significantly different at 0.05 significance level using Tukey's multiple comparison).

$$y = 0.321 + 0.024x \qquad [r^2 = 0.664] \qquad (3.15)$$

where

y = ash content (%, dry basis)

x = storage time (months)

3.4.1.3 Energy Content

At the start of the storage period energy content of pine woodchips was 20.32 MJ/kg. However, at the end of the 6 months storage time, energy content reduced significantly ($p < 0.05$) to 20.111 MJ/kg. Carter et al. (2013) also reported 20.18 MJ/kg for loblolly pine. Figure 3.9 shows this energy degradation which is in agreement with results reported in published literature. Casal et al. (2010) stored pinewood chips for 12 months in pile and reported that its lower heating value (LHV) decreased from 18.85 MJ/kg to 18.28 MJ/kg. Also, Afzal et al. (2010) reported a reduction (although insignificant at a significance level of 0.05) in energy content of white birch stored in bundles and as covered, underneath plastic sheath, and uncovered woodchips piles in their study. Jirjis (2005) observed a decline in energy content for willow chips stored in pile from 19.57 MJ/kg to 19.48 MJ/kg in 2 months.

Changes in energy content may be a reflection of the reduction in volatile matter content (Figure 3.7) because lower volatile matter content represents lower quantity of combustible constituent. The relationship between volatile matter content and energy content was expressed by a linear equation (Equation 3.16).

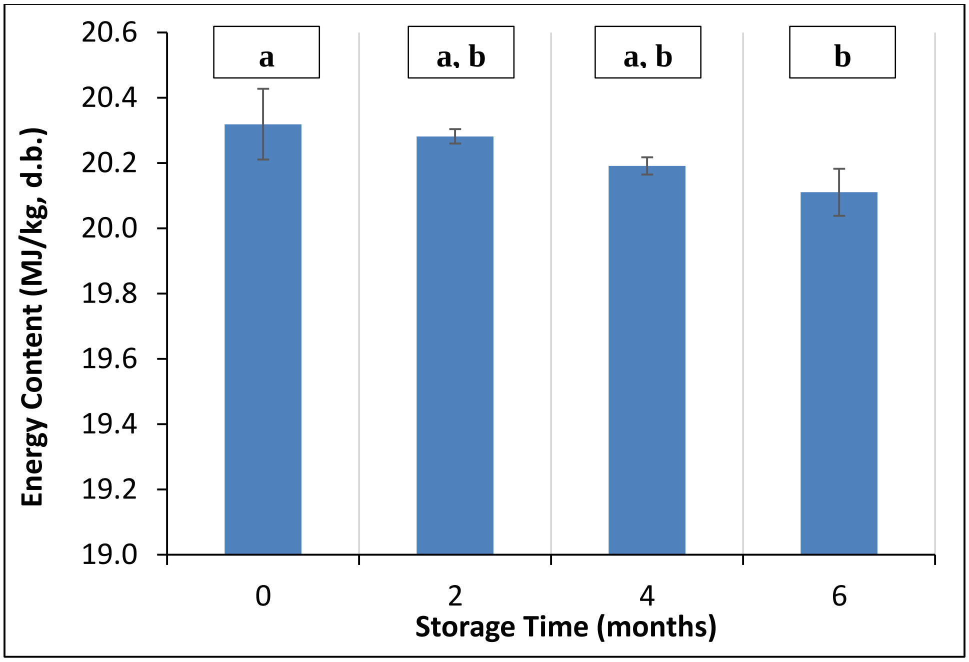

Figure 3.9. Effect of storage time on energy content of loblolly pine (means with different letters are significantly different at 0.05 significance level using Tukey's multiple comparison).

$$y = 20.333 - 0.036x \qquad [r^2 = 0.668] \qquad (3.16)$$

where

y = energy content (MJ/kg, dry basis)

x = storage time (months)

3.4.2 Moisture, Storage Time, and Hammer mill Screen Size Effects on Grinding Characteristics

3.4.2.1 Moisture Loss during Grinding

Heat generated in the grinding chamber resulted in increase in the temperature of woodchips and grinds and consequently resulted in moisture loss that increased as initial moisture content of woodchips increased (Figure 3.10). This is in agreement with the report of Probst et al. (2013) where greater moisture loss was noticed for corn and corncobs with higher moisture content. In their study, the authors reported that moisture loss during grinding reduced from 9.38% to 1.80% for corncobs and 2.41% to 0.07% for corn when initial moisture content of feedstock decreased from ~20% to ~10%. Moisture loss also occurred in this study, with the moisture loss significantly decreasing ($p < 0.05$) with increase in screen size and with decrease in woodchips moisture content (Figure 3.10; Table 3.2). The amount of moisture loss during grinding ranged from 3.14% to 36.52% for woodchips ground through 3.175 mm and from 1.09% to 20.56% for woodchips ground through 6.350 mm. This is because the duration of recirculation of grinds before passing through the hammer mill screen was shorter (hence, the extent of impact and heat generated was smaller) when grinding through 6.350 mm screen size. The combined increase in moisture loss during grinding with increasing initial moisture content of woodchips and decreasing hammer mill screen size caused a significant interaction ($p < 0.05$) between moisture content of woodchips and hammer mill screen size (Table 3.2).

Mean moisture loss significantly increased ($p < 0.05$) with increasing storage time from 12.01 % (0 month) to 14.22% (4 month). However, pairwise comparison showed that there was no significant difference ($p < 0.05$) in moisture loss for samples stored for 2 months and those stored for 4 months. We suspect that the variability in ambient temperature and relative humidity during grinding may have contributed to the changes in the moisture loss.

The results obtained from moisture loss during grinding may be used in the selection of initial moisture content of woodchips prior to grinding, in the mass balance calculations during grinding operation, and in the determination of energy requirements during post-grinding drying operation.

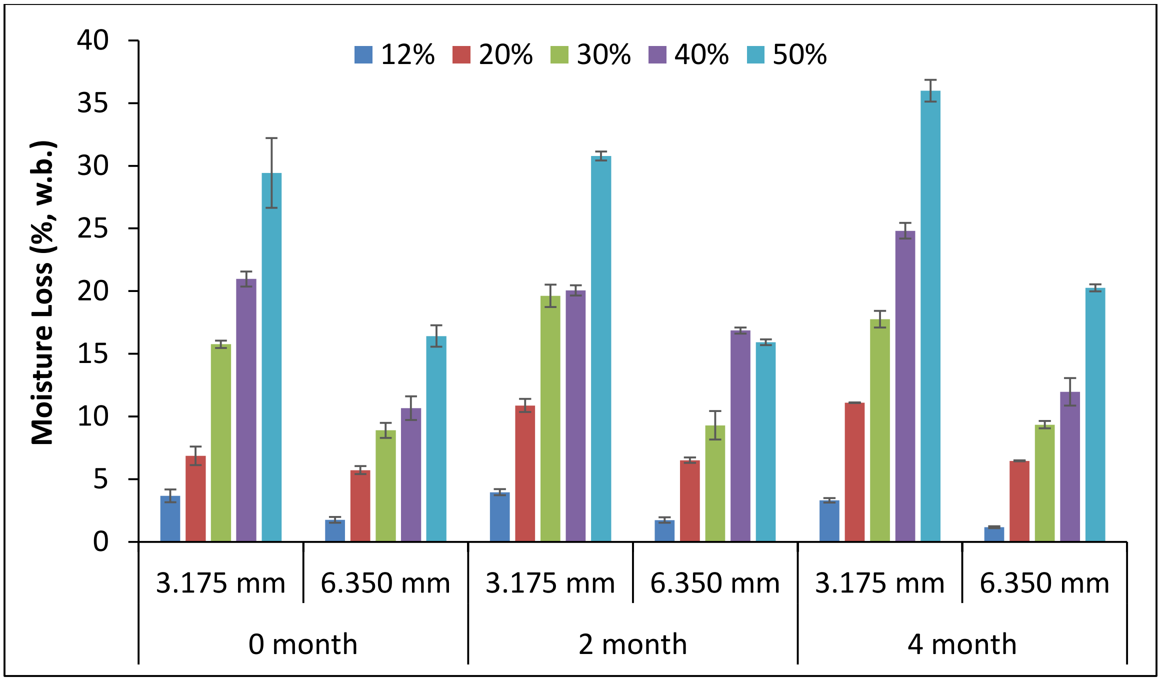

Figure 3.10. Effect of moisture content, storage time, and screen size moisture loss during grinding.

Table 3.2. ANOVA result of moisture, storage time, and screen size effect on moisture loss during grinding.

Effect	**Num DF[a]**	**Den DF[b]**	**F Value**	**Pr > F**
Storage Time	2	6	13.4	0.0061
Moisture Content (MC)	4	72	459.82	<.0001
Screen Size (SS)	1	72	436.07	<.0001
MC*SS	4	72	38.5	<.0001

[a]Num DF means numerator degree of freedom
[b]Den DF means denominator degree of freedom

3.4.2.2 Bulk Density

Figure 3.11 shows that the oven-dried bulk density of loblolly pine grinds varied from 273.64 kg/m^3 (for samples ground at 12% moisture content and through 3.175 mm screen size) to 106.03 kg/m^3 (for samples ground at 50% moisture content and through 3.175 mm screen size). Results reported by Oginni (2014) and Wadkins et al. (2013) (232.51 kg/m^3 and 217.10 kg/m^3, respectively) for loblolly pine are comparable to the value for the grinds (ground through 3.175

mm screen size) in this study. ANOVA result (Table 3.3) showed that oven-dried bulk density was not significantly affected ($p < 0.05$) by storage time. The oven-dried bulk densities at the storage times were therefore averaged and referred to as mean oven-dried bulk density.

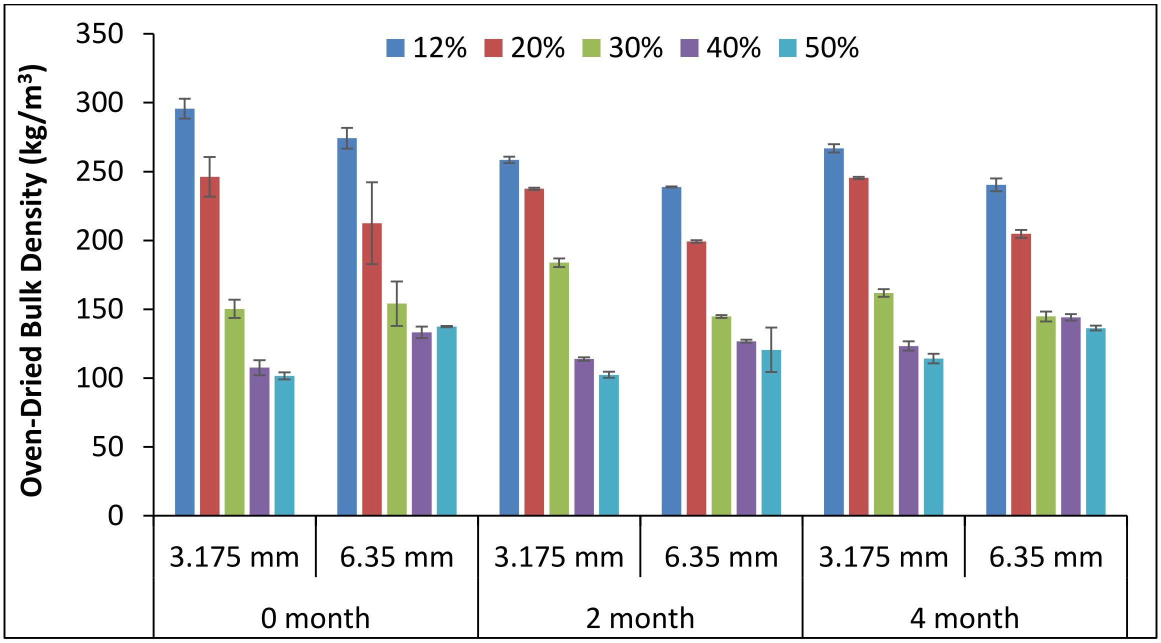

Figure 3.11. Effect of moisture content, storage time, and screen size on oven-dried bulk density of grinds.

Table 3.3. ANOVA result of moisture, storage time, and screen size effect on oven-dried bulk density of loblolly pine grinds.

Effect	Num DF[a]	Den DF[b]	F Value	Pr > F
Storage Time	2	6	3.95	0.0806
Moisture Content (MC)	4	72	494.16	<.0001
Screen Size (SS)	1	72	6.44	0.0133
MC*SS	4	72	23.41	<.0001

[a]Num DF means numerator degree of freedom
[b]Den DF means denominator degree of freedom

The moisture content of woodchips had significant effect ($p < 0.05$) on the oven-dried bulk density of grinds (Table 3.3). The mean oven-dried bulk density of grinds reduced with increase in moisture content of woodchips (Figure 3.12) because as the moisture content of wood chips increased, ground particles produced were more fluffy and woolly (Figure 3.13). Lehmann et al. (2012) reported similar observation for comminution of wet miscanthus and woodchips (sawmill by-product. This fluffiness caused high inter-particle porosity which resulted in low bulk density of the grinds. The implication of this is that it will be more expensive to transport products ground at higher moisture content than it would be for those ground at lower moisture content (Schroeder et al., 2007). A polynomial relationship was established between oven-dried bulk density and the moisture content of woodchips (Figure 3.12).

Screen size negatively correlates with oven-dried bulk density. Therefore, the higher the screen size, the lower the oven-dried bulk density of grinds (Figure 3.12). This is because smaller particles fill more inter-particle pore spaces than larger particles. Hence, higher bulkiness is observed for products ground through lower screen size. Miao et al. (2011) also observed that the bulk density of miscanthus, switchgrass, and willow grinds decreased with increase in hammer mill screen size. In a similar study, Mani et al. (2004) reported an increase in bulk density of wheat straw grinds from 77 kg/m^3 (geometric mean diameter of 1.43 mm) to 115 kg/m^3 (geometric mean diameter of 0.25 mm).

There was a significant interaction ($p < 0.05$) between moisture content and hammer mill screen size (Table 3.3). Grinds produced through hammer mill screen size of 3.175 mm have higher oven-dried bulk density at lower moisture content (12% – 30%). The reverse is the case at moisture levels of 40% and 50% (Figure 3.12). This significant cross-over interaction was because fluffiness of grinds increased with both decreasing hammer mill screen size and increasing moisture content of woodchips.

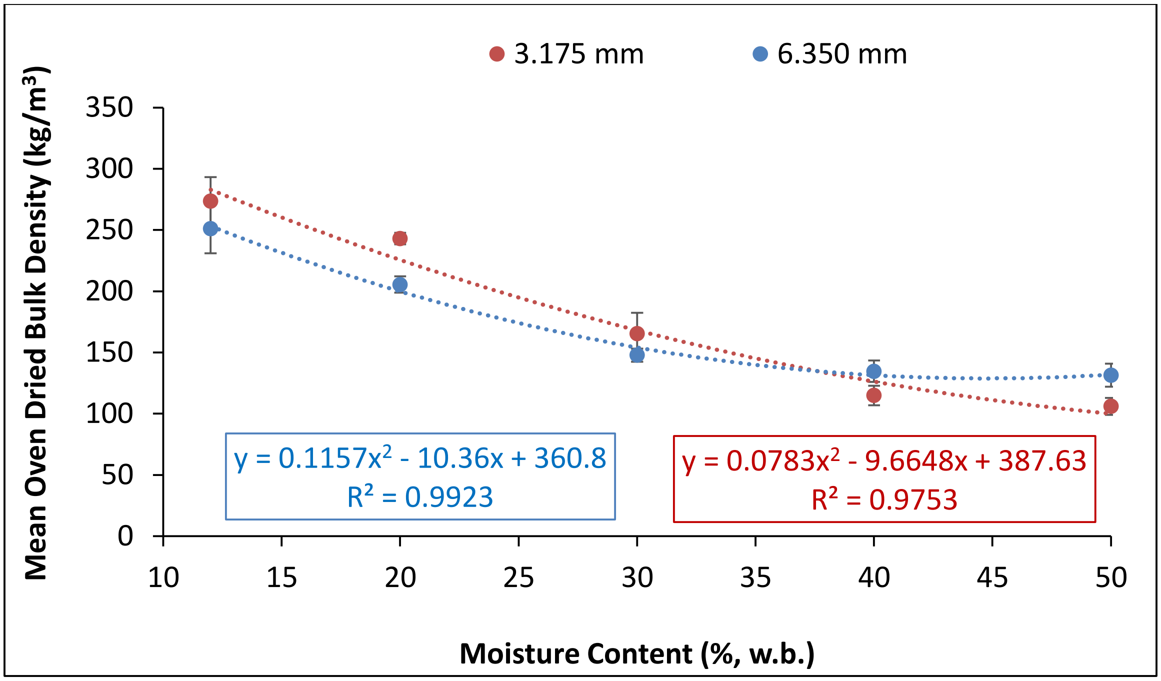

Figure 3.12. Effect of woodchips moisture content and screen size on oven-dried bulk density of grinds.

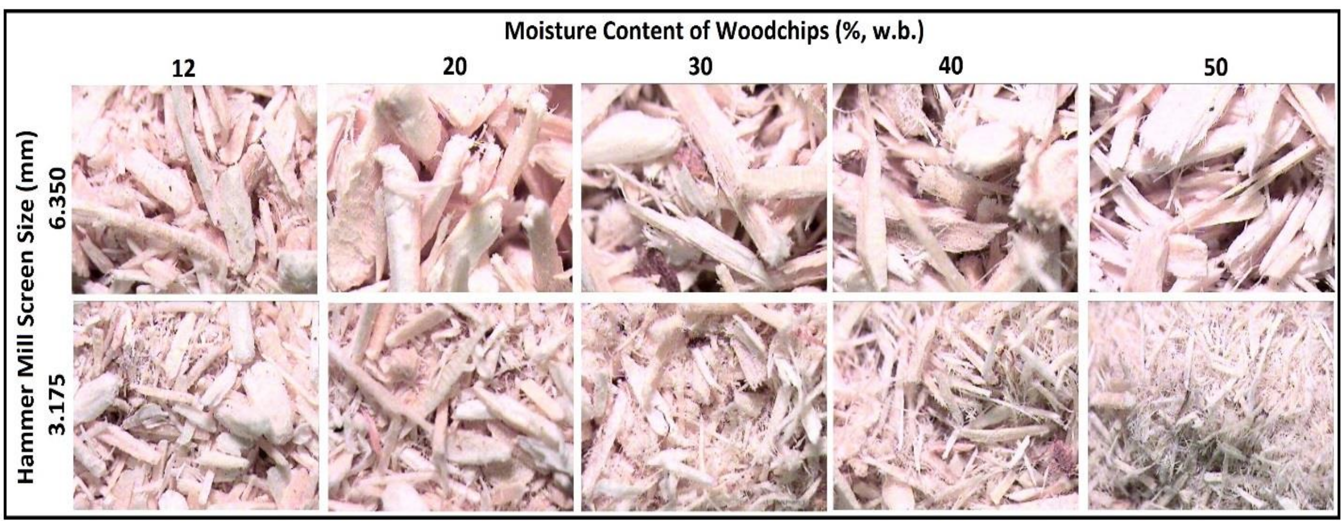

Figure 3.13. Effect of woodchips moisture content on morphology of grinds.

Table 3.4 compares the wet bulk density to the oven-dried bulk density of the grinds. The following were observed between the wet bulk density and oven-dried bulk density of grinds:

a) There was no significant difference ($p < 0.05$) between the wet bulk density and oven-dried bulk density for grinds produced from woodchips at 12% moisture content (3.175 mm and 6.350 mm screen size). This result was because the moisture content of these grinds was low (ranged from 8.01% to 10.67%). Oginni (2014) also reported that there was no significant change in the bulk density of loblolly pine as moisture content reduced from 8.69 % to 4.78%.

b) Oven-dried bulk density was higher than wet bulk density for grinds produced from woodchips at 20% moisture content (3.175 mm and 6.350 mm screen size) and grinds produced from woodchips at 30% moisture content (3.175mm screen size only). A possible explanation for this observation is that the moisture in woody biomass at fiber saturation point (about 25% moisture content, wet basis) and below enter the fiber cells wall, and therefore expands the fiber cell. Hence, biomass has an increase in volume and lower bulk density.

c) Oven-dried bulk density was lower than wet bulk density of grinds when moisture content of woodchips was greater than 30%. This observation is in agreement with Hollenbach et al. (1982) equation (Equation 3.17), which expresses the relationship between wet bulk density and oven-dried bulk density of samples as a mixture equation (Lam et al., 2008). A Pearson correlation of 0.939 was established when the result from this study was compared with Equation 3.17 (Figure 3.14). Therefore, the higher the moisture content of grinds, the higher their bulk density. This result implies that the mass increase due to moisture addition exceeds the volumetric expansion. Increase in moisture above fiber saturation point goes into the cell lumen, and does not result in expansion.

Table 3.4. Wet and oven-dried bulk densities of loblolly pine grinds.

Storage Time (month)	Screen Size (mm)	Moisture Content (%, w.b.)	Wet Bulk Density* (kg/m^3)	Oven-dried Bulk Density* (kg/m^3)
0	3.175	12	$290.90^a \pm 23.55$	$295.64^a \pm 7.18$
		20	$226.13^b \pm 14.37$	$265.65^a \pm 13.27$
		30	$150.30^b \pm 6.56$	$166.13^a \pm 5.00$
		40	$132.20^a \pm 3.56$	$107.61^b \pm 5.41$
		50	$113.12^a \pm 1.52$	$101.62^b \pm 2.51$
	6.350	12	$275.24^a \pm 13.36$	$274.25^a \pm 7.58$
		20	$212.49^b \pm 29.86$	$234.01^a \pm 16.14$
		30	$177.81^a \pm 2.05$	$154.12^b \pm 16.15$
		40	$169.58^a \pm 4.23$	$133.15^b \pm 4.16$
		50	$198.14^a \pm 2.54$	$137.43^b \pm 0.43$
2	3.175	12	$253.78^a \pm 1.05$	$258.50^a \pm 2.35$
		20	$246.84^b \pm 0.76$	$237.52^a \pm 0.87$
		30	$167.91^b \pm 0.64$	$183.85^a \pm 3.16$
		40	$128.56^a \pm 1.45$	$113.89^b \pm 1.26$
		50	$110.30^a \pm 1.38$	$102.36^b \pm 2.18$
	6.350	12	$241.09^a \pm 0.98$	$238.82^a \pm 0.42$
		20	$185.92^b \pm 2.06$	$199.25^a \pm 0.90$
		30	$159.07^a \pm 1.48$	$144.77^b \pm 1.08$
		40	$146.64^a \pm 1.15$	$126.73^b \pm 1.05$
		50	$169.55^a \pm 1.98$	$120.49^b \pm 16.15$
4	3.175	12	$265.88^a \pm 3.34$	$266.88^a \pm 2.99$
		20	$237.92^b \pm 2.73$	$245.39^a \pm 0.88$
		30	$157.39^b \pm 3.37$	$162.01^a \pm 2.83$
		40	$135.67^a \pm 1.32$	$123.43^b \pm 3.37$
		50	$122.18^a \pm 4.06$	$114.28^b \pm 3.45$
	6.350	12	$240.30^a \pm 6.24$	$240.51^a \pm 4.67$
		20	$189.68^b \pm 2.52$	$204.90^a \pm 2.84$
		30	$160.63^a \pm 5.57$	$144.92^b \pm 3.52$
		40	$165.51^a \pm 2.14$	$144.28^b \pm 2.34$
		50	$183.45^a \pm 0.27$	$136.43^b \pm 1.71$

*Value in each cell = estimate ± standard deviation. Values in each row with same letter (a-b) are not significantly different ($p<0.05$)

$$\rho_b = \rho_d(1 + M_w) \tag{3.17}$$

where

ρ_b = bulk density of wet sample (kg/m^3)

ρ_d = oven-dried bulk density of sample (kg/m^3)

M_w = moisture content of sample (%, wet basis)

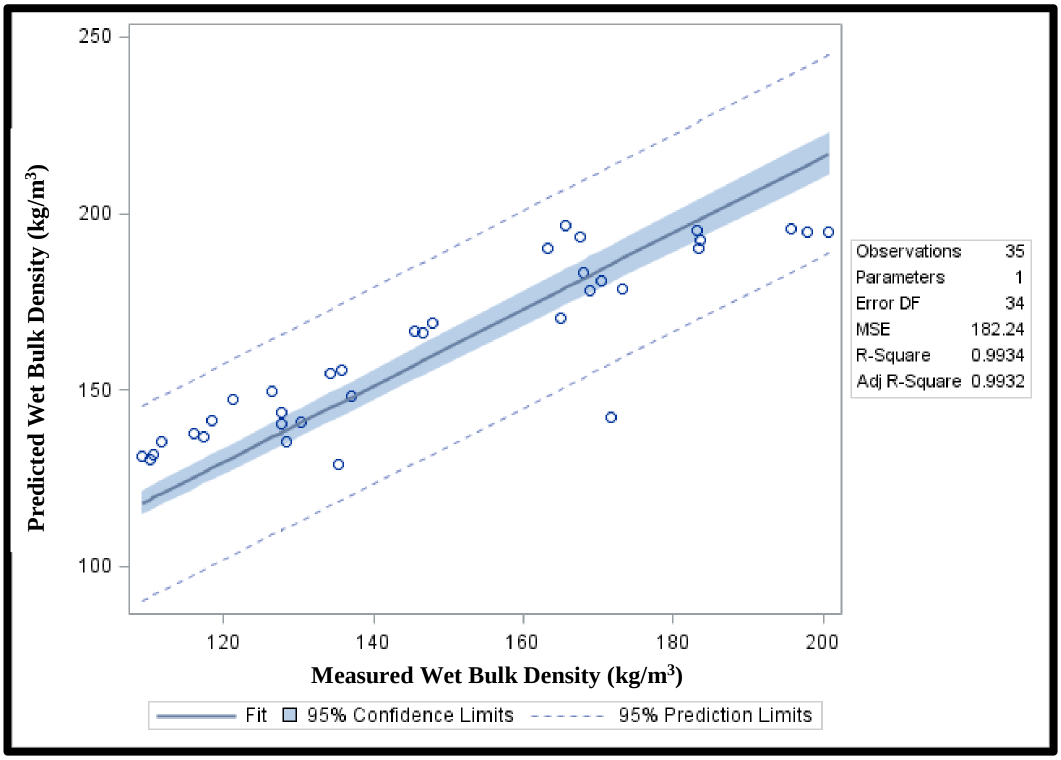

Figure 3.14. Comparison of measured and predicted wet bulk density for loblolly pine using Equation 3.17.

3.4.2.3 Particle Size Distribution

The particle size distribution (plot of mass percentage retained on screen against nominal screen size) for the grinds exhibited a slightly skewed normal distribution (Figures 3.15 – 3.17). This distribution behavior is similar to that reported by Ramachandriya et al. (2014) for Eastern redcedar, Phanphanich and Mani (2011) for loblolly pine, and Bergström et al. (2008) for Scots pine. The highest mass fraction was retained on sieve #18 (1.00 mm) for particles ground through 3.175 mm hammer mill screen size and on sieve #8 (2.380 mm) for particles ground through 6.350 mm hammer mill screen size (with the exception of woodchips at 12% moisture content).

The geometric mean diameter of grinds was significantly affected ($p < 0.05$) by moisture content (Table 3.5). Generally, geometric mean diameter of grinds increased with moisture content between 12% and 20%, and then decreased after 30% moisture level (Figure 3.18). The changes in geometric mean diameter can be attributed to the difference in fracture characteristics (Probst et al., 2013). As moisture increases, the ductility of the woodchips increases (Lewicki, 2004). This causes an increase in the particle size of resulting grinds. Similar result was reported by Dziki et al. (2012) for quince (the average particle size of grinds increased from 0.52 mm to 0.60 mm when the moisture content of feedstock increased from 10% to 14%). However, in this study, fluffy grinds which reduce the flow of materials through hammer mill screens are produced above 30% moisture level (Figure 3.13). This increased the contact time between the hammers and the sample, hence the size of grinds from woodchips above 30% moisture level are further reduced.

As expected, the geometric mean diameter significantly increased ($p < 0.05$) with increasing hammer mill screen size (Table 3.5). Samples ground through 6.350 mm screen size produced larger particles (geometric mean diameter of 1.323 mm to 2.042 mm) than those ground through 3.175 mm screen size (geometric mean diameter of 0.599 mm to 1.325 mm). Geometric mean diameter was also significantly affected ($p < 0.05$) by storage time (Table 3.5). Fresh woodchips (0 month storage time) produced the highest geometric mean diameter for both hammer mill screen sizes (Figure 3.18). This may be associated with the sample preparation procedure because moisture adjustment was achieved through drying for the fresh woodchips which is in contrast to the artificial moisture addition carried out for stored woodchips. It was also observed that the geometric mean diameter of grinds at 20% moisture content for fresh woodchips was drastically high compared to others. We cannot provide explanation for this unexpected behavior at this moisture level, but we suspect that this is because this moisture level is close to the fiber saturation point which is usually about 25% moisture content for loblolly pine wood, wet basis (Zelinka et al., 2012).

The variation in average geometric standard deviation of grinds obtained from hammer mill comminution was large (varied from 0.990 mm to 1.489 mm for 6.350 mm hammer mill screen size and 0.541 mm to 1.320 mm for 3.175 mm hammer mill screen size) (Table 3.5). Consequently, coefficient of variation of grinds was large and varied from 71.06% to 112.07%. Even though this wide distribution is suitable for densification operations such as pelletizing and cubing due to the ability of the small particles to fill the air space between the large particles (Tabil et al., 2011), it is a potential problem for processes that are dependent on uniformity of the size of particles (e.g. fluidization, hydrolysis, and gasification) (Mani et al., 2004). Wide distribution has been reported for alfalfa grinds by Ghorbani et al. (2010) (coefficient of variation ranged from 48.89% to 104.98%). Also, Bitra et al. (2009b) reported that the coefficient of variation for grinds in their study on particle size characteristics of switchgrass, wheat straw, and corn stover ground using hammer mill was between 232.53% and 560.47%.

The coefficient of variation of grinds obtained in this study ranged from 66.31% to 112.73%. It was significantly affected ($p < 0.05$) by hammer mill screen size. As hammer mill screen increased, the coefficient of variation reduced (Figure 3.19). Also, it was observed that coefficient of variation was significantly affected ($p < 0.05$) by storage time and moisture content (Table 3.5).

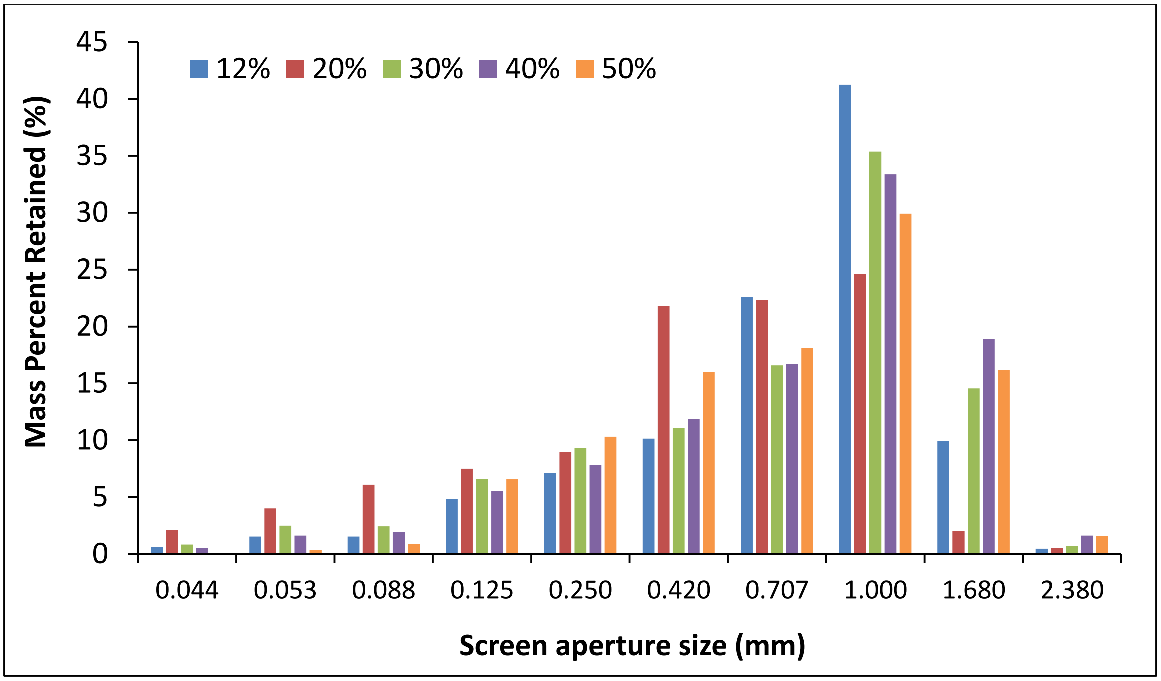

Figure 3.15a. Effects of moisture content on size distribution for oven-dried grinds obtained from fresh woodchips (3.175 mm hammer mill screen size).

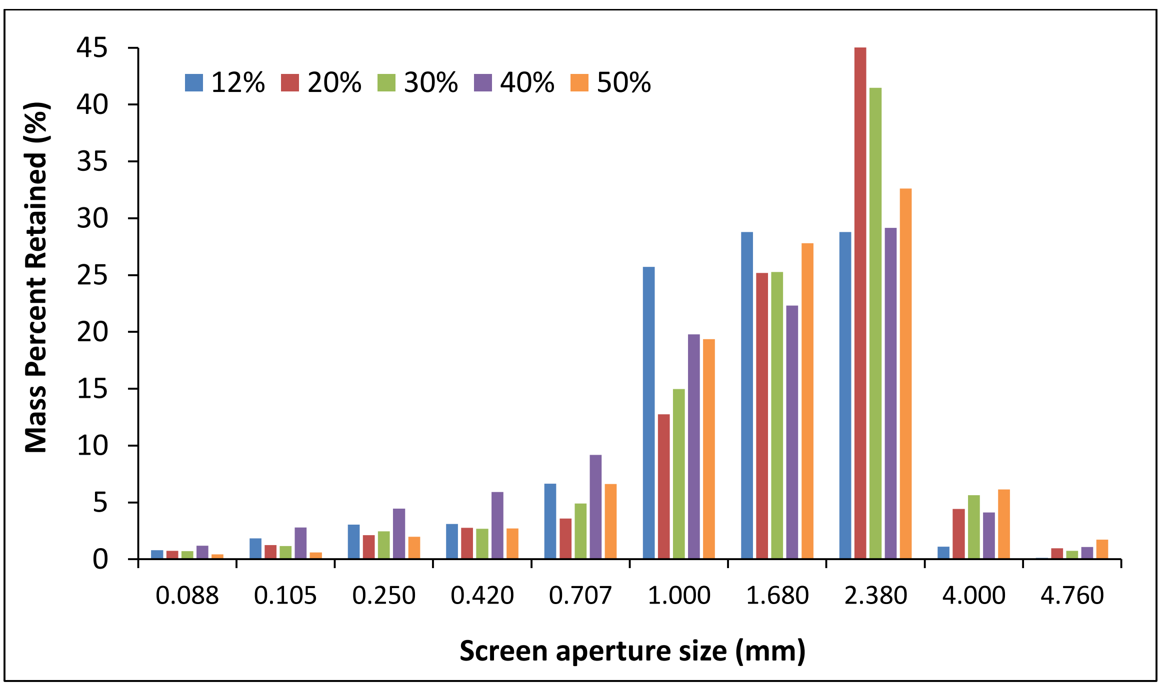

Figure 3.15b. Effects of moisture content on size distribution for oven-dried grinds obtained from fresh woodchips (6.350 mm hammer mill screen size).

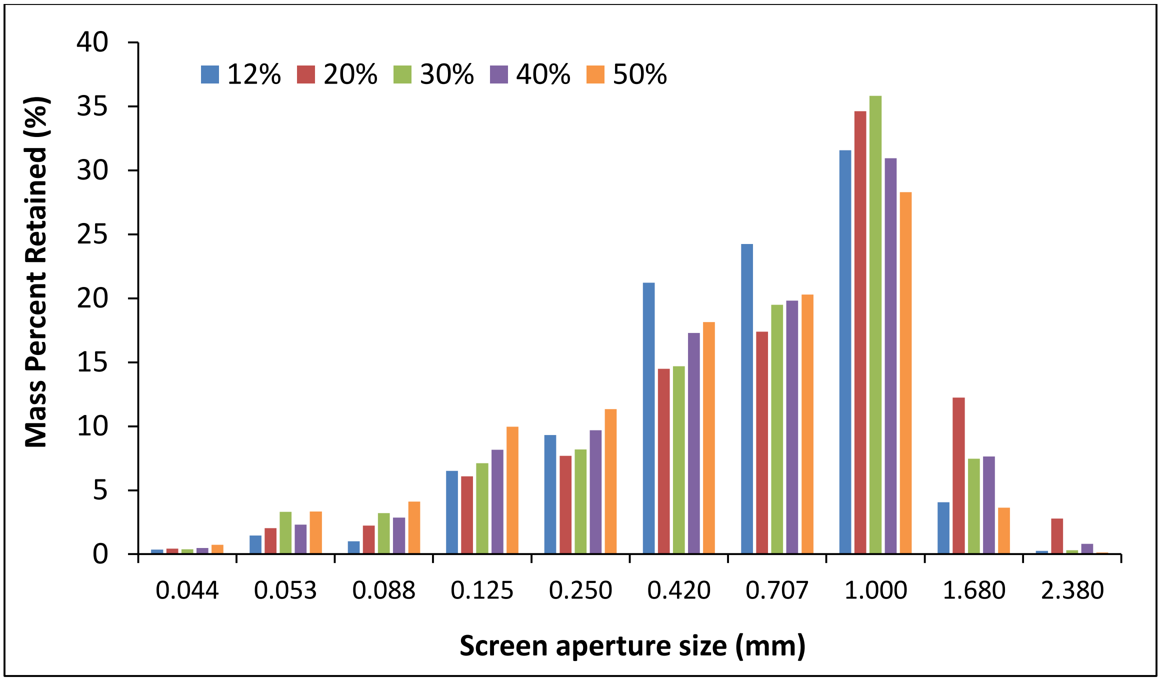

Figure 3.16a. Effects of moisture content on size distribution for oven-dried grinds obtained from woodchips stored for 2 month storage time (3.175 mm hammer mill screen size).

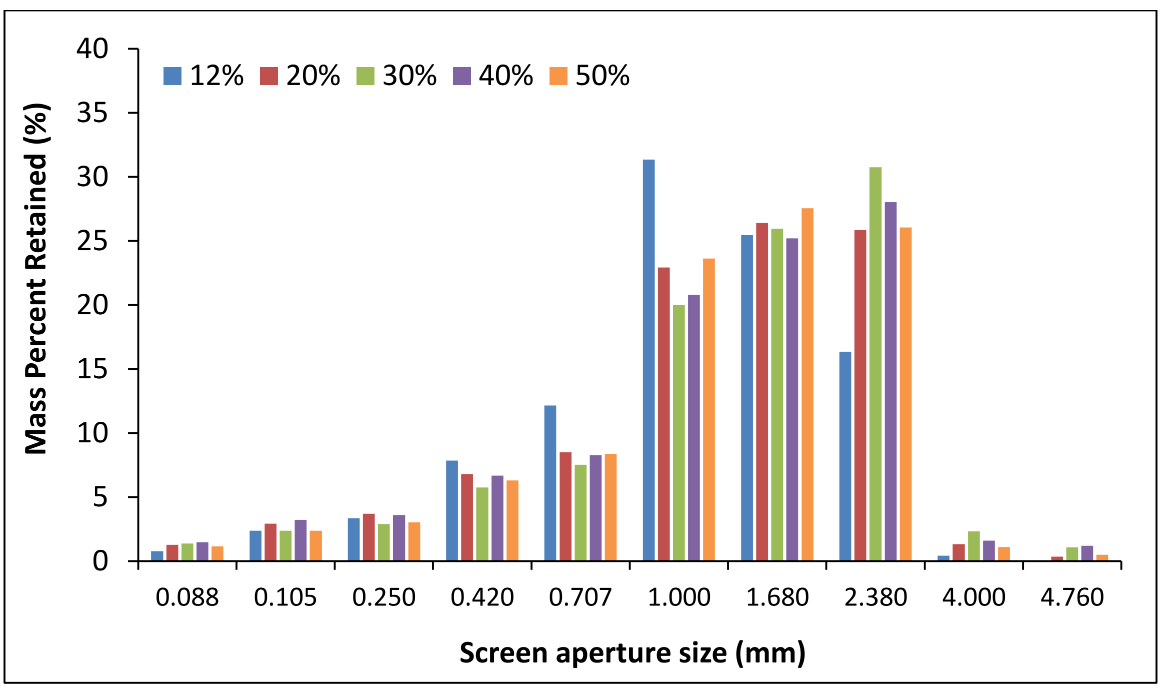

Figure 3.16b. Effects of moisture content on size distribution for oven-dried grinds obtained from woodchips stored for 2 month storage time (6.350 mm hammer mill screen size).

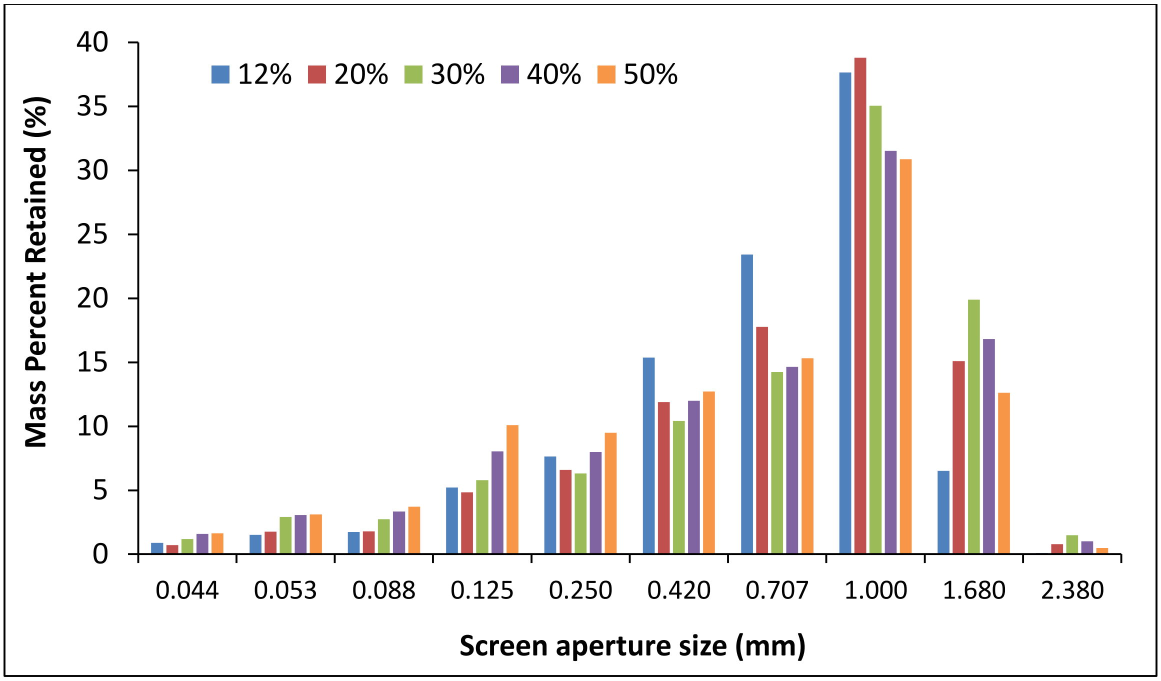

Figure 3.17. Effects of moisture content on size distribution for oven-dried grinds obtained from woodchips stored for 4 month storage time (3.175 mm hammer mill screen size).

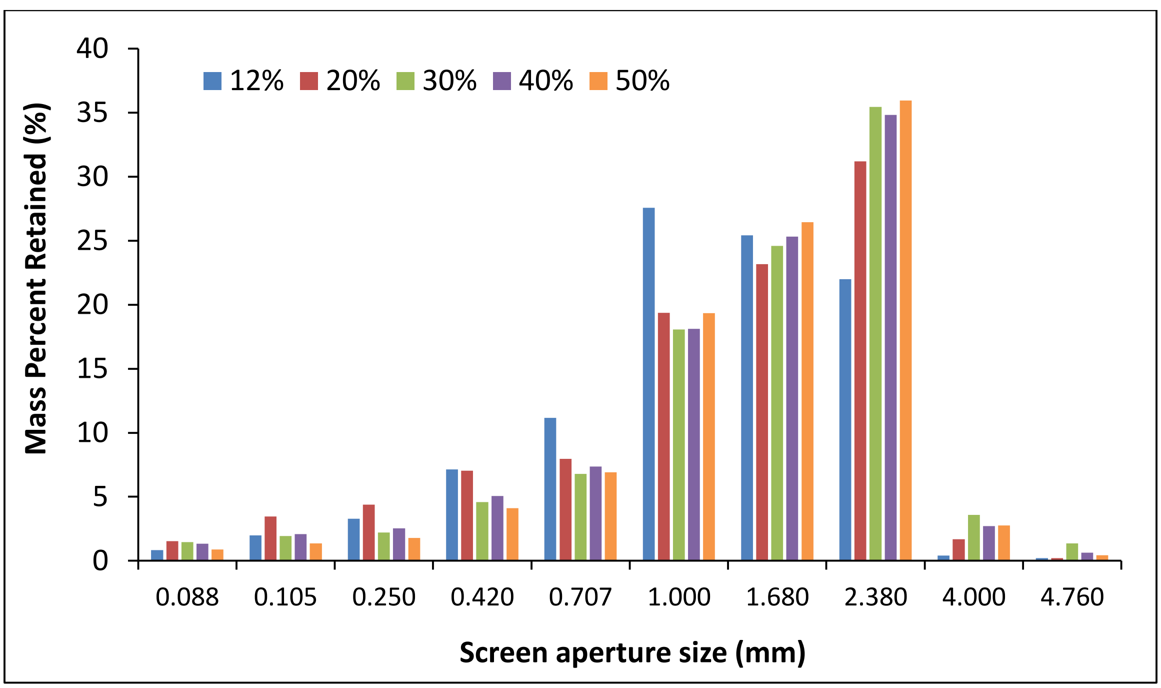

Figure 3.17. Effects of moisture content on size distribution for oven-dried grinds obtained from woodchips stored for 4 month storage time (6.350 mm hammer mill screen size).

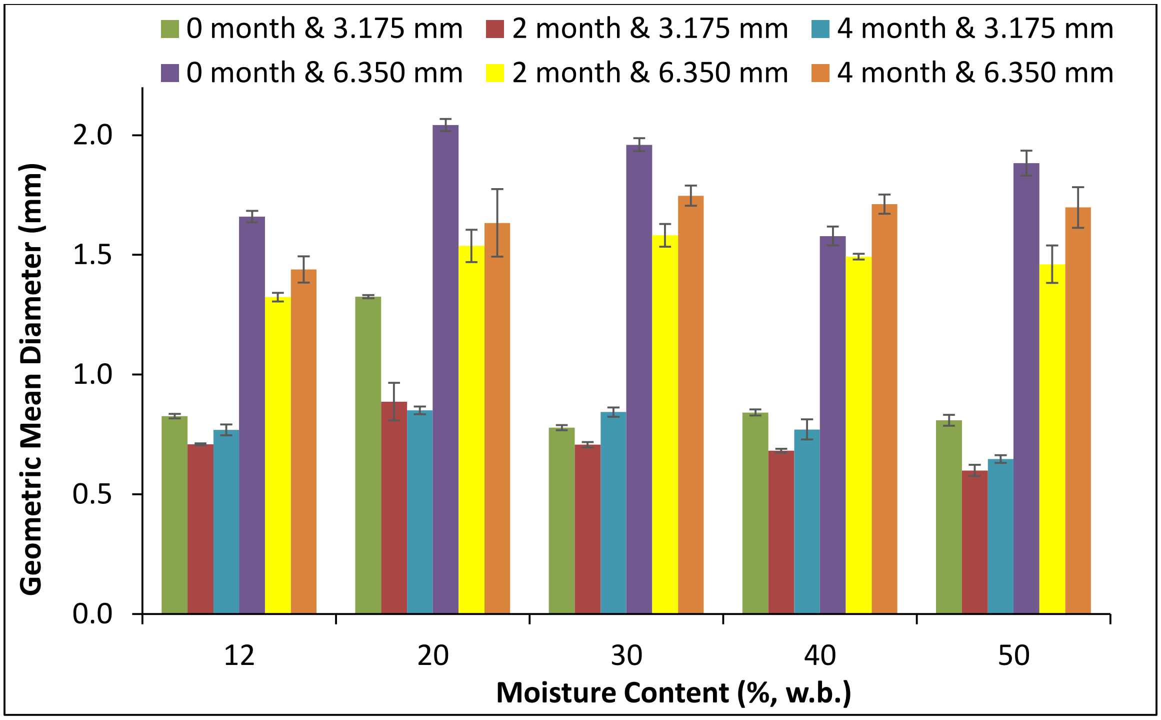

Figure 3.18. Effect of moisture content, storage time, and screen size on geometric mean diameter of oven-dried samples.

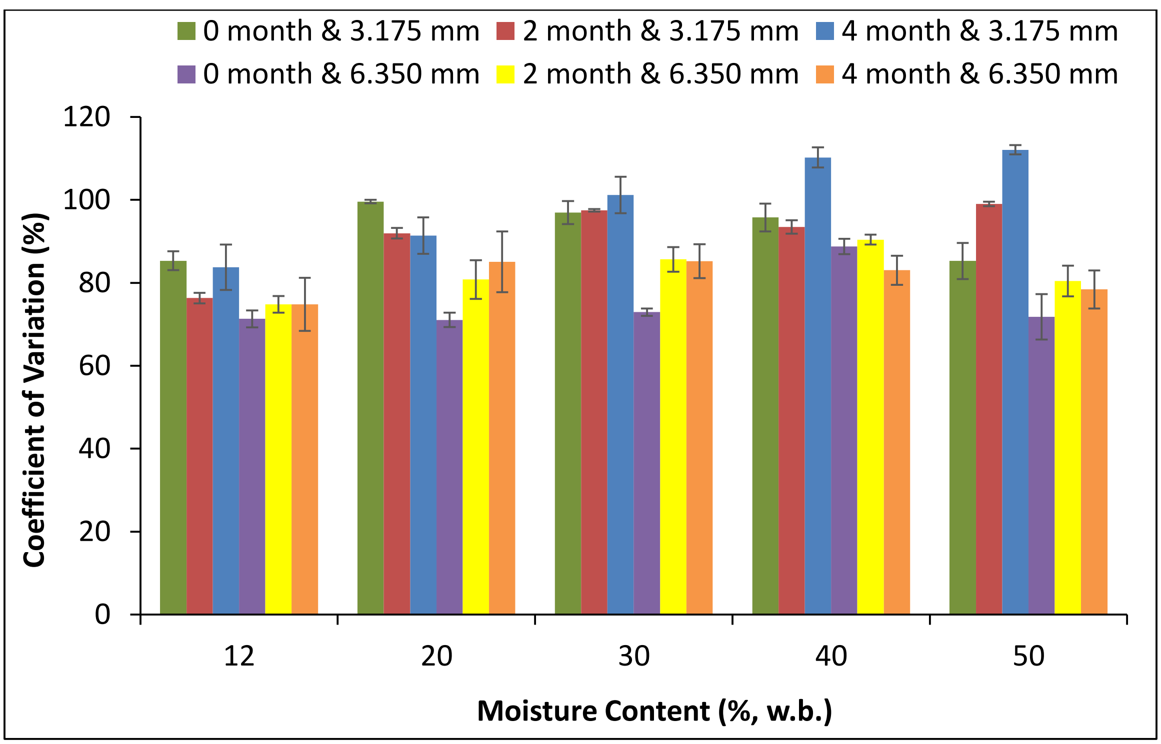

Figure 3.19. Effect of moisture content, storage time, and screen size on coefficient of variation of oven-dried samples.

Table 3.5. Effect of moisture content, storage time, and screen size on particle size distribution of oven-dried grinds.

Effects	Geometric Mean Diameter* (mm)	Geometric Standard Deviation* (mm)	Coefficient of Variation* (%)
Storage Time** (month)			
0	$1.371^{a} \pm 0.018$	$1.109^{a} \pm 0.015$	$83.878^{b} \pm 1.091$
2	$1.098^{c} \pm 0.018$	$0.939^{c} \pm 0.015$	$87.042^{a,b} \pm 1.091$
4	$1.211^{b} \pm 0.018$	$1.055^{b} \pm 0.015$	$90.526^{a} \pm 1.091$
Moisture Content*** (%, w.b.)			
12	$1.121^{y} \pm 0.023$	$0.856^{y} \pm 0.020$	$77.730^{x} \pm 1.409$
20	$1.379^{v} \pm 0.023$	$1.164^{v} \pm 0.020$	$86.649^{w} \pm 1.409$
30	$1.270^{w} \pm 0.023$	$1.095^{v,w} \pm 0.020$	$89.902^{v,w} \pm 1.409$
40	$1.180^{w,y} \pm 0.023$	$1.077^{w} \pm 0.020$	$93.613^{v} \pm 1.409$
50	$1.183^{w,y} \pm 0.023$	$0.977^{x} \pm 0.020$	$87.849^{w} \pm 1.409$
Screen Size**** (mm)			
6.35	$1.650^{r} \pm 0.015$	$1.308^{r} \pm 0.013$	$79.636^{s} \pm 0.891$
3.175	$0.803^{s} \pm 0.015$	$0.760^{s} \pm 0.013$	$94.661^{r} \pm 0.891$

*Value in each cell = estimate ± standard error.
**Storage time effect: values in each column with same letter (a-c) are not significantly different (p<0.05).
***Moisture effect: values in each column with same letter (v-z) are not significantly different (p<0.05).
****Screen size effect: values in each column with same letter (r-s) are not significantly different (p<0.05).

3.4.2.4 Specific Grinding Energy Requirement and Grinding Difficulty

Figure 3.20 shows the plot of the specific energy consumed during the grinding of loblolly pine woodchips as affected by storage time, moisture content of woodchips, and hammer mill screen size. Less energy was consumed as screen size increased (i.e. as size reduction ratio decreased). The specific grinding energy ranged from 187.79 kJ/kg d.b. to 789.46 kJ/kg d.b. for woodchips ground through 3.175 mm screen size and from 59.91 kJ/kg d.b. to 192.13 kJ/kg d.b. for woodchips ground through 6.350 mm screen size. These values are comparable to those reported by Hehar (2013) for grinding energy requirement of loblolly pine woodchips which ranged from 97.97 kJ/kg d.b. (14.70% moisture content and 3.175 mm hammer mill screen size) to 309.82 kJ/kg d.b. (23.60% moisture content and 3.175 mm hammer mill screen size). Naimi et al. (2012) reported that the specific grinding energy requirement for wood species (at about 10% moisture content and through a 2 mm knife mill screen size) varied from 143.15 kJ/kg d.b. (aspen) to 323.15 kJ/kg d.b. (hybrid poplar). According to Miao et al. (2011) the energy required to grind willow woodchips (at 10% moisture content) through a 1 mm, 2 mm, and 4 mm knife mill screen size was about 2275 kJ/kg d.b., 1400 kJ/kg d.b., and 525 kJ/kg d.b., respectively.

As discussed in the previous section on the particle size distribution of grinds, the particle size and particle size distribution of grinds produced through the same screen are influenced by moisture content and storage time (Figure 3.18). The implication of this is that size reduction ratio is a function of woodchips properties. Therefore, "characteristic grindability constant" was employed in the assessment and analysis of specific grinding energy requirement in relation to the particle size distribution of the grinds produced. The "characteristic grindability constant" corresponds to the model constants of the three main grinding models (Equations 3.1 – 3.3) and is referred to as grinding difficulty henceforth.

These grindability constants (i.e. grinding difficulty) were expressed as a function of moisture content, storage time, and screen size (Equation 3.18). Table 3.6 shows that storage time did not have significant effect ($p < 0.05$) on the specific grinding energy requirement of loblolly pine woodchips. Hence, grinding difficulty was independent of storage time. This suggests that despite the changes that were observed in the various properties of the biomass during the storage, the energy consumed during grinding remain significantly ($p < 0.05$) unchanged over the storage periods considered.

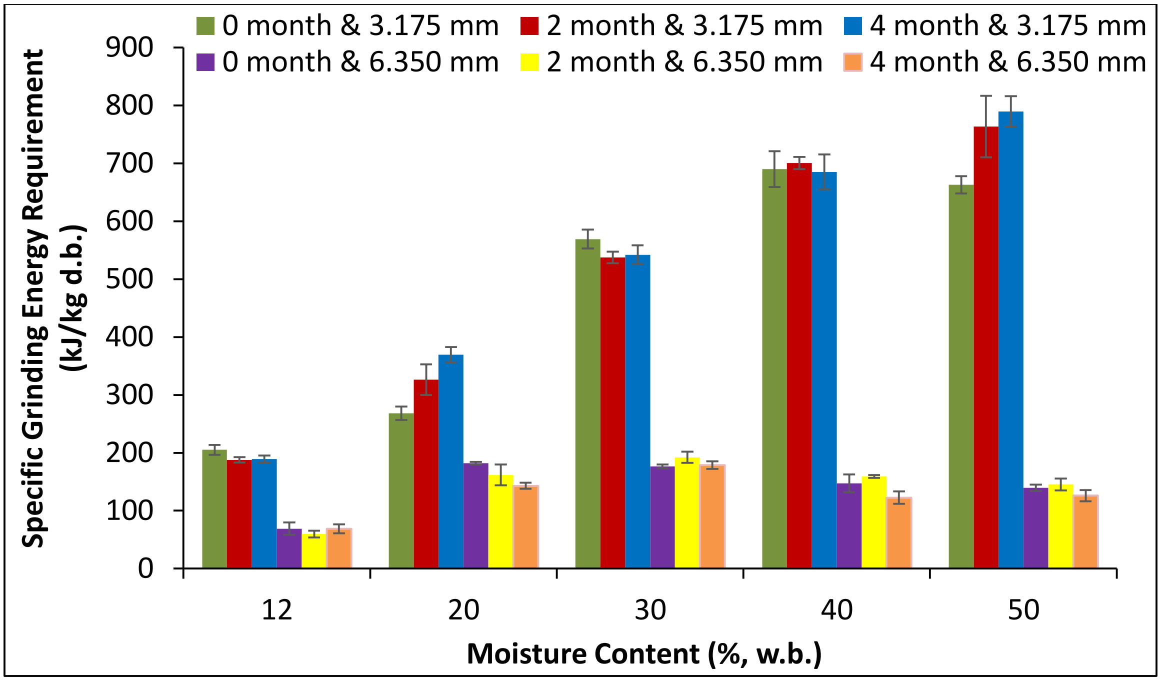

Figure 3.20. Effect of moisture content, storage time, and screen size on specific grinding energy consumption.

$$K_K; K_R; K_b = \propto + (\beta * MC) + (\lambda * MC * SS) + (\rho * MC^2) + (\eta * SS) \quad (3.18)$$

where

K_K, K_R, K_b = Kick's, Rittinger's, and Bond's constant, respectively (characteristic grindability constant)

MC = moisture content (%, w.b.)

SS = screen size (mm)

$\propto$, β, λ, ρ, η = non-linear regression coefficients

Table 3.6. ANOVA result of moisture, storage time, and screen size effect on specific grinding energy of loblolly pine woodchips

Effect	Num DF[a]	Den DF[b]	F Value	Pr > F
Storage Time	2	6	1.61	0.2756
Moisture Content (MC)	4	72	363.31	<.0001
Screen Size (SS)	1	72	3494.15	<.0001
MC*SS	4	72	252.44	<.0001

[a]Num DF means numerator degree of freedom
[b]Den DF means denominator degree of freedom

Also, there was an expected decrease in grinding difficulty with increase in screen size (Figures 3.21 – 3.23). This response was similarly obtained by Mani et al. (2004). The authors reported that the specific grinding energy consumption of hammer mill reduced from 185.58 kJ/kg to 40.90 kJ/kg for wheat straw, 190.80 kJ/kg to 49.64 kJ/kg for barley straw, 79.45 kJ/kg to 25.06 kJ/kg for corn stover, and 225.180 kJ/kg to 85.82 kJ/kg for switchgrass when screen size was increased from 0.8 mm to 3.2 mm. Ghorbani et al. (2010) also reported reduction in specific grinding energy consumption as hammer mill screen size increased when grinding alfalfa chops. This observation can be explained by the amount of work accomplished by the grinder because at lower screen size, size reduction ratio and work done are higher, and more power is dissipated.

When samples were ground through 3.175 mm screen size, grinding difficulty increased with increase in moisture content, with the rate of increase decreasing as moisture content increased. Grinding difficulty for 6.350 mm screen size was highest at 30%, and further increase in moisture resulted in reduction in grinding difficulty (Figures 3.21 – 3.23). Literature on the influence of moisture and other liquids on the mechanical properties and grinding of biological material suggest that these relationships is due to the contrasting plasticizing and lubricating effect of moisture (Lewicki, 2004; Brandt et al., 2012). Increase in moisture causes biomass to become tougher and therefore requires more energy to permanently deform them. However, the moisture in woody biomass at moisture contents above fiber saturation point (usually about 25% moisture content, wet basis—Zelinka et al., 2012) is in the form of free water which acts as a lubricant during the grinding process. This lubricating effect was not apparent for the 3.175 mm screen size. However, the slope of the grinding difficulty–moisture content curve was reduced for woodchips with moisture content greater than 30%.

Model adequacy metrics (Pearson's coefficient of determination, mean relative deviation, and root mean square of error) were used to evaluate the suitability of the three grinding equations (Equations 3.1 – 3.3) when coupled with Equation 3.18 to predict the specific grinding energy for loblolly pine woodchips (Table 3.7). Mean relative deviation was calculated as shown in Equation 3.19 (Littlefield et al., 2011). It was observed that Bond's equation had the best prediction for loblolly pine at the conditions tested in this study with R^2 of 0.9839, MRD of 0.1076, and RMSE of 30.4353. However, Kick model had the least MRD. When the predicted specific grinding energy was fitted against the measured specific grinding energy, all the models gave a slope very close to one (Figure 3.24)—an indication of the closeness of predicted values to the measured values.

$$MRD = \frac{1}{n}\sum_{i=1}^{n}\left(\left|\frac{E-\hat{E}}{E}\right|\right) \tag{3.19}$$

where

MRD = mean relative deviation

n = number of observations

E = measured specific grinding energy (kJ/kg d.b.)

Ê = predicted specific grinding energy (kJ/kg d.b.)

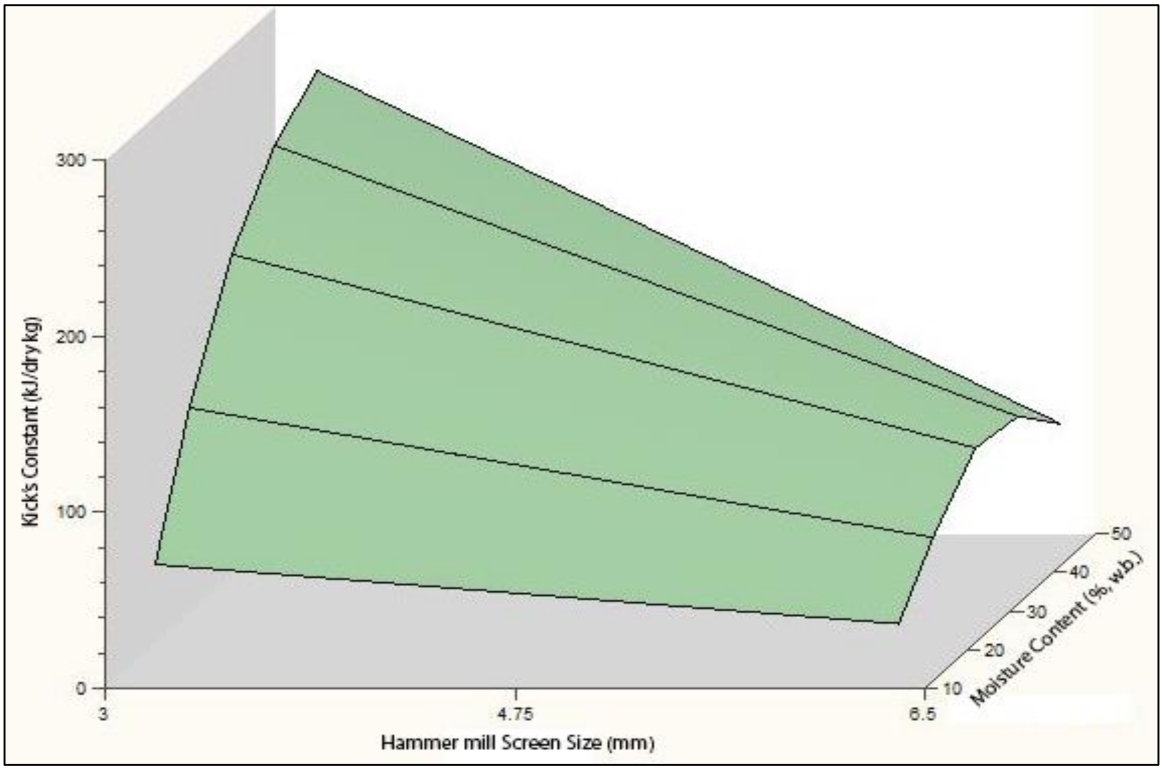

Figure 3.21. Effect of moisture content and screen size on Kick's constant

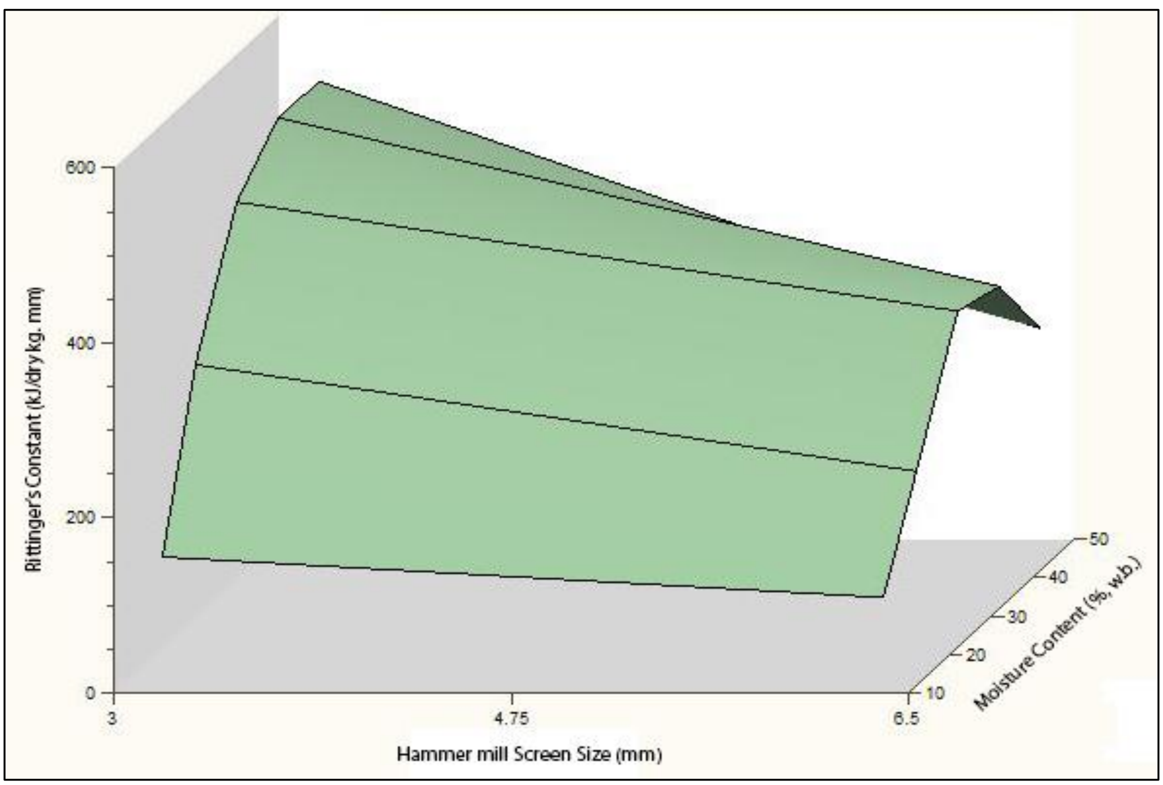

Figure 3.22. Effect of moisture content and screen size on Rittinger's constant

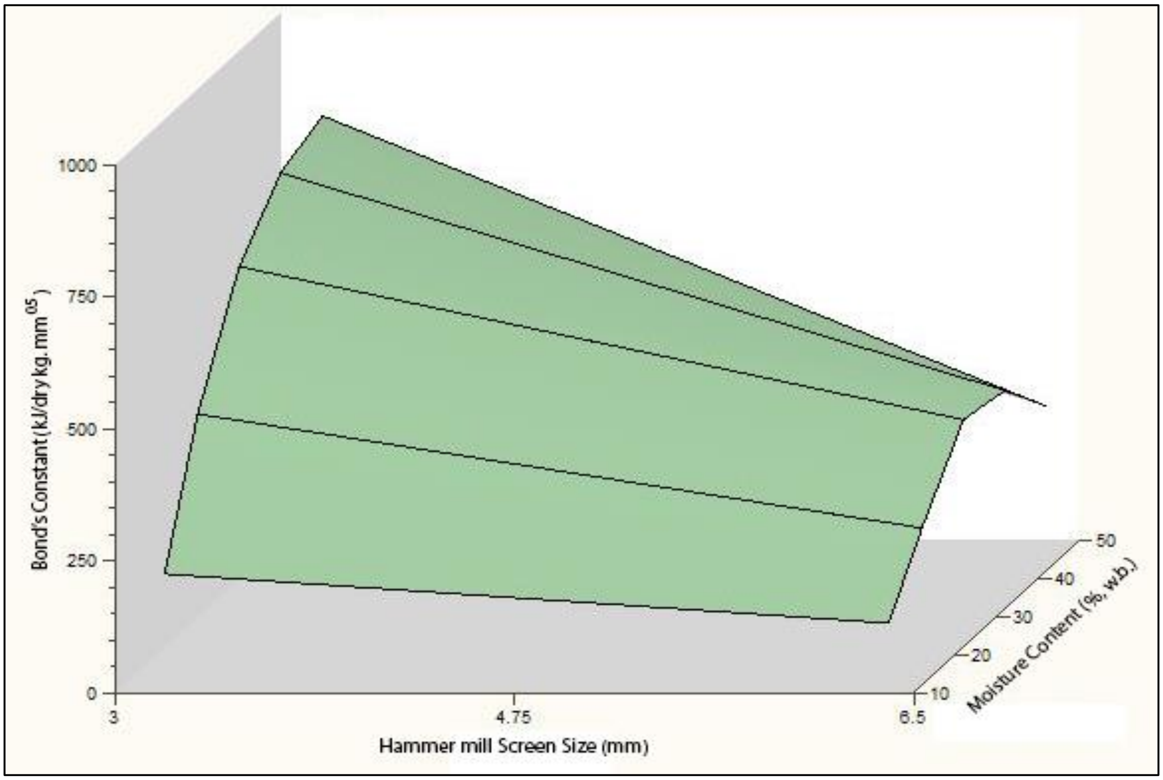

Figure 3.23. Effect of moisture content and screen size on Bond's constant

Table 3.7. Estimates of parameters of grinding models and statistical parameters associated with the equations.

Model	Parameter Estimates					Statistics of fitting		
	α	β	λ	ρ	*η*	r^2	RMSE	MRD
Kick	330.1 ± 5.240	3.059 ± 0.110	-1.546 ± 0.069	-0.099 ± 0.009	-50.18 ± 0.935	0.9826	31.6174	0.1013
Rittinger	285.8 ± 4.706	6.561 ± 0.346	-3.002 ± 0.218	-0.294 ± 0.022	-140.40 ± 2.686	0.9830	31.2410	0.1159
Bond	309.7 ± 4.852	9.639 ± 0.392	-4.700 ± 0.247	-0.359 ± 0.030	-178.90 ± 3.252	*0.9839*	30.4353	0.1076

∝, β, λ, ρ, and η are described in Equation 3.19

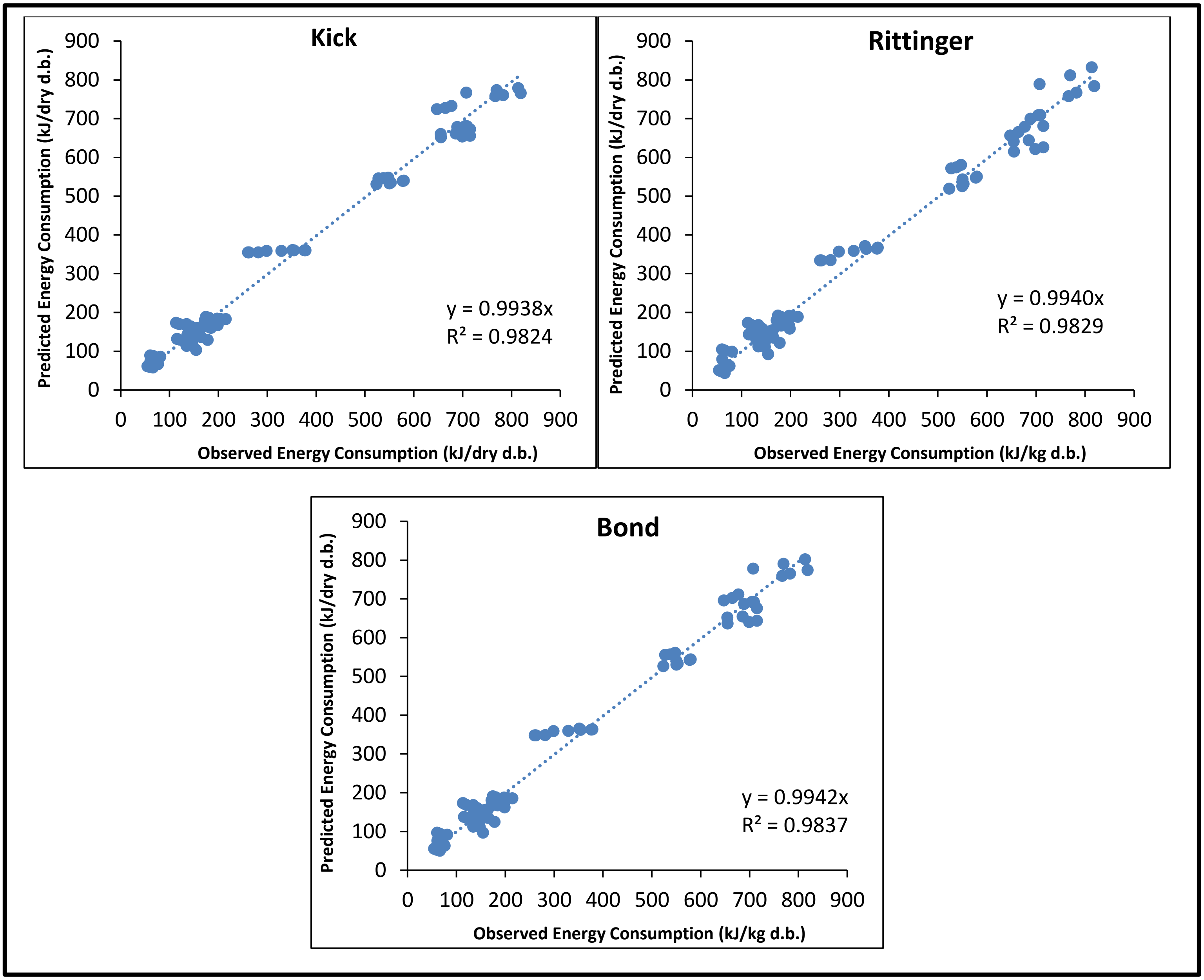

Figure 3.24. Comparison of observed and predicted specific grinding energy consumption from Kick, Rittinger, and Bond equation.

3.5 Conclusions

It was established that as storage time of loblolly pine woodchips increased, volatile matter and energy content reduced while ash content increased. Moisture loss during grinding positively correlated with the moisture content of woodchips and negatively correlated with hammer mill screen size. Grinding of woodchips above 30% moisture content produced fluffy particles. The oven-dried bulk density of grinds decreased with increase in the moisture content of woodchips and hammer mill screen size. Geometric mean diameter of grinds was significantly affected ($p < 0.05$) by moisture content and hammer mill screen size. Grinds produced from fresh woodchips had higher geometric mean diameter than grinds produced from stored woodchips. Furthermore, the specific grinding energy requirement of loblolly pine was not significantly affected ($p < 0.05$) by storage time but decreased with increase in hammer mill screen size. In addition, average specific grinding energy initially increased from 193 kJ/kg d.b. to 549 kJ/kg d.b. (for screen size 3.175 mm) and from 65 kJ/kg d.b. to 182 kJ/kg d.b. (for screen size 6.350 mm) as moisture content increased from 12% to 30%, then increased to 738 kJ/kg d.b. and reduced to 137 kJ/kg d.b. with further increase to 50% moisture content for screen sizes 3.175 mm and 6.350 mm, respectively. The increase in required grinding energy with increasing moisture content was attributed to the plasticizing effect of moisture while the decrease in required grinding energy with increasing moisture was attributed to the lubricating effect of moisture. Out of the three equations (Kick’s, Rittinger’s, and Bond’s equations), Bond’s equation best fit the specific grinding energy data for loblolly pine woodchips.

Chapter 4: Influence of Moisture Content, Tree Height, and Tree Radius on Toughness and Stress of Loblolly Pine

4.1 Abstract

Bending force-displacement test is a measure of materials ductility and can be used to understand energy required to grind biological materials, such as woody biomass. A three point bending test was used to assess the effects of moisture content and location within a tree on specific toughness and stress of loblolly pine. Test samples were collected at 20%, 40%, 60%, and 80% radial distance from the center of the tree at four heights (0 feet/tree base, 4 feet/breast height, 12 feet, and 18 feet) of five randomly selected loblolly pine tree stems. The wood samples were cut into cuboid shapes (40 mm by 10 mm by 0.5 mm). The samples from the radius and height treatment combinations were adjusted to three moisture levels (10%, 20%, and 30% wet basis). Results show that average toughness varied between 14.41 kJ/m^2 and 100.75 kJ/m^2 while average bending stress varied between 35.14 MPa and 117.65 MPa. Bending stress of loblolly pine decreased with increase in moisture content, height, and radial distance from outside to inside. Similar relationships were obtained between toughness and height, and toughness and radial distance. Toughness however increased with moisture content. These observations confirm that wood materials get tougher and stronger as they mature. Regression analysis showed that moisture content was the most important factor in altering toughness and bending stress. The r^2 for the polynomial model developed to predict toughness and bending stress from moisture content, tree height, and tree radius were 0.614 and 0.662, respectively.

4.2 Introduction

The values of the mechanical properties of biomass can be used in the design of grinding equipment since during grinding, biomass is subjected to one or a combination of mechanical forces (impact force is predominately utilized in hammer mills, shear force in knife mill, and compressive force in roller mill – Fellows, 2000). Mechanical properties also influence the energy and ease to fracture and grind biological materials (Brennan, 2006). Brittle materials are easy to fracture and grind while ductile materials are not. The mechanical behavior of biological material is useful in explaining how these materials response to mechanical forces and stresses during grinding operation.

Properties such as elasticity, toughness, ductility, stiffness, strength, and compressibility are used to characterize the mechanical behavior of biological materials (Bright and Kleis, 1964; Broughton and Brady, 1984). These properties are often obtained from force-displacement data of tests such as tensile test, compressive test, bending test, shearing test, and hardness test. The choice and applicability of these tests is primarily influenced by the process the material will be subjected to. For example, in studies involving cutting of plant stems during harvest operation, shearing test is most appropriate (Womac et al., 2005), while compressive test is relevant in food sensory (Peleg, 2003). Although the study of the behavior of materials when subjected to force appears as a protracted process, data obtained from the force-displacement curve usually allow for the simultaneous measurement of modulus, strength, and breakage energy (Broughton and Brady, 1984).

Mechanical properties of materials have been documented to be a function of temperature, loading conditions, and material characteristics (Stroshine, 1998). Therefore, it is important that the load conditions (loading rate and loading duration) are reported together with the experimental values for mechanical properties. Some of the important material characteristics include moisture content, biomass type, material size, maturity, cellular structure, and composition (O'Dogherty et al., 1995; Bright and Kleis, 1964). The mechanical properties of biological materials have been documented in several studies. According to Dahlen et al. (2012), the mean bending stress (modulus of rupture) of Southern pine and Douglas fir at 15% moisture content was 48.26 MPa and 42.10 MPa, respectively.

Ozbek et al. (2009) reported a negative second order polynomial relationship between bending stress and moisture content of safflower stalk (bending stress increased from 13.81 MPa to 54.02 MPa as moisture content was decreased from 64.48% to 11.31%) but an opposite relationship was observed between shearing stress and moisture content (shearing was varied from 0.68 MPa for 11.13% moisture content to 1.37 MPa for 68.48% moisture content). Ince et al. (2005) also reported a decrease in the shearing stress for sunflower stalk from 62.09 MPa to 34.77 MPa when moisture content reduced from 75% to 15%. In their work on wheat straw, Esehaghbeygi et al. (2009), highlighted the significance of moisture content and stem height to bending stress. As moisture content increased from 15% to 45%, bending stress decreased from 26.77 MPa to 17.74 MPa, and as stem height increased from 100 mm to 300 mm, bending stress decreased from 21.14 MPa to 17.85 MPa. Similar results were reached by Shahbazi and Galedar (2012) for safflower stalk. Additionally, Taghijarah et al. (2011) established that shearing rate positively correlates with shearing stress while working on shearing characteristics of sugar cane.

Hernandez et al. (2005), in their study on the utilization of ponderosa pine, explained that the mechanical strength of trees increases as radial distance increases (from tree pith to tree bark) because trees initially produce juvenile wood as they grow from age 0 to 5 years before producing mature wood. The higher microfibril angle, shorter cell length, and lower specific gravity of juvenile woods compared with mature woods result in their lower mechanical strength. This explanation is supported by the observations of Bao et al. (2001) in a study on 10 woody biomass species (Table 4.1). Furthermore, juvenile wood proportion increases with tree height (from tree base to tree top). Kiaei (2011) studied the effect of height on mechanical properties of elm wood and reported that the mean bending stress decreased from 138.89 MPa (at tree base) to 123.00 MPa (at tree top).

Table 4.1. Effect of juvenile and mature wood on bending, tensile and shearing strength of 10 wood species (Bao et al., 2001).

Wood Type	Juvenile Wood Or Mature Wood	Bending Stress (MPa)	Tensile Stress (MPa)	Shearing Stress (MPa)
Common China-fir	Juvenile	54.9	73.1	5.5
	Mature	65.1	77.1	7.1
Korean larch	Juvenile	90.3	108.3	10.1
	Mature	99.5	108	10.5
Masson pine	Juvenile	82.7	103.1	7.6
	Mature	87.9	106.9	10.2
Yunnan pine	Juvenile	85.1	148.5	9.8
	Mature	121.4	167.3	11.8
Japanese larch	Juvenile	90.2	98.8	10.4
	Mature	134.5	185.3	13.5
Slash pine	Juvenile	69.3	79.1	9.9
	Mature	86.3	115.5	11.2
Loblolly pine	Juvenile	67.5	87.3	8.9
	Mature	98.9	107.1	12.3
Lemon eucalyptus	Juvenile	146	154.2	16.8
	Mature	161.5	139.2	17.7
Lankao paulownia	Juvenile	30.8	36.5	4.5
	Mature	29.7	27.2	4.8
Sanbei poplar	Juvenile	58	89.5	6.5
	Mature	63.2	84.4	7

In chapter 3, results showed that moisture content significantly influence the energy required to grind loblolly pine woodchips and the quality (particle size distribution and bulk density) of the resulting grinds. The study in this chapter was conducted to quantify the influence of moisture content and location within tree (tree height and tree radius) on the strength and toughness of loblolly pine wood using three point bending test.

4.3 Materials and Methods

4.3.1 Sample Preparation

Loblolly pine disks were obtained from Auburn University, AL. Five tree stems (approximately 12 years old) were harvested, and disks (Figure 4.1b) were cut as highlighted in Figure 4.1a from each tree stem (at 0 feet—base, 4.6 feet—breast height, 12 feet, and 18 feet height). Disks were labelled and dried to 10% moisture content (wet basis) using a humidity chamber (model ESL-2CA, Espec North America, Inc., MI). Cuboidal wood samples (40 mm by 10 mm by 5 mm) (Figure 4.1c) were thereafter cut from the disks at 20%, 40%, 60%, and 80% radial distance between the tree pith and tree bark using a power saw (Figure 4.1b).

4.3.2 Moisture Adjustment and Moisture Content Measurement

The moisture content of wood samples was adjusted to 10%, 20%, and 30% by adding a predetermined quantity of water. The sample was kept in an airtight plastic bag, then placed in a refrigerator at 4±1°C for about 10 days to ensure equilibration of moisture (Al-Mahasneh and Rababah 2007; Balasubramanian and Viswanathan, 2010). In order to validate the moisture adjustment process, moisture content of samples was determined according to ASABE Standard S358.3 (ASABE Standards, 2012) by placing 20 g of the sample in an oven at 103±2°C for 24 hours. All deviations from the expected moisture content were within 2% of the targeted moisture content.

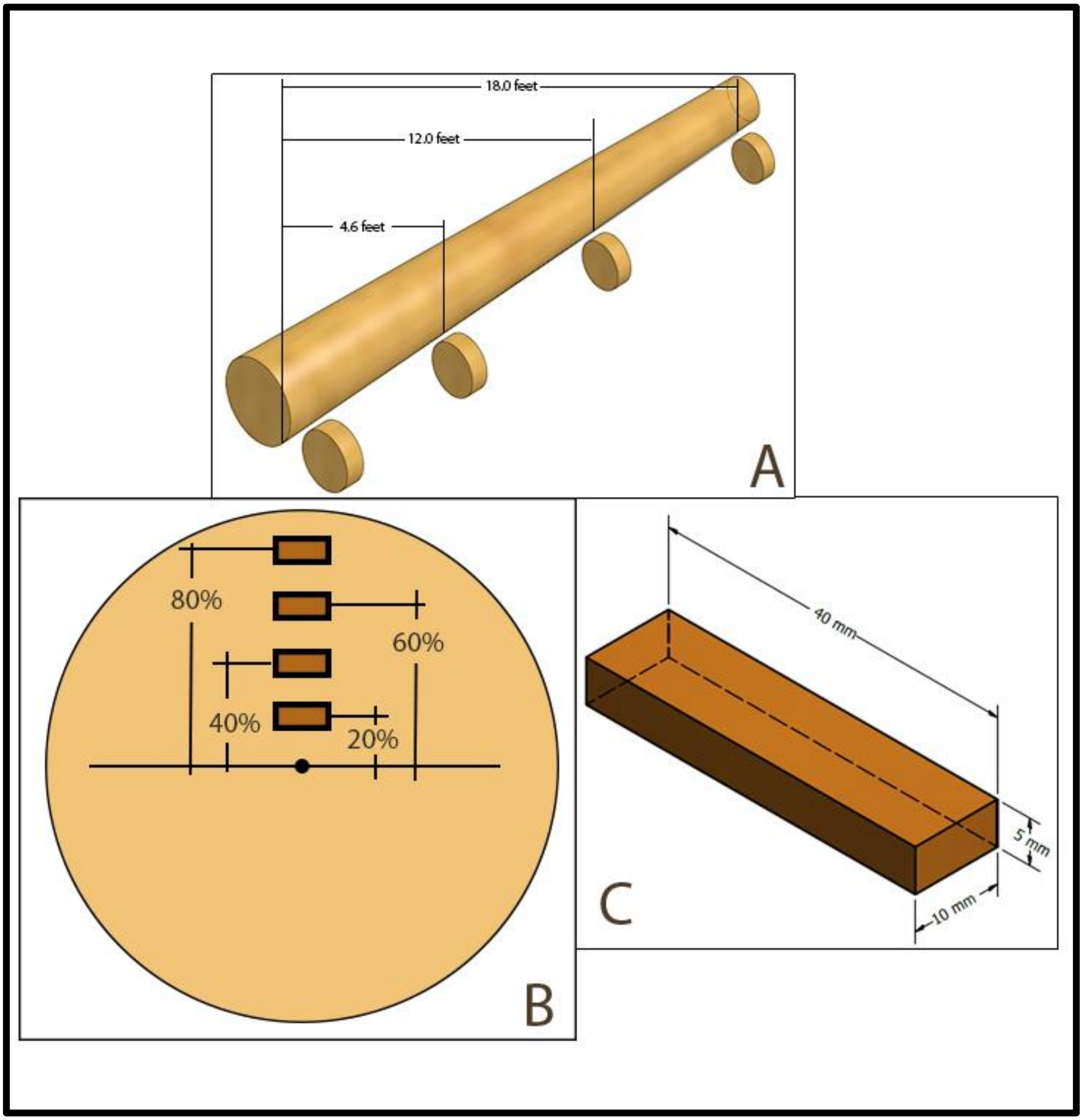

Figure 4.1. Sample preparation.

4.3.3 Bending Test

A Texture analyzer (model TA-HDi, Stable Micro Systems, Texture Technologies Corp., NY) (Figure 4.2) was used to perform three point bending test. The experimental setup was conducted similar to Ozbek et al. (2009) study on safflower stalk. Two 3.2 mm diameter supports were placed 19.5 mm apart, and a 3.2 mm diameter indenter was applied at the center of the sample (Figure 4.3). Loading speed was set at 0.8 mm/s. Force-displacement curve and data were obtained through the software of the texture analyzer. The maximum height and area under the curve were determined and recorded (Figure 4.4). Bending stress was computed according to Equation 4.2 (Jiang et al., 2007) while the specific toughness was calculated as the ratio of the area under the curve to the initial cross-sectional area of the wood sample (Ozbek et al., 2009).

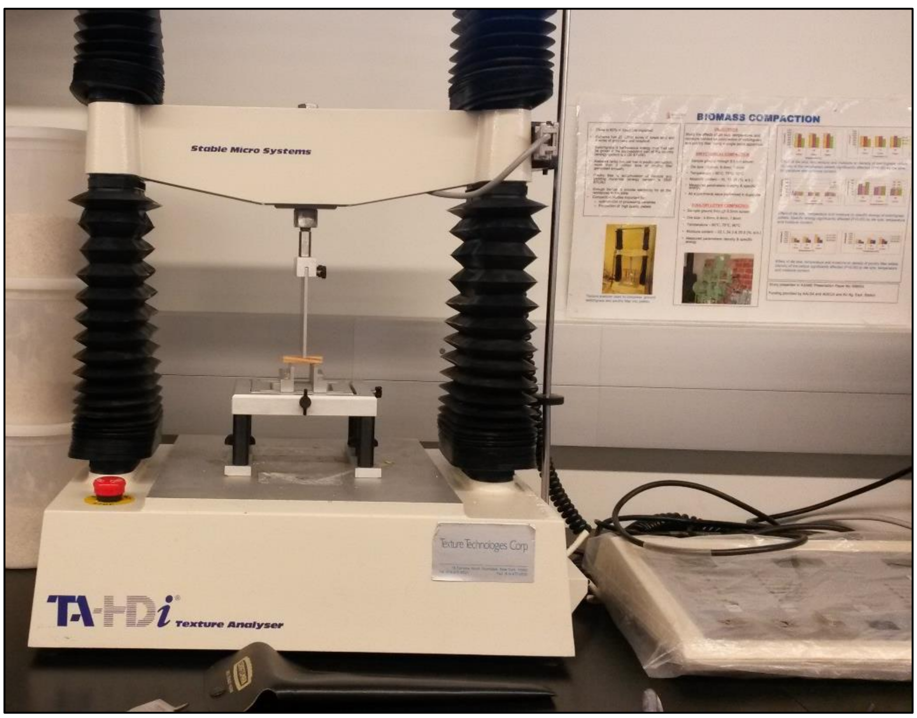

Figure 4.2. Texture analyzer used for three point bending test.

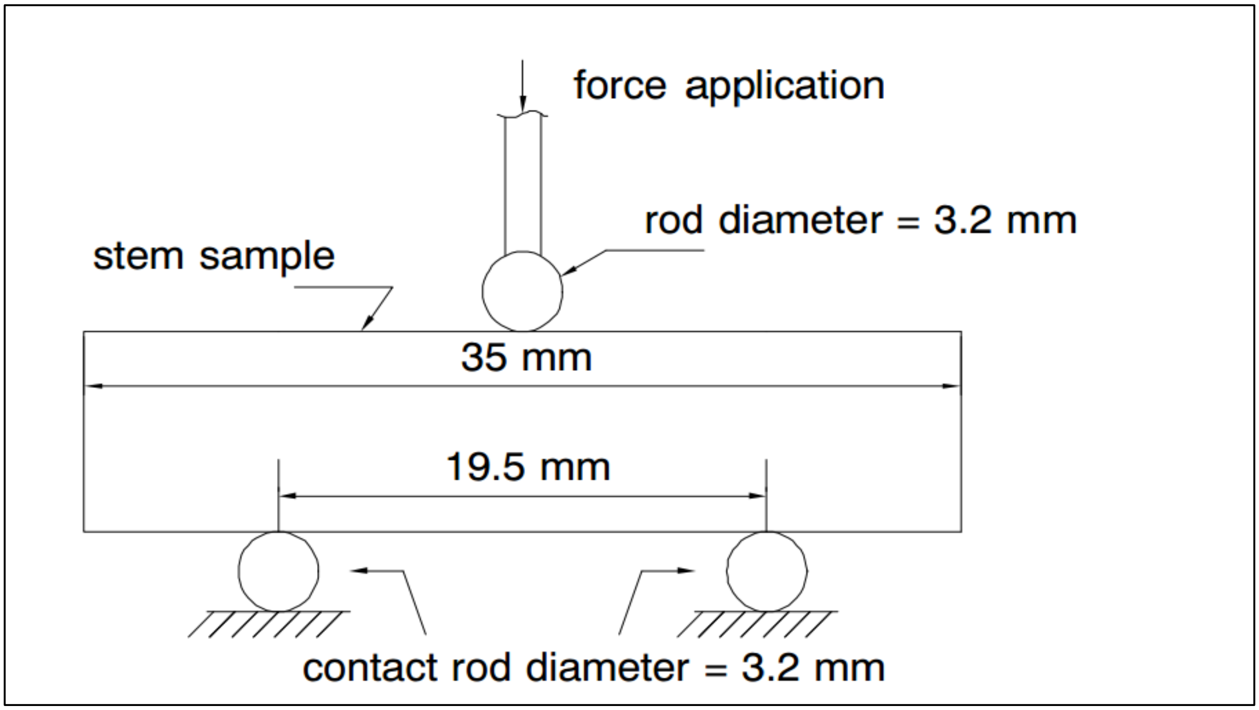

Figure 4.3. Bending test experiment setup (Ozbek et al., 2009).

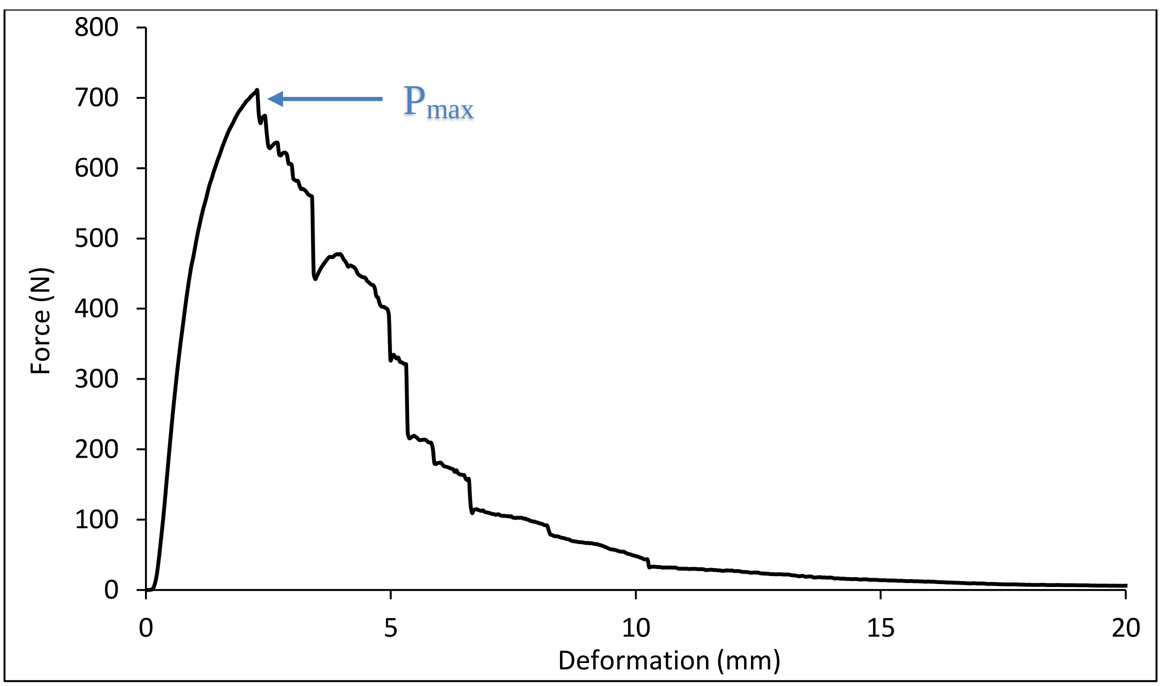

Figure 4.4. Typical force-displacement curve.

$$\tau = \frac{3P_{max}L}{2bh^2} \tag{4.2}$$

where

τ = bending stress (MPa)

P_{max} = maximum bending force (N)

L = distance between supports, Span (m)

b = sample width (m)

h = sample thickness (m)

4.3.4 Experiment Design and Data Analysis

Randomized complete block design (consisting of five blocks of tree stems) was followed in this study. Data for each treatment combination were collected in triplicate. Statistical analysis of experimental data was conducted with SAS software (SAS 9.3, SAS Institute Inc., Cary, N.C.) using PROC REG, PROC UNIVARIATE and PROC BOXCOX. PROC REG was used to develop regression models. A 5% significance level was chosen for all significance testing carried out, and all possible main effects and two way interactions were considered. Variables selection was carried out according to the stepwise elimination procedure using the SELECTION=STEPWISE option in PROC REG.

4.4 Result and Discussions

4.4.1 Force-Displacement Curve

The force-displacement curve provides a qualitative as well as quantitative assessment of the response of the material to stress. The quantitative properties are discussed in the specific toughness and bending stress sections (sections 4.4.2 and 4.4.3). The three moisture levels considered in this study produced distinct force-displacement curves (Figure 4.5). It was observed that the tendency of materials to break abruptly—brittleness reduced as moisture content increased. In other words, the higher the moisture content, the higher the ductility of loblolly pine wood. This evaluation was achieved by observing the spikiness of the curves which illustrates the suddenness of wood rupture and brittleness (Nussinovitch et al., 2001). As the moisture content of sample increases, the spikiness of the curves disappeared.

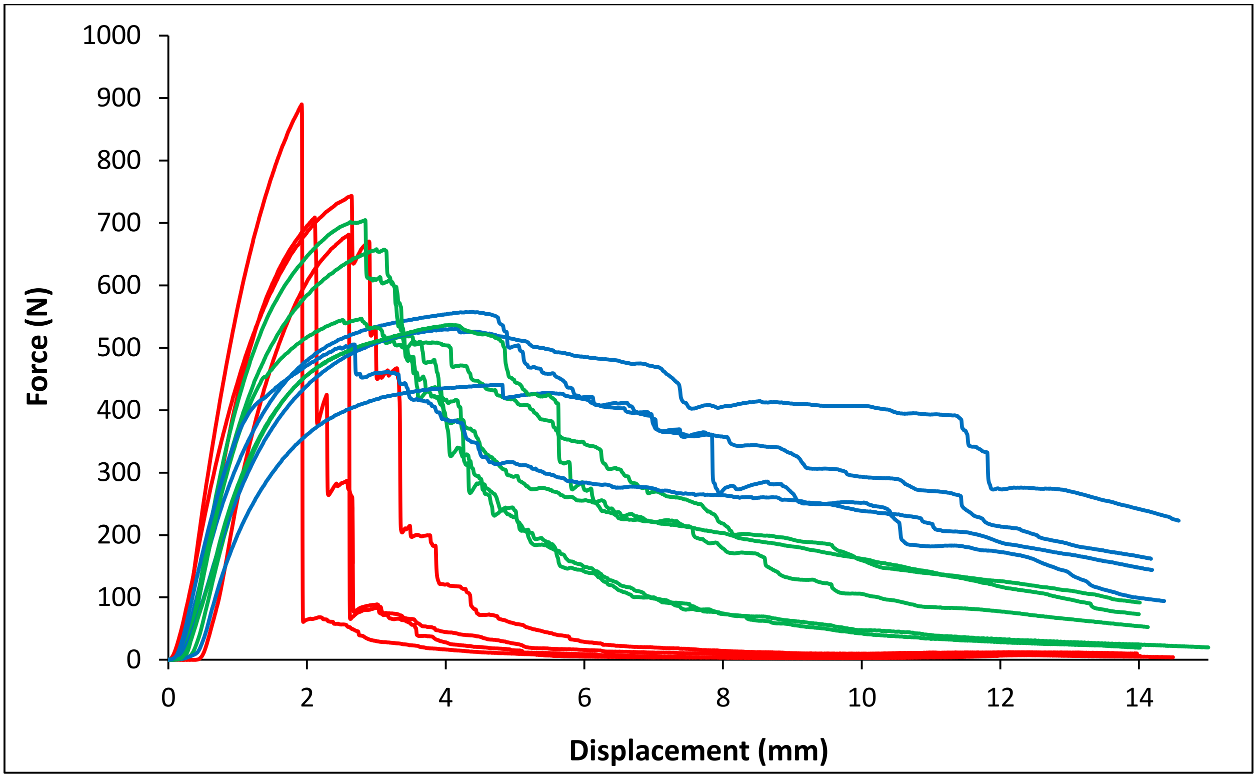

Figure 4.5. Typical effect of moisture content on force-displacement curves for three point bending test of loblolly pine sample (Loading rate = 0.8 mm/s; Span = 19.5 mm; ——10%; —— 20%; —— 30% moisture content).

4.4.2 Specific Toughness

The specific toughness of biomass is the energy consumed in the rupturing of biomass, and it relates to the grinding energy requirement of biomass. The average specific toughness of loblolly pine wood in this study ranged from 14.41 kJ/m^2 to 100.75 kJ/m^2 (Table 4.2). In comparison, Bao et al. (2001) reported 27.6 kJ/m^2 and 30.1 kJ/m^2 for juvenile and mature loblolly pine, respectively. Although there was an overlap in the range of these values and the values reported in this study, the highest value in this study is about 335% higher than that reported by Bao et al. (2001). This may be due to the moisture content (15%), age (26 years), and dimension (2 m long) of the sample used in the study reported by Bao et al. (2001).

Specific toughness of loblolly pine wood was evaluated as a function of moisture content, tree height, and tree radius. The specific toughness of loblolly pine increased as moisture content increased (Table 4.2). This result is explained by Lewicki (2004) as a result of the plasticizing effect of moisture on the structure of biomass. Through hydrophilic and hydrophobic interactions, water alters the structure of biomass and promotes polymer chain mobility. Consequently, water causes plasticization which results in lowered hardness, loss of brittleness, and increased toughness. The positive correlation between specific toughness and moisture content supports the observation in chapter 3, where specific grinding energy consumption of loblolly pine woodchips increases with moisture content.

Table 4.2 shows that as tree height increased, the specific toughness was reduced. This observation implies that the grinding energy requirement of loblolly pine wood will decrease with increasing tree height, since specific toughness is expected to positively correlate with the energy requirements during hammer milling (Fellows, 2000). Generally, wood materials closer to the tree bark were tougher than those closer to the pith (particularly for tree height of 12 feet, and 18 feet). This result was attributed to maturity, as mature wood is located closer to the bark of trees while juvenile wood is located closer to the tree pith (Forest Products Laboratory, 1998; Kretschmann, 2010). At the tree base and breast height (0 feet and 4.6 feet, respectively), it was observed that specific toughness initially increased with increasing tree radius, and then reduced slightly when tree radius increased to 80%.

Table 4.2. Effect of moisture on specific toughness for different tree height and radius combinations

Tree Height (feet)	Tree Radius (%)	Specific Toughness (kJ/m^2) at Moisture Content Level* (%, w.b.)								
		10			20			30		
		Mean	SD	CV	Mean	SD	CV	Mean	SD	CV
0.0	20	30.18	11.37	37.68	71.97	21.00	29.18	83.64	27.67	33.08
	40	51.68	18.60	5.99)	100.70	20.30	20.16	87.78	32.51	37.03
	60	46.52	12.33	26.49	99.60	5.39	25.49	96.87	30.00	30.97
	80	39.50	13.24	33.52	88.64	27.51	31.04	91.79	36.59	39.87
4.6	20	27.37	12.18	44.49	58.53	11.05	18.88	61.71	19.16	31.04
	40	33.16	13.48	40.66	76.51	18.86	24.65	87.11	18.00	20.67
	60	30.10	10.52	34.96	81.86	19.22	23.49	83.87	22.75	27.13
	80	32.08	9.35	29.16	69.15	16.97	24.55	80.63	20.80	25.80
12.0	20	14.41	3.35	23.22	44.25	12.15	27.46	46.73	12.00	25.67
	40	19.30	3.95	20.47	41.20	13.65	33.13	50.19	14.72	29.32
	60	25.14	6.84	27.21	46.48	18.06	38.85	51.08	18.13	35.50
	80	26.57	4.83	18.17	55.33	15.32	27.68	55.83	13.65	24.44
18.0	20	16.02	4.76	29.72	31.38	12.22	38.94	43.88	11.77	26.82
	40	21.98	3.80	17.27	40.92	8.63	38.94	38.42	18.46	48.04
	60	25.96	5.34	20.56	58.64	20.49	34.94	50.02	26.05	52.08
	80	23.09	9.80	42.43	50.39	16.74	33.23	66.03	14.88	22.54

* SD is standard deviation, and CV is coefficient of variation.

4.4.3 Bending Stress

Bending stress of any material describes the magnitude of stress that can be absorbed by the material before rupture. It is significant in determining the stress required to be provided by the beaters of hammer mills. Breakage into smaller pieces will occur when the stress impacted on a material exceeds the bending stress of the material (Earle and Earle, 1983). It was observed that bending stress decreased with increase in moisture content (Table 4.3). Average bending stress of loblolly pine varied from 59.08 – 117.65 MPa, 35.14 – 83.15 MPa, and 37.70 – 74.59 MPa for 10%, 20%, and 30% moisture content. This result is in agreement with the trend reported by several authors for wheat straw, safflower stalk, and sunflower stalk (Annoussamy et al., 2000; Ozbek et al., 2009; Ince et al., 2005). The decrease in bending stress can described as an evidence of departure from brittleness with increase in moisture content. Documented work on mechanical properties of biomass reveals that the mean bending stress for sunflower stalk value varied from 37.77 to 62.09 MPa (Ince et al., 2005); safflower stalk, 21.98 to 59.19 MPa (Shahbazi and Galedar, 2012); wheat stem, 17.74 to 26.77 MPa (Esehaghbeygi et al., 2009); and sugar cane, 9.58 to 9.20 MPa (Taghijarah et al., 2012).

Bending stress was also evaluated as a function of tree height and tree radius. Table 4.3 shows that bending stress reduced with increase in tree height. Galedar et al. (2008) and Esehaghbeygi et al. (2009) obtained similar relationships for alfalfa stems and wheat stem, respectively. It was also observed that the closer the wood materials are to the tree bark, the larger their bending stress. A similar correlation was observed by Bao et al. (2001), who explained that it is as a result of changes in microfibril angle. Typically, larger microfibril angle is present in juvenile wood (wood closer to the tree pith) which causes their mechanical properties to be lower than that of mature wood (wood closer to the tree bark).

Table 4.3. Effect of moisture on bending stress for different tree height and radius combinations.

Tree Height (feet)	Tree Radius (%)	Specific Toughness (kJ/m^2) at Moisture Content Level* (%, w.b.)								
		10			20			30		
		Mean	SD	CV	Mean	SD	CV	Mean	SD	CV
0.0	20	40.97	13.55	33.08	31.78	8.55	26.91	29.58	8.67	29.33
	40	62.47	10.96	17.55	38.53	6.87	17.84	33.13	8.68	26.20
	60	64.26	9.74	15.16	44.15	10.26	23.24	39.52	9.10	23.04
	80	73.55	16.38	22.27	46.79	8.60	18.38	42.92	9.16	21.33
4.6	20	46.10	1.10	24.09	29.80	4.66	15.65	26.70	7.29	27.29
	40	62.33	9.56	15.33	45.15	11.81	26.17	37.99	5.46	14.36
	60	67.43	12.10	17.94	47.74	9.12	19.10	46.08	6.25	13.58
	80	78.43	13.10	16.70	55.43	7.82	14.11	49.72	9.12	18.34
12.0	20	40.60	9.37	23.08	27.08	5.82	21.49	26.02	4.06	15.61
	40	49.42	9.02	18.25	29.94	5.99	20.02	29.11	5.05	17.34
	60	64.14	9.52	14.85	35.70	7.54	21.13	31.77	7.96	25.07
	80	73.03	12.47	17.08	43.04	8.29	19.25	37.53	6.48	17.27
18.0	20	39.38	10.19	25.87	24.76	6.51	26.30	25.13	4.50	17.90
	40	49.28	7.79	15.81	30.61	3.74	12.21	27.64	6.98	25.27
	60	59.59	11.49	19.28	39.63	8.29	20.93	33.93	7.12	21.00
	80	57.71	17.56	30.43	42.69	8.24	19.31	42.68	5.43	12.71

* SD is standard deviation, and CV is coefficient of variation.

4.4.4 Regression Model Development

Regression models (Equation 4.3 and 4.4) were developed to predict the specific toughness and bending stress of loblolly pine wood. The model parameters were selected according to stepwise elimination method ($\alpha = 0.05$). Results from variance inflation factor (VIF) showed that there was a violation of the assumption of orthogonality of model variables (multi-collinearity). Multi-collinearity results in model instability and poor parameter estimation. VIF values that exceed 10 indicates severe multi-collinearity (Montgomery et al., 2012). The VIF results from regression analysis in this study initially varied from 1.00 to 48.06 for bending stress data and 14.58 to 48.40 for toughness data. Therefore, the multi-collinearity is considered severe. Principal component regression was then conducted to remove multi-collinearity by constructing principal components variables and fitting a regression model using the principal components variables.

$$\mathrm{E} = -38.976 + 8.07\mathrm{M} - 4.281\mathrm{H} + 0.914\mathrm{R} - 0.154\mathrm{M}^2 + 0.124\mathrm{H}^2 - 0.007R^2 \tag{4.3}$$

$$\tau = 115.851 - 6.732\mathrm{M} - 0.092\mathrm{H} + 0.793\mathrm{R} + 0.125\mathrm{M}^2 - 0.035\mathrm{H}^2 - 0.003\mathrm{R}^2 \tag{4.4}$$

where

E = specific toughness (kJ/m^2)

Ʈ = bending stress (MPa)

M = moisture content of sample (% wet basis)

H = tree height (feet)

R = tree radius (%)

The parameter estimate, standardized estimate, root mean square error (RMSE), Pearson correlation coefficient (r^2), and adjusted r^2 for specific toughness and bending stress are listed in Table 4.4. The r^2 for specific toughness and bending stress was 0.614 and 0.662, respectively. Guntekin and Aydın (2013) also reported a low r^2 value of 0.670 for the bending stress of Turkish red pine (moisture content and density were used as independent variables). This observation is because of the high variability in the mechanical properties of wood material. The coefficient of variation for specific toughness varied from 17.27% to 52.08% (Table 4.2) while the coefficient of variation for bending stress varied from 12.21% to 33.08% (Table 4.3). Dahlen et al. (2012) also reported that the coefficient of variation for the bending stress of Douglas fir and Southern pine was 39% and 42%, respectively. Moisture content had the highest standardized estimates (Table 4.4) for specific toughness and bending stress. This result implies that moisture content is the most significant factor affecting the specific toughness and bending stress of loblolly pine wood in this study.

Table 4.4. The selected models determined by the stepwise procedure to describe toughness and bending stress of loblolly pine wood.

Response	Variable*	Parameter Estimate	Pr > |t|	Standardized Estimate	R-Square	Adj R-Sq	RMSE
Toughness (kJ/m^2)	Intercept	-38.976	<0.0001	0.000	0.614	0.611	18.537
	M	8.070	<0.0001	63.388			
	H	-4.281	<0.0001	-13.109			
	Ra	0.914	<0.0001	49.458			
	M^2	-0.154	<0.0001	0.344			
	H^2	0.124	<0.0001	-0.297			
	Ra^2	-0.007	<0.0001	0.096			
Bending Stress (MPa)	Intercept	115.851	<0.0001	0.000	0.662	0.659	14.407
	M	-6.732	<0.0001	-63.665			
	H	-0.092	<0.0001	-0.345			
	Ra	0.793	<0.0001	51.652			
	M^2	0.125	<0.0001	-0.380			
	H^2	-0.035	0.0140	-0.149			
	Ra^2	-0.003	0.0100	0.324			

* M = Moisture content; H = Height; Ra = Radius.

Analysis of variance (ANOVA—Table 4.3) shows that there was significant difference ($p < 0.05$) among the specific toughness and bending stress of the trees stems tested in this study. This result validates the randomized complete block design used for the experimentation in this study.

According to Montgomery (2013), the significant effect of block (tree stems) in ANOVA result rationalizes the use of blocking and improves the robustness of the developed model.

Table 4.3. ANOVA result for the effects of moisture content, tree radius, and tree height on the specific toughness and bending stress of loblolly pine wood.

Source	Specific Toughness			Bending Stress		
	DF	Sum of Squares	Pr > F	DF	Sum of Squares	Pr > F
Tree stem	4	14473.70	<.0001	4	17571.06	<.0001
Moisture content	2	177301.85	<.0001	2	142931.88	<.0001
Tree radius	3	18427.84	<.0001	3	96593.52	<.0001
Tree height	3	108900.94	<.0001	3	16082.66	<.0001

4.5 Conclusions

The influences of moisture content, tree height, and tree radius on the bending stress and toughness of loblolly pine were investigated. The resulting analysis established that bending stress and toughness of loblolly pine are polynomial functions of moisture content, tree height, and tree radius. Also, it established that moisture content had the highest weight (standardize estimate) and therefore the most significant factor controlling the mechanical properties of loblolly pine wood among the parameters considered. As moisture content of wood increased, ductility increased. This increase caused a resultant increase in toughness and a decrease in bending stress. More energy will therefore be required to reduce the size of wet loblolly pine wood compared to dried ones. It was also observed that toughness and bending stress increased along longitudinal direction from the tree base to tree top. Equally, toughness and bending stress increased as tree radial distance increased.

Chapter 5: Effects of Grinding and Drying Sequence on the Energy Required To Grind and Dry Loblolly Pine

5.1 Abstract

The ordering of critical unit operations for biomass preparation impacts the total energy requirement and particle properties. In this study, six drying-grinding sequences (12_8, 30_8, 50_8, 30_4_12_8, 50_4_12_8, and 50_4_30_8) were studied. Their effects on the specific grinding energy requirement, minimum drying energy requirement, particle size distribution, aspect ratio, flow index, and bulk density of grinds were quantified and analyzed. Particle properties were measured and analyzed on oven-dried basis to remove the effect of moisture. It was observed that drying-grinding sequence significantly affected the bulk density of grinds. Oven-dried bulk density of grinds varied from 267.083 kg/m^3 (sequence 12_8) to 97.947 kg/m^3 (sequence 50_8). This was attributed to the changes in the breakage characteristics of feedstock which was also observed in the particle morphology. Aspect ratio increased from 4.30 for sequence 12_8 to 6.36 for sequence 50_8. The geometric mean diameter was also drying-grinding sequence specific. It was lowest for sequence 50_8 and highest for sequence 30_8. Furthermore, specific grinding energy requirement was smallest for 12_8. However, a trade-off was noted between specific grinding energy requirement and minimum specific drying energy requirement. This was attributed to the reduction in specific grinding energy requirement and moisture loss during grinding with decrease in moisture content of feedstock. Multi-phase grinding had the least cumulative energy requirement.

5.2 Introduction

Bioenergy from lignocellulosic biomass has low net emissions of greenhouse and acid gases, such as carbon-dioxide (Reijnders, 2006). It is also the only renewable resource that can be used to directly produce liquid transportation fuels, chemicals, polymers, and other products (USDA, 2015; Soetaert and Vandamme, 2009). In the Southeastern United States, loblolly pine is one of the most available biomass resources with about 12.9 million hectares of loblolly pine plantation cultivated in this region (Zhao et al., 2014; Samuelson et al., 2013). Similar to other biomass feedstock, loblolly pine, has to be preprocessed (for example drying, grinding, and densification) before it can be fed into the throat of conversion plants (Tabil et al., 2011).

Drying and grinding are essential to biomass preparation. Drying focuses on the removal of moisture while grinding reduces particle size. Minimization of the energy consumed in these unit operations is pivotal to the viability and economic competiveness of the bioenergy production process since one of the goals is the production of energy. Grinding is however highly energy inefficient and highly energy intensive. Less than 1% of the energy consumed in grinding operations is actually needed (Mohsenin, 1986). Energy consumed in the comminution of biomass is also reported to account for one-third of the total energy consumed in the production of bioethanol (Bitra et al., 2008; Cadoche and Lopez, 1989). In spite of its inefficiency, grinding is required to produce particle sizes that are suitable for downstream operations. Grinding also changes biomass properties such as total surface area, bulk density and energy density, porosity, and inter-particle contact points (Bitra et al., 2009a; Mani et al., 2004).

Energy consumption during grinding depends on initial and final particle size, biomass type and moisture content, prior pretreatments, grinding equipment type, and grinding equipment operating variables (Phanphanich and Mani, 2011; Kokko et al., 2012). Mani et al. (2004) studied the grinding of wheat straws, barley straws, corn stover, and switchgrass using a hammer mill fitted with 3.2 mm, 1.6 mm, or 0.8 mm screen sizes. The authors concluded that there was a negative correlation between specific grinding energy requirement and hammer mill screen size. They also reported that specific grinding energy consumption was significantly different for the biomass type. Switchgrass grinding consumed the highest energy of 113.03 kJ/kg d.b. and 231.42 kJ/kg d.b. (3.2 mm and 0.8 mm screen size, respectively), and corn stover had the least specific energy consumption of 45.16 kJ/kg d.b. and 140.32 kJ/g d.b. (3.2 mm and 0.8 mm screen size, respectively). Soucek et al. (2003) reported that the specific grinding energy for knot weed increased from 210.09 kJ/kg d.b. to 1039.58 kJ/kg d.b. when moisture content of the material was increased from 9.18% to 19.66%. Miao et al. (2011) reported that the specific grinding energy for switchgrass through a 1 mm screen size was about 213.33 kJ/kg d.b. for hammer mill and about 573.33 kJ/kg d.b for knife mill.

The three equations commonly used to estimate grinding energy requirement are Kick's, Rittinger's, and Bond's equations (Earle and Earle, 1983; Henderson and Perry 1970). According to Fellows (2000), the effectiveness of these models depends on the intensity of grinding. Kick's law produced good result for coarse grinding while Rittinger's law works better for fine grinding. Bond's law provides an in-between option for intermediate grinding. Both Rittinger's and Bond's laws suggest that grinding energy requirement rapidly increases as particle size reduces.

Drying is equally energy and cost intensive (Liu et al., 2014). According to Sokhansanj (2013), drying operation costs about 1100 kJ/kg—33.08% of the total energy consumed in the manufacturing biomass pellets. Biomass drying is necessary because thermochemical conversion of biomass usually require low moisture content feedstocks for optimum efficiency. For instance, in the pyrolytic conversion of biomass to bio-oil, feedstocks are usually dried to about 10% for optimal compression (Shaw, 2006). Gasification efficiency of downdraft gasifier increased from 37% to 65% when moisture content of feedstock (woodchips and mustard oil cake mixture) was reduced from 45% to 15% (Kumar et al., 2014).

Grinding and drying are usually done in succession. The typical sequence applying these two unit operations to biomass is to dry and then grind. Various studies have shown that grinding energy increases with increase in moisture content of biomass (Miao et al., 2011; Mani et al., 2004). Therefore, drying before grinding is recommended to enhance grindability. However, these studies are limited to grassy biomass and moisture levels below 20%. Results in chapter 3 show that for hammer milling through 6.350 mm screen size, the specific grinding energy initially increased as moisture content increased from 12% to 30%, then decrease with further increase in moisture content. According to Dooley et al. (2013), one of the limitations of grinding (particularly with hammer mills) is the excessive production of dust and dust pollution. High moisture grinding however helps reduce problems associated with dust generation—material loss, health hazards incidences, and dust explosion risks (Hehar et al., 2014). In addition, grinding is vital to drying rate, as it produces increased surface area which positively correlates with heat flow rate and drying rate (Yu et al., 2006). Several studies have also noted that significant amount of moisture is lost due to heat produced in the grinding chamber. Ghorbani et al. (2010) studied the grinding of alfalfa chops and recorded moisture loss of about 4.10% to 9.37%. Probst et al. (2013) reported 7.20% moisture loss while grinding corncobs at initial moisture content of 20.13%. There is therefore the potential that grinding before drying can reduce the amount of moisture that has to be removed in subsequent drying. Consequently, there is the need to conduct detailed study on the drying-grinding sequence and their influence on biomass quality. This will be especially important for woody biomass, such as loblolly pine, that are harvested at moisture content of about 50% (Cutshall et al., 2011)

This study focuses on understanding the effect of drying-grinding sequence on the energy requirements and the properties of loblolly pine grinds. The specific objectives of the study were to:

a) Investigate the influence of different drying-grinding sequence on the energy required to grind and minimum drying energy requirement (estimated from information on moisture loss during grinding).
b) Quantify and compare the properties (particle size distribution, bulk density, aspect ratio, flow index) of the grinds obtained from (a).

5.3 Materials and Methods

5.3.1 Sample Preparation

Loblolly pine woodchips were collected from West Fraser Mill, Opelika, AL. The moisture content of the woodchips was 52% moisture content, and they were air dried to 12% moisture content and stored indoors for six months before use. Moisture content was measured following the ASABE Standard S358.3 (ASABE Standards, 2012) by drying about 10 g of sample using a convection oven at 103±2°C for 24 h. The values of physical and chemical properties of the loblolly pine wood were measured and are shown (in addition to the standards used) in Table 5.1.

Woodchips were divided and adjusted into three moisture levels (12%, 30%, and 50%) before the first phase grinding experiment by adding and mixing calculated quantity of water. Woodchips at each moisture level was enclosed in an airtight plastic bag placed in a refrigerator (about 4°C) for 10 days after moisture adjustment. Grinds used for the second phase grinding were air dried to a moisture content of 12% or 30% as desired. Table 5.2 shows the notation used to describe the different drying-grinding sequence in subsequent part of this work.

Table 5.1. Properties of loblolly pine woodchips.

Property	Reference	Value**
Ash Content (%)*	NREL (2005)	0.457 (0.08)
Volatile Matter Content (%)*	ISO 562 (2010)	85.47 (0.19)
Energy Content (%)*	ASTM D 5865 (2004)	20.11 (0.07)
Particle Density (kg/m^3)	Fasina (2006)	1455.94 (4.21)
Geometric Mean Diameter (mm)	ASABE S319.3 (2003)	11.62 (6.98)

* Values are reported on dry basis.
** Values in parenthesis are standard deviation from triplicate.

Table 5.2. Notation for drying-grinding sequence used in this study.

Treatment Notation	Step One	Step Two	Step Three	Step Four
12_8	Dried from 50% to 12%	Ground through 3.175 mm screen size	---	---
30_8	Dried from 50% to 30%	Ground through 3.175 mm screen size	---	---
50_8	---	Ground through 3.175 mm screen size	---	---
30_4_12_8	Dried from 50% to 30%	Ground through 6.350 mm screen size	Dried to 12%	Ground through 3.175 mm screen size
50_4_30_8	---	Ground through 6.350 mm screen size	Dried to 30%	Ground through 3.175 mm screen size
50_4_12_8	---	Ground through 6.350 mm screen size	Dried to 12%	Ground through 3.175 mm screen size

5.3.2 Grinding Operation and Specific Grinding Energy

A hammer mill (model 10HBLPK, C.S. Bell Co., Tiffin, Ohio) (Figure 5.1) with 12 swinging beaters powered with a 3 hp, 3-phase electric motor (model C182T17FB29D, LEESON Electric Corporation, Grafton, Wisc.) was used to grind woodchips. Two screen sizes (3.175 mm and 6.350 mm) were fitted to the hammer mill as desired (Table 5.2). For each grinding experimental run, 3 kg of biomass was fed into the hammer mill in 9.5±0.5 min, as established in preliminary studies. Grinding energy was measured by a wattmeter (model A3314/02, EZ meter, Santa Ynez, Cal.). Prior to grinding, power consumption when running the hammer mill empty (unloaded) was determined. Thereafter, grinding energy was recorded for loaded conditions, and specific grinding energy was computed as expressed in Equation 5.1 (Miao et al., 2011). Each experimental treatment was replicated thrice.

Figure 5.1. C.S. Bell Co. hammer mill used for grinding experiments.

$$E = \frac{E_t - E_p}{M_{dry}} \tag{5.1}$$

where

E = specific grinding energy (kJ/kg d.b.)

E_t = total energy consumed (kJ)

E_p = energy consumed by empty hammer mill (kJ)

M_{dry} = dry matter mass of biomass (kg)

5.3.3 Moisture Loss and Minimum Drying Energy Requirements

The difference between the moisture content of loblolly woodchips and grinds was computed as the moisture loss during grinding (Equation 5.2). Moisture content measurement was carried out according to the ASABE Standard S358.3 (ASABE Standards, 2012) by drying about 10 g of sample in an oven at 103±2°C for 24 h. Minimum drying energy requirement was defined as the amount of energy needed to supply latent heat of evaporation (Equation 5.3) (Sokhansanj, 1997). Usually, the actual energy required to remove water from biological material is higher because some energy is used up in overcoming capillary actions (in the case of free water) and breaking chemical bonds (in the case of bound water). The term, minimum, was thus used to acknowledge that calculations were done with the assumptions of water in liquid state. Other assumptions that were made in computing the minimum drying energy are as follows:

1. The moisture content of loblolly pine woodchips is 50% and desired moisture content of grinds is 9%
2. Water is the only substance that wets loblolly pine
3. Drying is carried out at 1.01 MPa (1 atm) and 100ºC.

$$MC_{loss} = \frac{(MC_i - MC_f)}{100 - (MC_i - MC_f)} \tag{5.2}$$

where

MC_{loss} = moisture loss during grinding (%, wet basis)

MC_i = moisture content of woodchips (%, dry basis)

MC_f = moisture content of grinds (%, dry basis)

$$\mathrm{H} = (1 - M_i) \int_{T_i}^{T_f} C_p \, dT + M_i \int_{T_i}^{T_f} C_w \, dT + (M_i - M_f) C_l \quad (5.3)$$

where

H = minimum drying energy (kJ/kg)

M_i = initial moisture content of biomass (%, wet basis)

M_f = final moisture content of biomass (%, wet basis)

T_i = initial temperature of biomass (K)

T_f = drying temperature (K)

C_w = specific heat capacity of water (kJ/kg K) (Table 5.3)

C_p = specific heat capacity of biomass (kJ/kg K) (Table 5.3)

C_l = latent heat of vaporization for free water (kJ/kg) (Table 5.3)

Table 5.3. Relationship between temperature and thermal properties of water and wood.

Parameter	Value	Reference
C_w (kJ/kg K)	$5.117722 - 0.006066\mathrm{T} + 0.000010\mathrm{T}^2$	The Engineering ToolBox, 2015
C_l (kJ/kg)	2257	Earle and Earle, 1983
C_p (kJ/kg K)	$0.103100 - 0.003867\mathrm{T}$	TenWolde et al., 1988

T is temperature in kelvin

The properties of loblolly pine grinds obtained after hammer milling were measured for oven-dried samples (dried in an oven at 45ºC to bone dry weight) to remove the effect of moisture on these properties.

5.3.4 Bulk Density

A bulk density apparatus (Burrows Co., Evanston, Ill.) was used to measure the bulk density of grinds according to ASTM E 873 – 82 Standard (ASTM, 2006). The bulk density apparatus consisted mainly of a container and a hopper. The container of volume (1137 mm^3) was filled with grinds through the funnel which was 0.6 m above its top edge. Grinds in the container was flattened while avoiding compaction, and the mass of the sample that filled the container was measured. The ratio of the mass of grinds filling the container to the volume of the container was computed as the bulk density of the grind (Equation 5.4).

$$\rho_b = \frac{M_s}{V_c} \tag{5.4}$$

where

ρ_b = bulk density of grind (kg/m^3)

M_s = mass of grind filling the container (kg)

V_c = volume of the container (m^3)

5.3.5 Aspect Ratio

Oven-dried samples from each grinds was fractionated into 5 fractions using ISO screens size 1.680 mm (#12), 1.000 mm (18), 0.707 mm (#25), 0.420 mm (#40), and 0.250 mm (#60). Twenty particles then were randomly selected from each fraction. Image of these particles were taken using a light microscope (model DC3-420TH, National Optical Instruments, Schertz, Texas), and their dimensions were measured using the microscope software (Figure 5.2). Particle aspect ratio was calculated as the ratio of the particle length to its width.

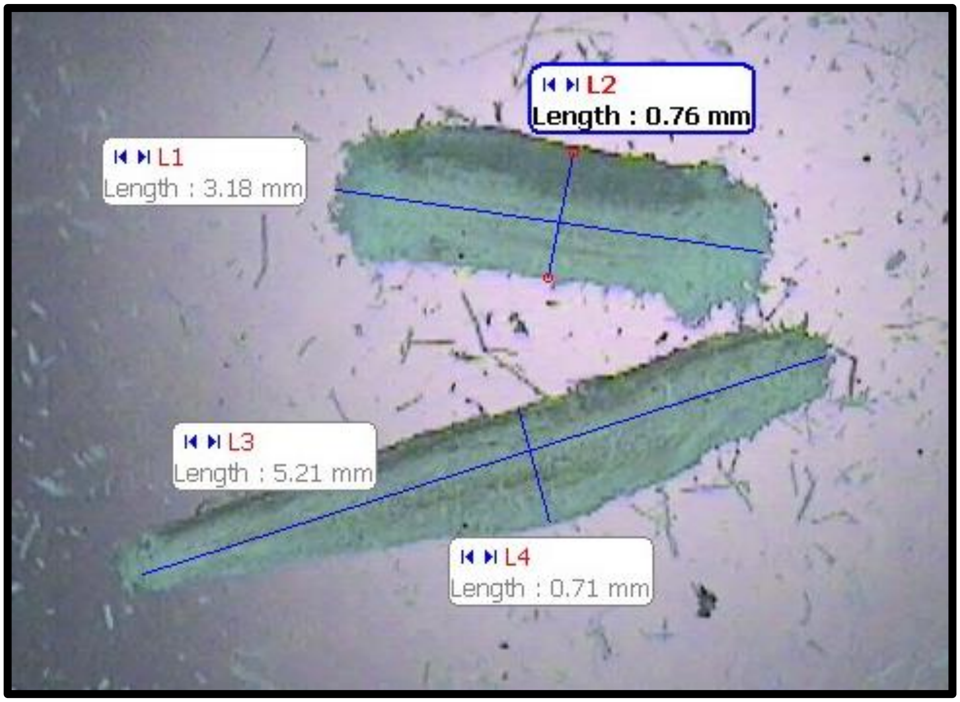

Figure 5.2. Aspect ratio measurement using light microscope.

5.3.6 Flow Index

Flow index of grinds was measured using a powder flow tester (model PFT, Brookfield Engineering Laboratories, Inc., Middleboro, Ma.). The equipment has a 152.40 mm diameter annular split cell sample trough and a vane lid (with 18 small compartments). The equipment sample trough was filled with samples of known mass and levelled using the shaping blade. The flow tester was then operated to shear the sample packed between the vane lid and trough. Flow index (measure of flowability) was obtained from the software of the tester.

5.3.7 Particle Size Distribution

Samples were analyzed to determine their particle size distribution according to ASABE standard S319.4 (ASABE Standards, 2008) with the aid of a Ro-Tap test sieve shaker (model RX – 29, W.S. Tyler, Mentor, Ohio) and 10 ISO screens set. The sizes of the screen in the set were 2.380 mm (#8), 1.680 mm (#12), 1.000 mm (#18), 0.707 mm (#25), 0.420 mm (#40), 0.250 mm (#60), 0.125

mm (#120), 0.088 mm (#170), and 0.053 mm (#270). About 100 g of grinds was subjected to shaking for 15 minutes to ensure adequate classification of the sample according to size. The samples retained on each screen at the end of the sieving period were weighed.

The particle size distribution of grinds was constructed from a plot of the percentage mass retained on the each screen versus the nominal diameter of the screens to construct the. Particle size distribution characteristics: geometric mean diameter, geometric standard deviation, and coefficient of variation were calculated from the mass retained on each screen according to Equation 5.5 – 5.9.

$$\bar{d}_i = \sqrt{(d_i \times d_{i+1})} \tag{5.5}$$

$$d_{gw} = \log^{-1}\left[\frac{\sum_{i=1}^{n} W_i \log \bar{d}_i}{\sum_{i=1}^{n} W_i}\right] \tag{5.6}$$

$$S_{log} = \left[\frac{\sum_{i=1}^{n} W_i \left(\log \bar{d}_i - \log d_{gw}\right)^2}{\sum_{i=1}^{n} W_i}\right]^{1/2} \tag{5.7}$$

$$S_{gw} = \frac{1}{2}\, d_{gw}\left[\log^{-1} S_{log} - \left(\log^{-1} S_{log}\right)^{-1}\right] \tag{5.8}$$

$$CV = 50\,\frac{(d_{84} - d_{16})}{d_{50}} \tag{5.9}$$

where

d_{gw} = geometric mean diameter of particles by mass (mm)

W_i = mass on i^{th} screen (g)

$\bar{d}_i$ = geometric mean diameter of particles on i^{th} screen (mm)

d_i = nominal screen aperture size of the i^{th} screen (mm)

d_{i+1} = nominal screen aperture size in next larger than i^{th} screen, just above in a set (mm)

S_{gw} = geometric standard deviation of particle diameter by mass (mm)

S_{log} = geometric standard deviation of log-normal distribution by mass in ten-based logarithm (dimensionless)

d_{84} = particle diameter at 84% probability (mm)

d_{50} = particle diameter at 50% probability, d_{gw} (mm)

d_{16} = particle diameter at 16% probability (mm)

CV = coefficient of variation

5.3.8 Data Analysis

Experimentation followed a complete randomized design (CRD) with three replication. All statistical analyses were performed using SAS software (SAS 9.3, SAS Institute Inc., Cary, N.C.) but Microsoft Excel 2013 (Microsoft Excel 2013, Microsoft Corporation, Redmond, Wash.) was used to make graphs and plots. Analysis of variance (ANOVA) was implemented to investigate the significance of the differences among treatments at a significance level of 5%. Also, pairwise difference between treatments were carried out using Tukey's HSD (honest significant difference) test.

5.4 Results and Discussion

Values of the physical and chemical characteristics of loblolly pine woodchips are shown in Table 5.1. The energy content recorded was comparable to the value reported by Carter et al. (2013) and Hehar et al. (2014) (20.18 MJ/kg and 20.01 MJ/kg, respectively) for loblolly pine. These values were however higher than the heating value of wheat straw, barley straw, corn stover, switchgrass, and poultry litter (16.81 MJ/kg, 16.12 MJ/kg, 16.18 MJ/kg, 17.61 MJ/kg, and 13.11MJ/kg, respectively) as documented by Mani et al. (2002) and Bernhart and Fasina (2008). Higher energy content of loblolly pine may be as a result of its lower ash content compared to these other biological material. Ash content for wheat straw, barley straw, corn stover, switchgrass, and poultry litter was 8.32%, 10.72%, 7.46%, 5.49%, and 40.96%, respectively (Mani et al., 2002; Bernhart and Fasina, 2008). Fasina (2006) explained that the higher the ash content, the lower the quantity of combustible (mainly carbon, hydrogen, and volatiles). The particle destiny of loblolly pine passing through 1.00 mm screen size was 1455.94 kg/m^3. The geometric mean diameter is also recorded in Table 5.1.

5.4.1 Bulk Density

In order to eliminate the effect of moisture, bulk density of oven-dried samples (oven-dried bulk density) was analyzed. Figure 5.3 is the plot of the effect of the drying-grinding sequence on bulk density of oven-dried sample. There was a significant difference ($p < 0.05$) in the oven-dried bulk density of the drying-grinding sequences considered in this study. Sequence 12_8 had the highest oven dried bulk density of 267.08 ± 3.69 kg/m^3 and sequence 50_8 had the least oven dried bulk density of 97.95 ± 1.73 kg/m^3. This result implies that the cost of transportation and storage will be lowest for sequence 12_8 and highest for sequence 50_8. However, the oven-dried bulk density of sequence 50_4_12_8 was not significantly different from that of sequence 30_8. The difference in bulk density was ascribed to the variation in the morphology of grind particles (Figure 5.4). It was observed that breakage of woodchips ground through the 3.175 mm screen size at high moisture content occur along fibers which leads to the production of woolly and low bulk density grinds. A similar finding was reported in chapter 3. Lehmann et al. (2012) studied different grinding processes for wood-miscanthus pellets and also reported the production of a highly fibrous structure for wet comminution with shear stress.

The significant difference ($p < 0.05$) between the oven-dried bulk density of samples from sequences 50_8 and 50_4_30_8, 50_8 and 50_4_12_8, and 30_8 and 30_4_12_8 (Figure 5.3) shows that multi-phase grinding had effect on the oven-dried bulk density of loblolly pine grinds. Generally, the grinds produced from multi-phase grinding had higher oven-dried bulk density when compared to grinds from direct grinding where initial moisture content of the woodchips were the same. This result is because the initial grinding of woodchips through 6.350 mm screen at high moisture content produced less woolly grinds than 3.175mm (Figure 3.14). It was therefore concluded that multi-phase grinding reduced the fluffiness observed in high moisture content.

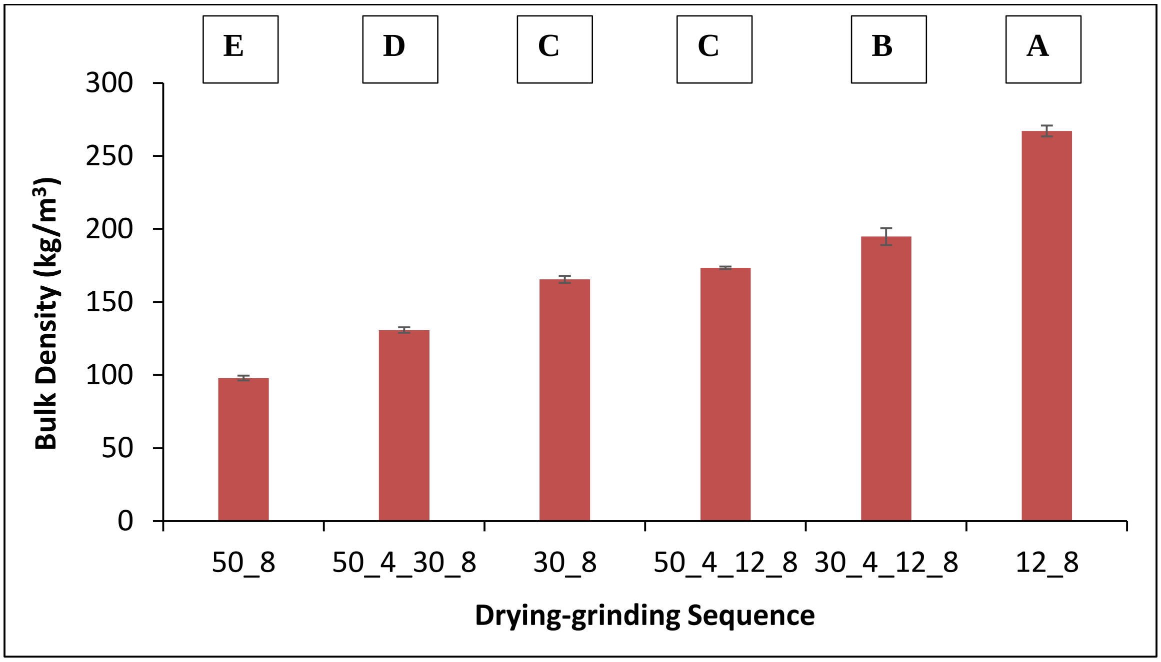

Figure 5.3. Effect of drying-grinding sequence on the bulk density of oven-dried grinds (means with different letters are significantly different at 0.05 significance level using Tukey's multiple comparison).

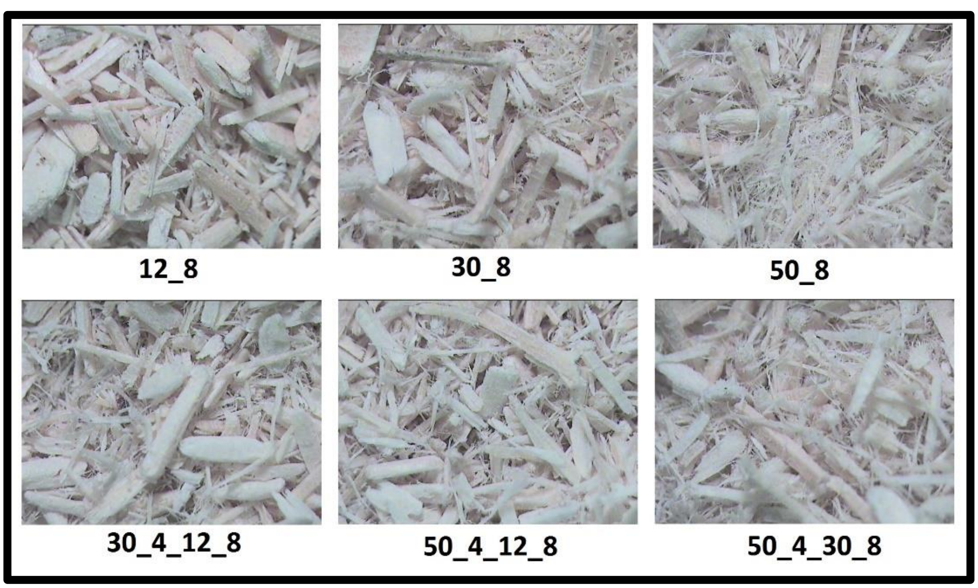

Figure 5.4. Morphology of grinds from drying-grinding sequence.

5.4.2 Aspect Ratio

Analysis of variance conducted showed that there was a significant effect ($p < 0.05$) of drying-grinding sequence on the aspect ratio of grinds. The mean aspect ratio of grinds ranged from 4.30 (for sequence 12_8) to 6.36 (for sequence 50_8). This result further validates the discussion on the changes in morphology in section 5.4.1. The high aspect ratios reported on this study are supported by the observation of Lu et al. (2008) that while pulverized coal has an aspect ratio close to that of a sphere (the aspect ratio of a sphere is 1.00), biomass usually has a large aspect ratio, and typically characterized as cylinders. Miao et al. (2011) also noted that the aspect ratios for miscanthus and switchgrass particles varied from 5 to 15. Equation 5.10 (Momenikouchaksaraei, 2013; Lu et al., 2004) indicates that larger aspect ratio would cause particles to heat up (and dry up) faster because of the increased surface area.

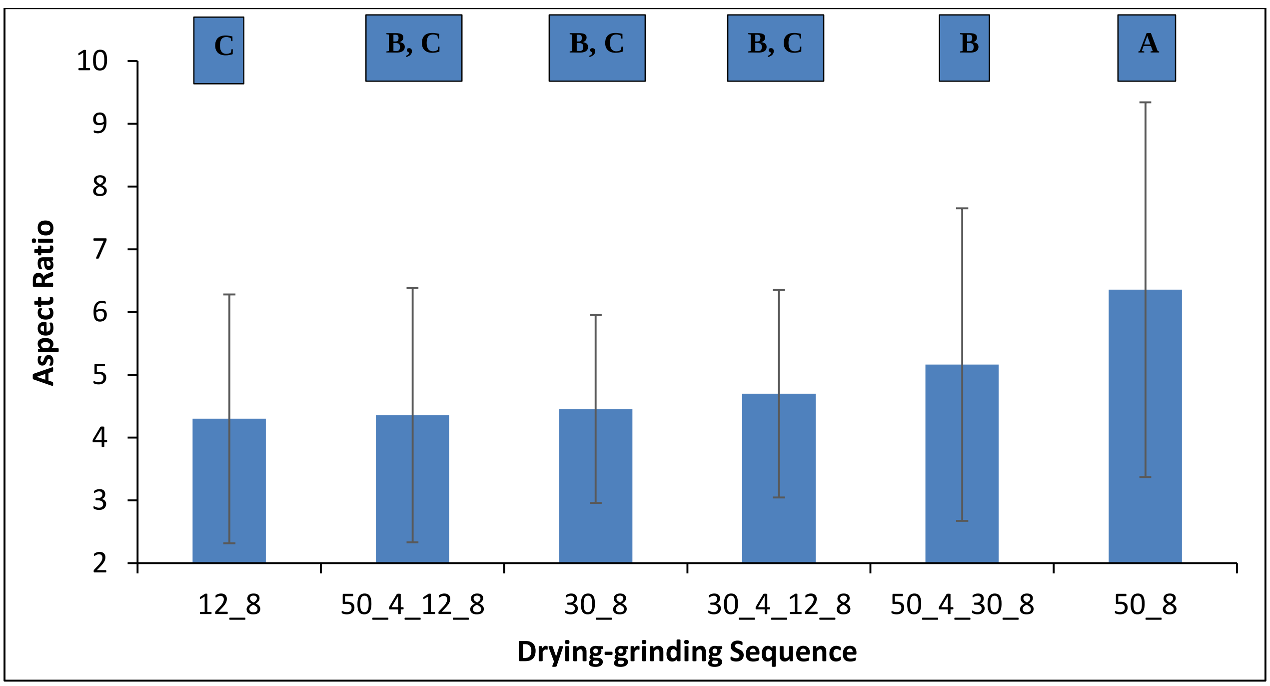

Figure 5.5. Effect of drying-grinding sequence on the aspect ratio of oven-dried grinds (means with different letters are significantly different at 0.05 significance level using Tukey's multiple comparison).

$$\text{Aspect Ratio} = \frac{\text{Particle Surface Area}}{\text{Particle Volume}} \tag{5.10}$$

5.4.3 Flow Index

The flow index of oven-dried grinds in this study was significantly affected ($p < 0.05$) by drying-grinding sequences (Figure 5.6). The flow index of loblolly pine grinds varied from 3.011 for sequence 12_8 to 2.085 for sequence 50_8. However, the flow index values for sequences 12_8, 50_4_12_8, and 30_4_12_8 were not significantly different ($p < 0.05$). Fasina (2006) reported that flow index is commonly used to categorize flowability of particulate materials as expressed in Table 5.4. The grinds from all the drying-grinding sequences considered in this study may be therefore classified as cohesive. Similar results were reported by Oginni (2014), who observed that the flow index of loblolly pine grinds ranged between 2.54 and 3.19.

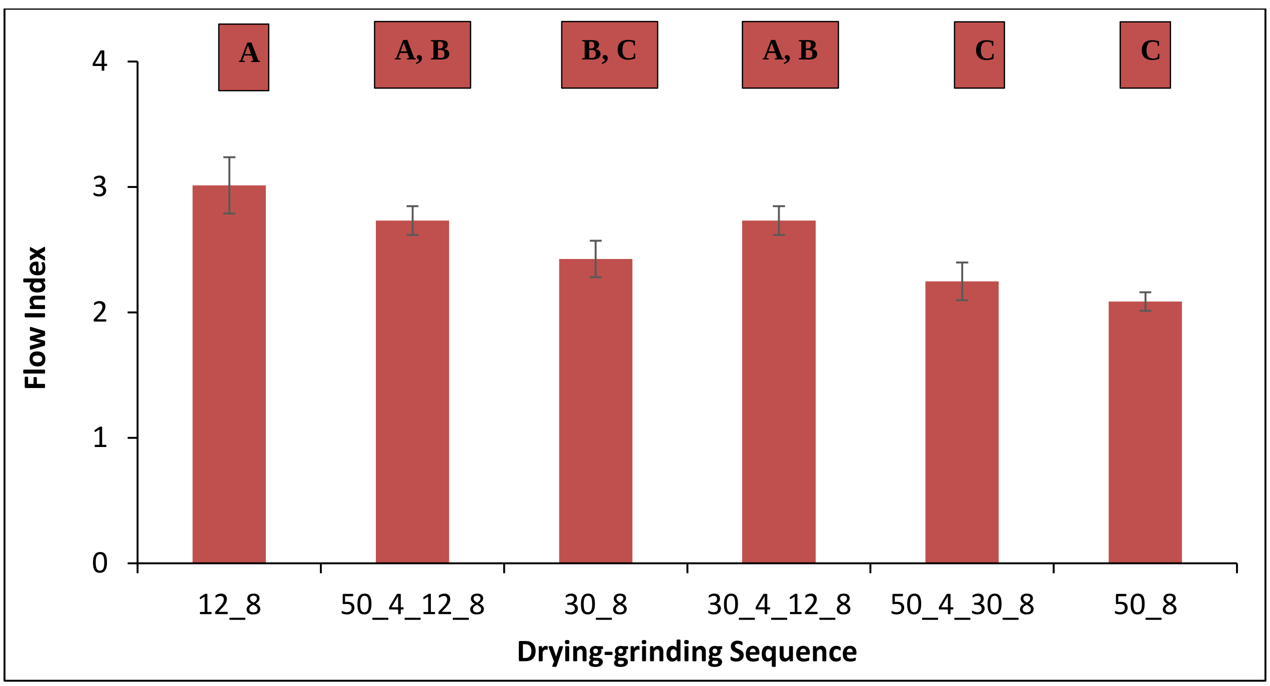

Figure 5.6. Effect of drying-grinding sequence on the flow index of oven-dried grinds (means with different letters are significantly different at 0.05 significance level using Tukey's multiple comparison).

Table 5.4. Classification of powder flowability by flow index (Jenike, 1964).

	Flowability			
	Very Cohesive	**Cohesive**	**Easy Flowing**	**Free Flowing**
Flow Index (FI)	FI < 2	2 < FI < 4	4 < FI < 10	FI > 10

5.4.4 Particle Size Distribution

Generally, if all other parameters remain fixed, grinding energy requirement is a function of the particle size distribution of both the feedstock and the resulting grinds. Also, particle size distribution of grinds is a major parameter in assessing the attributes of the grinds. It was therefore important to quantify the particle size distribution of the grinds as affected by treatments considered in this study. The particle size distribution of grinds from this study followed a slightly-skewed normal distribution (Figure 5.7). This result was also observed by Miao et al. (2011) for miscanthus, switchgrass, and willow. Probst et al. (2013) reported similar particle size distribution for corn ground at 16.02% moisture content. All the drying-grinding sequences produces grinds with similar particle size distribution, with highest mass fraction retained on sieve #18 (1.00 mm).

Statistical analysis indicated that drying-grinding sequence had significant effect ($p < 0.05$) on the geometric mean diameter, geometric standard deviation, and coefficient of variation (Table 5.5). Sequence 30_8 sequence produced the largest geometric mean diameter of 0.835 mm, while the smallest geometric mean diameter was produced by sequence 50_8. The difference between the geometric mean diameter of grinds from sequences with their final grinding phase through 3.175 mm screen size at 12% moisture content (sequences 12_8, 30_4_12_8, and 50_4_12_8) was not significant ($p < 0.05$). A similar observation was made for sequences with their final grinding phase through 3.175 mm screen size at 30% moisture content (sequences 30_8 and 50_4_30_8).

Grinds produced generally possessed large standard deviation which is common for hammer milled grinds (Ghorbani et al., 2010; Bitra et al., 2009a; Himmel et al., 1985). The geometric standard deviation of grinds varied from 0.640 mm (sequence 12_8) to 0.835 mm (sequence 30_8). The coefficient of variation was lowest for sequence 12_8 and was not significantly different ($p < 0.05$) from sequence 50_4_12_8.

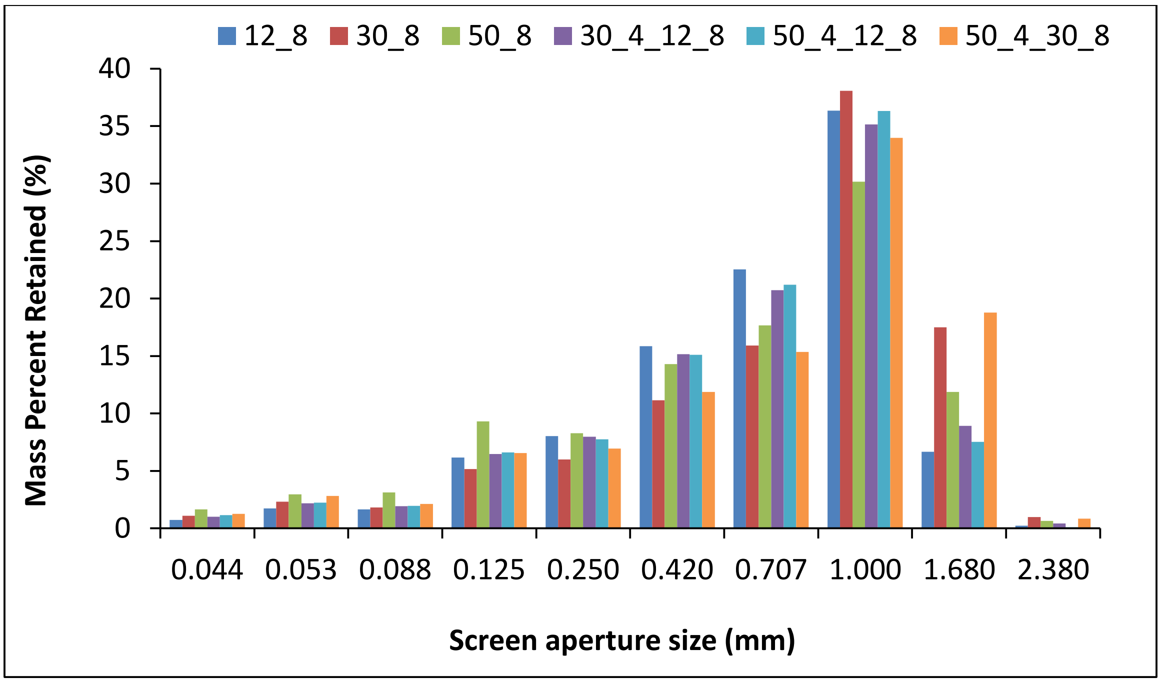

Figure 5.7. Effect of drying-grinding sequence on the particle size distribution of oven-dried grinds.

Table 5.5. Effect of drying-grinding sequence on particle size distribution of oven-dried grinds.

Treatment	Geometric Mean Diameter* (mm)	Geometric Standard Deviation* (mm)	Coefficient of Variation*
12_8	0.776 (0.032)[b, c]	0.640 (0.004)[d]	82.6 (2.9)[d]
30_4_12_8	0.738 (0.003)[c]	0.671 (0.008)[c]	90.9 (1.4)[c]
30_8	0.835 (0.014)[a]	0.820 (0.002)[a]	98.2 (1.7)[b]
50_4_12_8	0.740 (0.013)[c]	0.654 (0.015)[c, d]	88.5 (3.7)[c, d]
50_4_30_8	0.802 (0.003)[a, b]	0.841 (0.005)[a]	104.8 (0.2)[a]
50_8	0.649 (0.016)[d]	0.715 (0.007)[b]	110.2 (2.1)[a]

* Values in parenthesis are standard deviation from triplicate. In each column, values with the same letter are not significantly different ($p < 0.05$)

5.4.5 Specific Grinding Energy Consumption

There were significant differences ($p < 0.05$) among the specific grinding energy requirements for the different drying-grinding sequence considered in this study. Sequence 50_8 had the highest grinding energy requirement of 818.162 kJ/kg d.b. and sequence 12_8 had the lowest grinding energy requirement of 211.639 kJ/kg d.b. (Figure 5.8). The variance between these two drying-grinding sequences can be attributed directly to moisture effect on grindability which agrees with the results of Miao et al. (2011) where the grinding energy for miscanthus was about 50% higher when moisture content increased from 7% to about 15%. Also, it was observed that two-phase grinding coupled with intermediate drying caused a drop in the specific energy consumption during the grinding of loblolly pine woodchips. For instance, sequence 50_4_12_8 and sequence 50_4_30_8 required 543.059 kJ/kg d.b. and 379.336 kJ/kg d.b. less specific grinding energy than sequence 50_8. And, specific grinding energy consumption for sequence 30_4_12_8 was 283.373 kJ/kg d.b. lower than sequence 30_8. However, there was no significant difference between the specific grinding energy requirements for sequences 30_4_12_8 and 50_4_12_8.

Furthermore, in order to compare grinding energy requirements for the different sequences in relation to the particle size produced, the three main grinding equations (Kick, Rittinger, and Bond) (Equations 5.11 – 5.13) were used and their constants were compared. Pairwise comparison for all the models show a consistent trend with the analysis carried out on the specific grinding energy without reference to particle size (Table 5.6).

$$\text{Kick:} \quad E = -K_K \ln \frac{L_f}{L_p} \tag{5.11}$$

$$\textit{Rittinger:} \quad E = -K_R \left(\frac{1}{L_p} - \frac{1}{L_f} \right) \tag{5.12}$$

$$\text{Bond:} \quad E = -K_b \left(\frac{1}{L_p^{0.5}} - \frac{1}{L_f^{0.5}} \right) \tag{5.13}$$

where

E = energy requirement (kJ/kg d.b.)
K_K, K_R, K_b = Kick, Rittinger, and Bond constant respectively (characteristic grindability constant)
L_p = average particle size of grinds (mm)
L_f = average particle size of feedstock (mm)

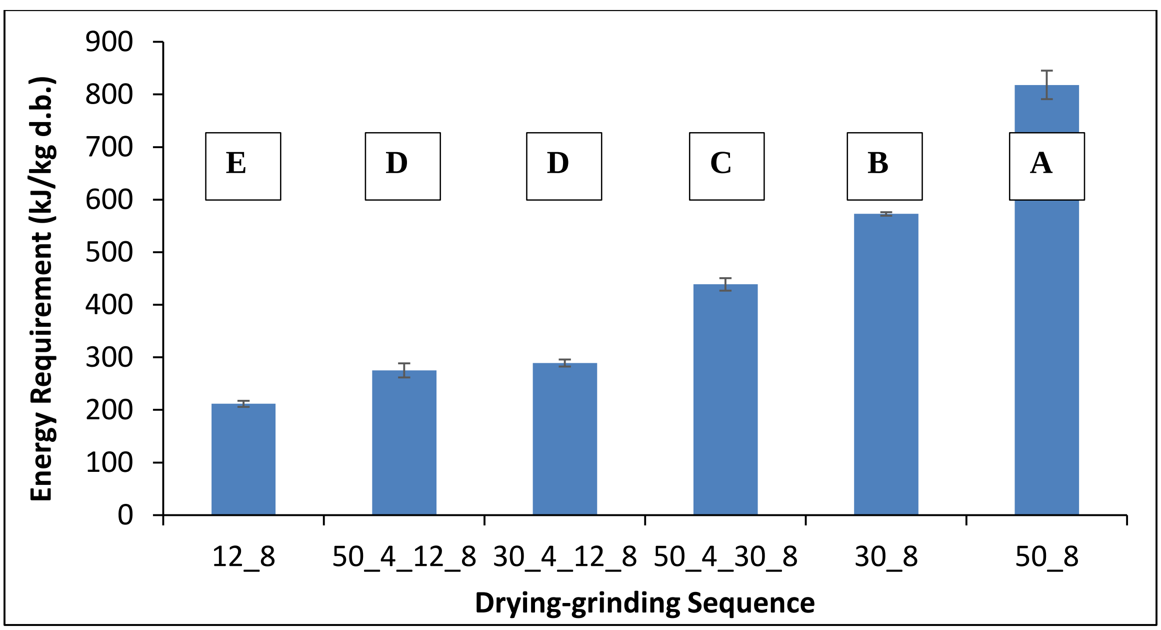

Figure 5.8. Effect of drying-grinding sequence on the specific grinding energy requirement of loblolly pine woodchips (means with different letters are significantly different at 0.05 significance level using Tukey's multiple comparison).

Table 5.6. Effect of drying-grinding sequence on the specific grinding energy requirement and grindability of loblolly pine woodchips

Sequence	Specific Grinding Energy (kJ/kg d.b.)*	Rittinger Constant (kJ/kg mm)*	Kick Constant (kJ/kg)*	Bond Constant (kJ/kg mm$^{0.5}$)*
50_8	818.16[a]	562.66[a]	283.62[a]	863.30[a]
30_8	572.84[b]	515.88[b]	217.63[b]	715.60[b]
50_4_30_8	438.83[c]	378.13[c]	164.16[c]	533.11[c]
30_4_12_8	288.57[d]	228.37[d]	105.05[d]	332.69[d]
50_4_12_8	275.10[d]	217.60[d]	99.92[d]	316.68[d]
12_8	211.64[e]	176.17[e]	78.22[e]	251.54[e]

* In each column, values with the same letter are not significantly different ($p < 0.05$)

5.4.6 Moisture Loss during Grinding

Moisture loss during grinding ranged from 3.15% (sequence 12_8) to 37.52% (sequence 50_8). Moisture loss during grinding was significantly affected ($p < 0.05$) by drying-grinding sequence (Figure 5.9). The comparison of the value of moisture loss during one-phase grinding (sequences 12_8, 30_8, and 50_8) shows that the higher the moisture content of feedstock, the higher the moisture loss during grinding. This observation is because there is increase in the quantity of water held by capillary action in the cell lumina at higher moisture content (Passarini et al., 2014). Moisture in the cell lumen are easily exposed during grinding and the heat generated in the grinder as a result of the beaters impact provides the latent heat of vaporization used to remove them.

Two-phase grinding had lower moisture loss during grinding when compared to one-phase grinding where the initial moisture content of woodchips was the same. One direct implication of moisture loss during grinding is that the amount of water needed to be taken off in the course of subsequent drying process is reduced, which in turn translates into a decrease in the required drying energy requirement.

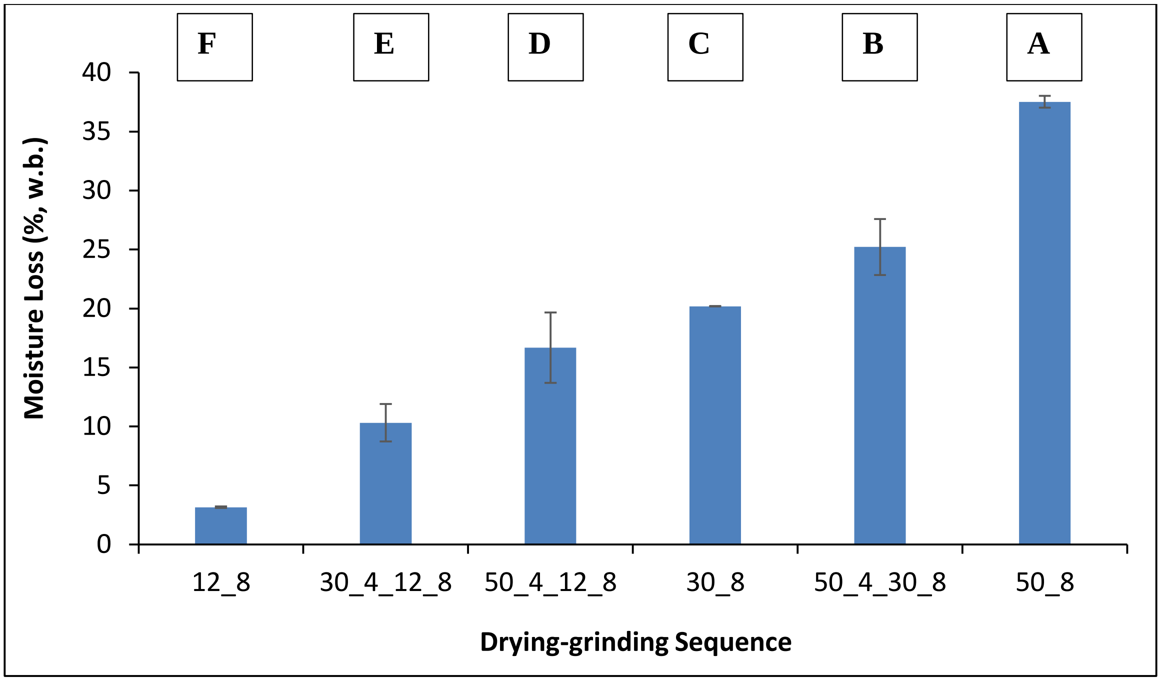

Figure 5.9. Effect of drying-grinding sequence on the moisture loss during grinding of loblolly pine woodchips (means with different letters are significantly different at 0.05 significance level using Tukey's multiple comparison).

5.4.7 Cumulative Energy Requirement

Drying energy required was calculated using the moisture loss data (Equation 5.3) and it was observed to vary from 695.252 kJ/kg (for sequence 50_8) to 1082.325 kJ/kg (for sequence 12_8). A study of the trend for the calculated minimum drying energy and the specific grinding energy requirement suggests a trade-off between both. This trade-off is because grinding of higher moisture content feedstock result in higher specific grinding energy requirement (Figure 5.8) and higher moisture loss (Figure 5.9), which reduces the drying energy requirement.

Cumulative energy requirement for loblolly pine woodchips was calculated as the sum of the minimum drying energy and specific grinding energy (Figure 5.10). Drying-grinding sequence significantly influenced the cumulative energy requirement. This result is in agreement with the conclusion of Miao et al. (2011) in their work on the hammer mill and knife mill grinding of switchgrass and miscanthus. The authors reported that the position of grinding operation in biomass logistics chain affects the performance of the entire chain. Furthermore, the comparison of 50_4_30_8 and 50_8, 50_4_12_8 and 50_8, and 30_4_12_8 and 30_8 shows that two-phase grinding had significantly lower cumulative energy requirement. This result implies that two-phase grinding can be utilized in the reduction of the total energy consumption for drying and grinding operation. Sokhansanj (2013) evaluated two pathways to the production of torrefied biomass pellet and reported that the cumulative energy required to grind and dry Douglas fir woodchips was about 1200 kJ/kg, which is comparable to the value reported for sequences 12_8, 50_4_30_8, 30_4_12_8, and 50_4_12_8 in this study.

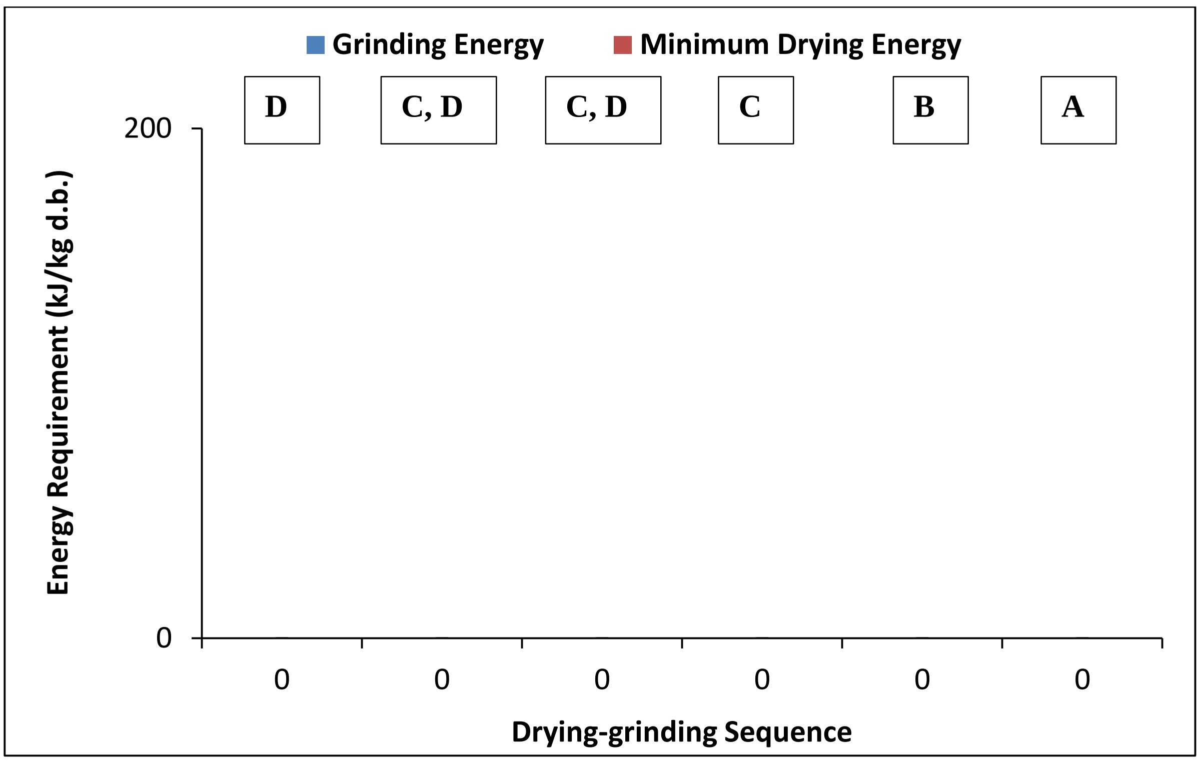

Figure 5.10. Effect of drying-grinding sequence on the cumulative energy consumption for drying and grinding of loblolly pine (means with different letters are significantly different at 0.05 significance level using Tukey's multiple comparison).

5.5 Conclusion

Analysis showed that the oven-dried bulk density of grinds was significantly affected by drying-grinding sequence. This observation was attributed to the variation in the morphology of resulting particles, which was captured in the aspect ratio results. Aspect ratio of grinds increased as the moisture content of feedstock increased and was lower for sequences with two-phase grinding. Particles ground directly through 3.175 mm screen size at a high moisture content had larger aspect ratio compared to other drying-grinding sequence. It is noteworthy that although the mean aspect ratio reported were as expected (judging from appearance), the variability (standard deviation) recorded for each measurement was large. The particle size distribution of grinds from the all the drying-grinding sequences was similar, but the geometric mean diameter of grinds was significantly affected ($p < 0.05$) by the drying-grinding sequence. Furthermore, it was observed that the specific grinding energy requirement and moisture loss during grinding were also significantly affected ($p < 0.05$) by the drying-grinding sequence. The specific grinding energy requirement reduced with decrease in moisture content of feedstock and the addition of a second-phase grinding. From the evaluation of the potential cumulative energy consumed in the drying and grinding of loblolly pine woodchips, the higher grinding energy requirement observed in high moisture grinding was compensated by a corresponding lower drying energy. Additionally, two-phase grinding had lower cumulative energy requirement than one-phase grinding.

Chapter 6: Summary and Recommendations

6.1 Summary

This work provides relevant information on the influence of moisture content, storage time, and hammer mill screen size on the grinding energy requirement and some physical properties of loblolly pine grinds. Analysis showed that the volatile matter content of loblolly pine woodchips decreased with increasing storage time, which resulted in an increase in ash content and a decrease in energy content. Results from three point bending test of loblolly pine wood show that as moisture content increased, specific toughness increased and bending stress decreased. During grinding, the grinding difficulty (and specific grinding energy requirement) of loblolly pine woodchips increased with increase in moisture content. This relationship was attributed to the plasticizing effect of moisture. However, above 30% moisture content, the grinding difficulty decreased with increasing moisture content for 6.350 mm hammer mill screen size and the slope of the grinding difficulty—moisture content curve decreased for 3.175 mm hammer mill screen size. This result was ascribed to the lubricating effect of free water present in the woodchips. The effect of storage time on the grinding difficulty was not significant ($p < 0.05$). The heat generated in the grinding chamber of the hammer mill caused the reduction moisture which will decrease the energy required to dry the sample after grinding. It was also observed that bulk density increased as hammer mill screen size and moisture content of woodchips decreased. Storage time significantly affected ($p < 0.05$) the geometric mean diameter of grinds. Additionally, the geometric mean diameter of grinds initially increased as moisture increased from 12% to 20%, then decreased as moisture content further increased through 50%.

Result showed that drying-grinding sequences significantly affected ($p < 0.05$) the bulk density, aspect ratio, flow index, and geometric mean diameter of the grinds produced. Aspect ratio of biomass grinds varied from 4.30 to 6.36. The cumulative energy requirement for drying and grinding of loblolly pine woodchips reduced with the addition of a second grinding phase.

6.2 Recommendations

It is important to note that the choice of drying-grinding sequence should be guided by the quality of feedstock needed for the target conversion process as well as the findings in this work. The data provided in this work may be used in selecting biomass logistics sequence, which will result in minimum energy consumption and production of suitable feedstock for biomass conversion plants. Grinding at high moisture content should be carried out near the bio-refinery site in order to avoid high transportation cost which may result from the low bulk density of resulting grinds. From a grinding energy requirement perspective, there are no advantages to storing of loblolly pine woodchips. However, it is important to investigate how this will change with different storage systems. The quantity of moisture loss during grinding should be factored in the selection of the initial moisture content of woodchips and in the mass balance calculation during grinding operation.

There is a need to evaluate the response of the different particle size distribution and morphology of grinds to different handling and conversion processes. Furthermore, in making techno-economic comparisons between wet and dry grinding, factors such as grinding rate, drying rate, quantity of material loss through dust, health hazard and fire explosion potential should be considered.

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Appendix A–Data

Table A.1–1. Effect of storage time on the volatile matter content of loblolly pine woodchips.

Storage Time (month)	Volatile Matter Content (%, d.b.)		
	Values	Mean	Standard Deviation
0	86.34		
0	86.76	86.74	0.40
0	87.13		
2	85.70		
2	86.12	86.23	0.59
2	86.86		
4	85.40		
4	85.42	85.53	0.21
4	85.78		
6	85.28		
6	85.47	85.47	0.19
6	85.65		

Table A.1–2. Effect of storage time on the ash content of loblolly pine woodchips.

Storage Time (month)	Ash Content (%, d.b.)		
	Values	Mean	Standard Deviation
0	0.32		
0	0.30	0.31	0.01
0	0.30		
2	0.40		
2	0.39	0.39	0.01
2	0.38		
4	0.46		
4	0.42	0.42	0.04
4	0.38		
6	0.54		
6	0.44	0.46	0.08
6	0.39		

Table A.1–3. Effect of storage time on the energy content of loblolly pine woodchips.

Storage Time (month)	Energy Content (MJ/kg)		
	Values	Mean	Standard Deviation
0	20.24		
0	20.28	20.32	0.11
0	20.44		
2	20.26		
2	20.29	20.28	0.02
2	20.30		
4	20.16		
4	20.20	20.19	0.03
4	20.21		
6	20.05		
6	20.09	20.11	0.07
6	20.19		

Table A.2–1. Effects of moisture content, and screen size on moisture loss during grinding of loblolly pine woodchips (0 month storage time).

Moisture Content (%, w.b.)	Screen Size (mm)	Moisture Loss (%, w.b.)		
		Values	Mean	Standard Deviation
12	3.175	3.68		
12	3.175	4.17	3.66	0.52
12	3.175	3.14		
12	6.350	1.80		
12	6.350	1.51	1.76	0.23
12	6.350	1.96		
20	3.175	7.64		
20	3.175	6.78	6.87	0.74
20	3.175	6.17		
20	6.350	6.05		
20	6.350	5.41	5.71	0.32
20	6.350	5.68		
30	3.175	15.93		
30	3.175	15.42	15.76	0.29
30	3.175	15.93		
30	6.350	9.56		
30	6.350	8.73	8.89	0.60
30	6.350	8.38		
40	3.175	20.90		
40	3.175	20.40	20.97	0.60
40	3.175	21.60		
40	6.350	10.65		
40	6.350	11.61	10.66	0.95
40	6.350	9.72		
50	3.175	30.51		
50	3.175	31.49	29.42	2.78
50	3.175	26.25		
50	6.350	16.37		
50	6.350	15.58	16.41	0.85
50	6.350	17.28		

Table A.2–2. Effects of moisture content, and screen size on moisture loss during grinding of loblolly pine woodchips (2 month storage time).

Moisture Content (%, w.b.)	Screen Size (mm)	Moisture Loss (%, w.b.)		
		Values	Mean	Standard Deviation
12	3.175	3.72		
12	3.175	3.96	3.95	0.24
12	3.175	4.19		
12	6.350	1.51		
12	6.350	1.74	1.74	0.23
12	6.350	1.96		
20	3.175	10.31		
20	3.175	10.97	10.87	0.52
20	3.175	11.34		
20	6.350	6.73		
20	6.350	6.48	6.51	0.21
20	6.350	6.31		
30	3.175	18.63		
30	3.175	19.87	19.63	0.90
30	3.175	20.38		
30	6.350	10.25		
30	6.350	8.05	9.29	1.13
30	6.350	9.59		
40	3.175	20.49		
40	3.175	20.05	20.07	0.41
40	3.175	19.67		
40	6.350	16.92		
40	6.350	16.59	16.86	0.25
40	6.350	17.07		
50	3.175	30.61		
50	3.175	31.20	30.79	0.36
50	3.175	30.55		
50	6.350	15.73		
50	6.350	16.18	15.93	0.23
50	6.350	15.88		

Table A.2–3. Effects of moisture content, and screen size on moisture loss during grinding of loblolly pine woodchips (4 month storage time).

Moisture Content (%, w.b.)	Screen Size (mm)	Moisture Loss (%, w.b.)		
		Values	Mean	Standard Deviation
12	3.175	3.14		
12	3.175	3.31	3.32	0.18
12	3.175	3.50		
12	6.350	1.09		
12	6.350	1.22	1.17	0.07
12	6.350	1.21		
20	3.175	11.11		
20	3.175	11.07	11.09	0.02
20	3.175	11.08		
20	6.350	6.44		
20	6.350	6.43	6.46	0.04
20	6.350	6.51		
30	3.175	18.44		
30	3.175	17.13	17.76	0.66
30	3.175	17.72		
30	6.350	9.10		
30	6.350	9.65	9.35	0.28
30	6.350	9.29		
40	3.175	25.47		
40	3.175	24.21	24.82	0.63
40	3.175	24.77		
40	6.350	10.83		
40	6.350	13.03	11.97	1.11
40	6.350	12.04		
50	3.175	36.45		
50	3.175	34.98	35.99	0.87
50	3.175	36.52		
50	6.350	20.56		
50	6.350	20.00	20.26	0.28
50	6.350	20.20		

Table A.3–1. Effects of moisture content and screen size on the oven dried bulk density of loblolly pine grinds (0 month storage time).

Moisture Content (%, w.b.)	Screen Size (mm)	Oven Dried Bulk Density (kg/m^3)		
		Values	Mean	Standard Deviation
12	3.175	287.80		
12	3.175	301.91	295.64	7.18
12	3.175	297.21		
12	6.350	282.59		
12	6.350	272.40	274.25	7.58
12	6.350	267.76		
20	3.175	258.05		
20	3.175	257.93	265.65	13.27
20	3.175	280.97		
20	6.350	251.86		
20	6.350	229.70	234.01	16.14
20	6.350	220.46		
30	3.175	162.83		
30	3.175	163.68	166.13	5.00
30	3.175	171.89		
30	6.350	172.55		
30	6.350	147.37	154.12	16.15
30	6.350	142.44		
40	3.175	113.01		
40	3.175	102.20	107.61	5.41
40	3.175	107.61		
40	6.350	128.48		
40	6.350	136.45	133.15	4.16
40	6.350	134.53		
50	3.175	102.63		
50	3.175	103.47	101.62	2.51
50	3.175	98.76		
50	6.350	137.92		
50	6.350	137.12	137.43	0.43
50	6.350	137.27		

Table A.3–2. Effects of moisture content and screen size on the oven dried bulk density of loblolly pine grinds (2 month storage time).

Moisture Content (%, w.b.)	Screen Size (mm)	Oven Dried Bulk Density (kg/m^3)		
		Values	Mean	Standard Deviation
12	3.175	255.80		
12	3.175	260.08	258.50	2.35
12	3.175	259.62		
12	6.350	238.34		
12	6.350	239.11	238.82	0.42
12	6.350	239.00		
20	3.175	238.35		
20	3.175	236.62	237.52	0.87
20	3.175	237.58		
20	6.350	198.46		
20	6.350	199.07	199.25	0.90
20	6.350	200.24		
30	3.175	187.49		
30	3.175	182.07	183.85	3.16
30	3.175	181.98		
30	6.350	143.73		
30	6.350	145.90	144.77	1.08
30	6.350	144.70		
40	3.175	115.33		
40	3.175	113.00	113.89	1.26
40	3.175	113.34		
40	6.350	126.24		
40	6.350	126.02	126.73	1.05
40	6.350	127.93		
50	3.175	100.81		
50	3.175	101.41	102.36	2.18
50	3.175	104.84		
50	6.350	127.85		
50	6.350	131.65	120.49	16.15
50	6.350	101.97		

Table A.3–3. Effects of moisture content and screen size on the oven dried bulk density of loblolly pine grinds (4 month storage time).

Moisture Content (%, w.b.)	Screen Size (mm)	Oven Dried Bulk Density (kg/m^3)		
		Values	Mean	Standard Deviation
12	3.175	263.80		
12	3.175	269.78	266.88	2.99
12	3.175	267.05		
12	6.350	235.53		
12	6.350	244.78	240.51	4.67
12	6.350	241.23		
20	3.175	245.79		
20	3.175	244.48	245.47	0.88
20	3.175	246.15		
20	6.350	206.76		
20	6.350	201.63	204.90	2.84
20	6.350	206.31		
30	3.175	163.06		
30	3.175	158.80	162.01	2.83
30	3.175	164.16		
30	6.350	140.87		
30	6.350	146.79	144.92	3.52
30	6.350	147.11		
40	3.175	124.83		
40	3.175	119.59	123.43	3.37
40	3.175	125.86		
40	6.350	141.87		
40	6.350	144.43	144.28	2.34
40	6.350	146.55		
50	3.175	117.19		
50	3.175	110.47	114.28	3.45
50	3.175	115.17		
50	6.350	138.22		
50	6.350	134.80	136.46	1.71
50	6.350	136.36		

Table A.3–4. Effects of moisture content and screen size on the wet bulk density of loblolly pine grinds (0 month storage time).

Moisture Content (%, w.b.)	Screen Size (mm)	Wet Bulk Density (kg/m^3)		
		Values	Mean	Standard Deviation
12	3.175	263.85		
12	3.175	306.80	290.90	23.55
12	3.175	302.05		
12	6.350	284.67		
12	6.350	281.09	275.24	13.36
12	6.350	259.95		
20	3.175	222.65		
20	3.175	213.83	226.13	14.37
20	3.175	241.92		
20	6.350	239.33		
20	6.350	217.80	212.49	29.86
20	6.350	180.32		
30	3.175	148.89		
30	3.175	144.56	150.30	6.56
30	3.175	157.44		
30	6.350	180.14		
30	6.350	177.38	177.88	2.05
30	6.350	176.12		
40	3.175	132.95		
40	3.175	135.33	132.20	3.56
40	3.175	128.32		
40	6.350	165.00		
40	6.350	170.42	169.58	4.23
40	6.350	173.32		
50	3.175	114.64		
50	3.175	113.11	113.12	1.52
50	3.175	111.61		
50	6.350	195.73		
50	6.350	200.79	198.14	2.54
50	6.350	197.89		

Table A.3–5. Effects of moisture content and screen size on the wet bulk density of loblolly pine grinds (2 month storage time).

Moisture Content (%, w.b.)	Screen Size (mm)	Wet Bulk Density (kg/m^3)		
		Values	Mean	Standard Deviation
12	3.175	254.18		
12	3.175	252.60	253.78	1.05
12	3.175	254.57		
12	6.350	242.17		
12	6.350	240.28	241.09	0.98
12	6.350	240.81		
20	3.175	247.42		
20	3.175	245.91	246.84	0.81
20	3.175	247.19		
20	6.350	183.64		
20	6.350	187.16	185.93	1.98
20	6.350	186.98		
30	3.175	168.65		
30	3.175	167.37	167.91	0.66
30	3.175	167.71		
30	6.350	158.05		
30	6.350	160.77	159.07	1.48
30	6.350	158.40		
40	3.175	127.62		
40	3.175	127.79	128.55	1.48
40	3.175	130.26		
40	6.350	145.56		
40	6.350	146.53	146.64	1.15
40	6.350	147.85		
50	3.175	110.07		
50	3.175	109.06	110.30	1.38
50	3.175	111.79		
50	6.350	168.95		
50	6.350	167.94	169.55	1.98
50	6.350	171.77		

Table A.3–6. Effects of moisture content and screen size on the wet bulk density of loblolly pine grinds (4 month storage time).

Moisture Content (%, w.b.)	Screen Size (mm)	Wet Bulk Density (kg/m^3)		
		Values	Mean	Standard Deviation
12	3.175	263.29		
12	3.175	269.66	265.88	3.34
12	3.175	264.71		
12	6.350	246.83		
12	6.350	239.68	240.30	6.24
12	6.350	234.39		
20	3.175	235.86		
20	3.175	236.90	237.92	2.73
20	3.175	241.01		
20	6.350	186.68		
20	6.350	191.18	189.68	2.60
20	6.350	191.19		
30	3.175	158.56		
30	3.175	153.60	157.39	3.37
30	3.175	160.02		
30	6.350	154.64		
30	6.350	165.66	160.63	5.57
30	6.350	161.60		
40	3.175	134.30		
40	3.175	137.02	135.67	1.36
40	3.175	135.70		
40	6.350	163.27		
40	6.350	167.53	165.51	2.14
40	6.350	165.75		
50	3.175	119.57		
50	3.175	126.86	122.18	4.06
50	3.175	120.11		
50	6.350	183.15		
50	6.350	183.51	183.45	0.27
50	6.350	183.68		

Table A.4–1. Effects of moisture content and screen size on the geometric mean diameter of oven-dried loblolly pine grinds (0 month storage time).

Moisture Content (%, w.b.)	Screen Size (mm)	Geometric Mean Diameter (mm)		
		Values	Mean	Standard Deviation
12	3.175	0.84		
12	3.175	0.83	0.83	0.01
12	3.175	0.82		
12	6.350	1.68		
12	6.350	1.66	1.66	0.02
12	6.350	1.64		
20	3.175	1.33		
20	3.175	1.33	1.33	0.01
20	3.175	1.32		
20	6.350	2.06		
20	6.350	2.05	2.04	0.03
20	6.350	2.01		
30	3.175	0.79		
30	3.175	0.78	0.78	0.01
30	3.175	0.77		
30	6.350	1.99		
30	6.350	1.96	1.96	0.03
30	6.350	1.93		
40	3.175	0.86		
40	3.175	0.84	0.84	0.01
40	3.175	0.83		
40	6.350	1.62		
40	6.350	1.58	1.58	0.04
40	6.350	1.54		
50	3.175	0.83		
50	3.175	0.81	0.81	0.02
50	3.175	0.79		
50	6.350	1.93		
50	6.350	1.89	1.88	0.05
50	6.350	1.83		

Table A.4–2. Effects of moisture content and screen size on the geometric mean diameter of oven-dried loblolly pine grinds (2 month storage time).

Moisture Content (%, w.b.)	Screen Size (mm)	Geometric Mean Diameter (mm)		
		Values	Mean	Standard Deviation
12	3.175	0.71		
12	3.175	0.71	0.71	0.00
12	3.175	0.71		
12	6.350	1.34		
12	6.350	1.32	1.32	0.02
12	6.350	1.31		
20	3.175	0.94		
20	3.175	0.92	0.89	0.08
20	3.175	0.80		
20	6.350	1.58		
20	6.350	1.57	1.54	0.07
20	6.350	1.46		
30	3.175	0.72		
30	3.175	0.71	0.71	0.01
30	3.175	0.69		
30	6.350	1.61		
30	6.350	1.61	1.58	0.05
30	6.350	1.53		
40	3.175	0.69		
40	3.175	0.68	0.68	0.01
40	3.175	0.68		
40	6.350	1.51		
40	6.350	1.49	1.49	0.01
40	6.350	1.48		
50	3.175	0.62		
50	3.175	0.60	0.60	0.02
50	3.175	0.58		
50	6.350	1.51		
50	6.350	1.50	1.46	0.08
50	6.350	1.37		

Table A.4–3. Effects of moisture content and screen size on the geometric mean diameter of oven-dried loblolly pine grinds (4 month storage time).

Moisture Content (%, w.b.)	Screen Size (mm)	Geometric Mean Diameter (mm)		
		Values	Mean	Standard Deviation
12	3.175	0.79		
12	3.175	0.76	0.77	0.02
12	3.175	0.75		
12	6.350	1.50		
12	6.350	1.42	1.44	0.05
12	6.350	1.40		
20	3.175	0.87		
20	3.175	0.85	0.85	0.02
20	3.175	0.83		
20	6.350	1.72		
20	6.350	1.71	1.63	0.14
20	6.350	1.47		
30	3.175	0.86		
30	3.175	0.84	0.84	0.02
30	3.175	0.82		
30	6.350	1.79		
30	6.350	1.75	1.75	0.04
30	6.350	1.70		
40	3.175	0.80		
40	3.175	0.79	0.77	0.04
40	3.175	0.72		
40	6.350	1.76		
40	6.350	1.69	1.71	0.04
40	6.350	1.69		
50	3.175	0.66		
50	3.175	0.65	0.65	0.02
50	3.175	0.63		
50	6.350	1.79		
50	6.350	1.68	1.70	0.09
50	6.350	1.63		

Table A.5–1. Effects of moisture content and screen size on the geometric standard deviation of oven-dried loblolly pine grinds (0 month storage time)

Moisture Content (%, w.b.)	Screen Size (mm)	Geometric Standard Deviation (mm)		
		Values	Mean	Standard Deviation
12	3.175	0.70		
12	3.175	0.70	0.71	0.01
12	3.175	0.72		
12	6.350	1.17		
12	6.350	1.18	1.18	0.02
12	6.350	1.20		
20	3.175	1.33		
20	3.175	1.32	1.32	0.01
20	3.175	1.31		
20	6.350	1.46		
20	6.350	1.42	1.45	0.02
20	6.350	1.47		
30	3.175	0.74		
30	3.175	0.75	0.75	0.01
30	3.175	0.77		
30	6.350	1.43		
30	6.350	1.42	1.43	0.01
30	6.350	1.43		
40	3.175	0.79		
40	3.175	0.80	0.81	0.02
40	3.175	0.82		
40	6.350	1.41		
40	6.350	1.40	1.40	0.01
40	6.350	1.40		
50	3.175	0.67		
50	3.175	0.69	0.69	0.02
50	3.175	0.70		
50	6.350	1.28		
50	6.350	1.36	1.35	0.07
50	6.350	1.41		

Table A.5–2. Effects of moisture content and screen size on the geometric standard deviation of oven-dried loblolly pine grinds (2 month storage time)

Moisture Content (%, w.b.)	Screen Size (mm)	Geometric Standard Deviation (mm)		
		Values	Mean	Standard Deviation
12	3.175	0.54		
12	3.175	0.54	0.54	0.01
12	3.175	0.55		
12	6.350	0.98		
12	6.350	0.99	0.99	0.01
12	6.350	1.00		
20	3.175	0.86		
20	3.175	0.85	0.81	0.06
20	3.175	0.74		
20	6.350	1.21		
20	6.350	1.25	1.24	0.02
20	6.350	1.25		
30	3.175	0.70		
30	3.175	0.69	0.69	0.01
30	3.175	0.68		
30	6.350	1.34		
30	6.350	1.37	1.35	0.02
30	6.350	1.36		
40	3.175	0.63		
40	3.175	0.64	0.64	0.00
40	3.175	0.64		
40	6.350	1.34		
40	6.350	1.35	1.35	0.01
40	6.350	1.36		
50	3.175	0.62		
50	3.175	0.59	0.59	0.02
50	3.175	0.57		
50	6.350	1.23		
50	6.350	1.15	1.17	0.05
50	6.350	1.15		

Table A.5–3. Effects of moisture content and screen size on the geometric standard deviation of oven-dried loblolly pine grinds (4 month storage time)

Moisture Content (%, w.b.)	Screen Size (mm)	Geometric Standard Deviation (mm)		
		Values	Mean	Standard Deviation
12	3.175	0.62		
12	3.175	0.64	0.64	0.03
12	3.175	0.67		
12	6.350	1.02		
12	6.350	1.08	1.07	0.05
12	6.350	1.12		
20	3.175	0.77		
20	3.175	0.76	0.78	0.02
20	3.175	0.80		
20	6.350	1.38		
20	6.350	1.39	1.38	0.01
20	6.350	1.38		
30	3.175	0.84		
30	3.175	0.85	0.85	0.02
30	3.175	0.87		
30	6.350	1.48		
30	6.350	1.45	1.49	0.04
30	6.350	1.53		
40	3.175	0.88		
40	3.175	0.85	0.85	0.04
40	3.175	0.81		
40	6.350	1.40		
40	6.350	1.46	1.42	0.03
40	6.350	1.41		
50	3.175	0.73		
50	3.175	0.73	0.73	0.01
50	3.175	0.71		
50	6.350	1.31		
50	6.350	1.38	1.33	0.04
50	6.350	1.30		

Table A.6–1. Effects of moisture content and screen size on the coefficient of variation of oven-dried loblolly pine grinds (0 month storage time).

Moisture Content (%, w.b.)	Screen Size (mm)	Coefficient of Variation (%)		
		Values	Mean	Standard Deviation
12	3.175	83.26		
12	3.175	84.96	85.32	2.26
12	3.175	87.74		
12	6.350	69.48		
12	6.350	70.92	71.31	2.06
12	6.350	73.54		
20	3.175	99.95		
20	3.175	99.14	99.61	0.42
20	3.175	99.73		
20	6.350	70.92		
20	6.350	69.39	71.06	1.75
20	6.350	72.88		
30	3.175	94.08		
30	3.175	97.05	96.95	2.81
30	3.175	99.71		
30	6.350	72.25		
30	6.350	72.60	72.92	0.89
30	6.350	73.93		
40	3.175	92.41		
40	3.175	95.74	95.76	3.36
40	3.175	99.12		
40	6.350	86.96		
40	6.350	88.65	88.75	1.85
40	6.350	90.65		
50	3.175	80.88		
50	3.175	85.38	85.28	4.35
50	3.175	89.58		
50	6.350	66.31		
50	6.350	71.88	71.82	5.48
50	6.350	77.27		

Table A.6–2. Effects of moisture content and screen size on the coefficient of variation of oven-dried loblolly pine grinds (2 month storage time).

Moisture Content (%, w.b.)	Screen Size (mm)	Coefficient of Variation (%)		
		Values	Mean	Standard Deviation
12	3.175	75.35		
12	3.175	75.90	76.32	1.24
12	3.175	77.72		
12	6.350	72.87		
12	6.350	74.65	74.79	2.00
12	6.350	76.87		
20	3.175	90.84		
20	3.175	91.71	91.96	1.26
20	3.175	93.33		
20	6.350	76.75		
20	6.350	79.71	80.80	4.68
20	6.350	85.92		
30	3.175	97.17		
30	3.175	97.78	97.48	0.30
30	3.175	97.50		
30	6.350	82.97		
30	6.350	85.15	85.65	2.97
30	6.350	88.84		
40	3.175	91.64		
40	3.175	94.46	93.48	1.60
40	3.175	94.35		
40	6.350	89.30		
40	6.350	90.27	90.42	1.21
40	6.350	91.70		
50	3.175	99.42		
50	3.175	98.45	99.05	0.53
50	3.175	99.30		
50	6.350	81.25		
50	6.350	76.42	80.44	3.69
50	6.350	83.66		

Table A.6–3. Effects of moisture content and screen size on the coefficient of variation of oven-dried loblolly pine grinds (4 month storage time).

Moisture Content (%, w.b.)	Screen Size (mm)	Coefficient of Variation (%)		
		Values	Mean	Standard Deviation
12	3.175	78.38		
12	3.175	83.65	83.79	5.48
12	3.175	89.33		
12	6.350	67.85		
12	6.350	76.26	74.84	6.40
12	6.350	80.41		
20	3.175	88.79		
20	3.175	88.97	91.42	4.40
20	3.175	96.50		
20	6.350	80.63		
20	6.350	81.01	85.05	7.33
20	6.350	93.51		
30	3.175	97.03		
30	3.175	100.70	101.19	4.42
30	3.175	105.83		
30	6.350	82.54		
30	6.350	83.23	85.22	4.06
30	6.350	89.88		
40	3.175	110.84		
40	3.175	107.56	110.23	2.42
40	3.175	112.29		
40	6.350	79.40		
40	6.350	86.45	83.03	3.53
40	6.350	83.26		
50	3.175	110.80		
50	3.175	112.73	112.07	1.11
50	3.175	112.70		
50	6.350	73.33		
50	6.350	82.24	78.42	4.59
50	6.350	79.69		

Table A.7–1. Effects of moisture content and screen size on the specific grinding energy requirement of loblolly pine woodchips (0 month storage time).

Moisture Content (%, w.b.)	Screen Size (mm)	Grinding Energy Requirement (kJ/kg d.b.)		
		Values	Mean	Standard Deviation
12	3.175	197.41		
12	3.175	203.10	205.02	8.72
12	3.175	214.54		
12	6.350	60.29		
12	6.350	65.69	68.97	10.70
12	6.350	80.93		
20	3.175	260.33		
20	3.175	262.78	268.27	11.69
20	3.175	281.70		
20	6.350	179.74		
20	6.350	181.84	181.96	2.28
20	6.350	184.30		
30	3.175	550.49		
30	3.175	577.49	569.14	16.19
30	3.175	579.44		
30	6.350	174.16		
30	6.350	174.93	176.52	3.45
30	6.350	180.49		
40	3.175	655.23		
40	3.175	698.92	689.74	30.97
40	3.175	715.08		
40	6.350	134.17		
40	6.350	142.72	147.08	15.56
40	6.350	164.36		
50	3.175	647.36		
50	3.175	664.61	662.99	14.89
50	3.175	677.01		
50	6.350	135.53		
50	6.350	137.67	139.66	5.41
50	6.350	145.79		

Table A.7–2. Effects of moisture content and screen size on the specific grinding energy requirement of loblolly pine woodchips (2 month storage time).

Moisture Content (%, w.b.)	Screen Size (mm)	Grinding Energy Requirement (kJ/kg d.b.)		
		Values	Mean	Standard Deviation
12	3.175	182.92		
12	3.175	188.11	187.79	4.71
12	3.175	192.33		
12	6.350	54.34		
12	6.350	59.45	59.91	5.81
12	6.350	65.94		
20	3.175	298.54		
20	3.175	328.89	326.32	26.60
20	3.175	351.54		
20	6.350	142.60		
20	6.350	164.99	161.84	17.88
20	6.350	177.94		
30	3.175	527.36		
30	3.175	537.72	537.51	10.06
30	3.175	547.47		
30	6.350	180.89		
30	6.350	197.15	192.13	9.75
30	6.350	198.36		
40	3.175	688.81		
40	3.175	704.67	700.74	10.53
40	3.175	708.75		
40	6.350	156.64		
40	6.350	158.92	159.16	2.65
40	6.350	161.92		
50	3.175	707.44		
50	3.175	769.54	763.50	53.30
50	3.175	813.52		
50	6.350	134.44		
50	6.350	147.50	145.46	10.15
50	6.350	154.43		

Table A.7–3. Effects of moisture content and screen size on the specific grinding energy requirement of loblolly pine woodchips (4 month storage time).

Moisture Content (%, w.b.)	Screen Size (mm)	Grinding Energy Requirement (kJ/kg d.b.)		
		Values	Mean	Standard Deviation
12	3.175	182.66		
12	3.175	189.33	189.01	6.19
12	3.175	195.04		
12	6.350	60.91		
12	6.350	69.27	68.71	7.53
12	6.350	75.94		
20	3.175	354.23		
20	3.175	376.09	369.44	13.20
20	3.175	377.99		
20	6.350	137.17		
20	6.350	144.10	142.99	5.35
20	6.350	147.70		
30	3.175	523.55		
30	3.175	549.99	542.10	16.13
30	3.175	552.77		
30	6.350	171.94		
30	6.350	179.14	178.70	6.55
30	6.350	185.01		
40	3.175	654.73		
40	3.175	685.68	685.16	30.18
40	3.175	715.07		
40	6.350	113.18		
40	6.350	119.84	122.50	10.90
40	6.350	134.48		
50	3.175	766.81		
50	3.175	782.74	789.46	26.65
50	3.175	818.82		
50	6.350	115.13		
50	6.350	129.65	126.12	9.72
50	6.350	133.59		

Appendix B–SAS Code and Results

```
*###CHAPTER THREE###;

*importing csv file containing the data on section 3.4.1;
proc import datafile="C:\Users\oao0003\Google Drive\MSc Research\Data and
Analysis\Analysis\Objective 1\Chemical Properties.csv"
     out=chemical_properties
     dbms=csv
     replace;
     getnames=yes;
run;
*effect of storage time on chemical properties;
proc reg data=chemical_properties;
      model volatile_matter_content = storage_time;
run;
proc reg data=chemical_properties;
      model ash_content = storage_time;
run;
proc reg data=chemical_properties;
      model HHV = storage_time;
run;
proc anova data=chemical_properties;
      class storage_time;
      model volatile_matter_content = storage_time;
      means storage_time/tukey;
run;
proc anova data=chemical_properties;
      class storage_time;
      model ash_content = storage_time;
      means storage_time/tukey;
run;
proc anova data=chemical_properties;
      class storage_time;
      model HHV = storage_time;
      means storage_time/tukey;
run;

*importing csv file containing the data on section 3.4.2;
proc import datafile="C:\Users\oao0003\Google Drive\MSc Research\Data and
Analysis\Analysis\Objective 1\Grinding Properties.csv"
     out=grinding_properties_i
     dbms=csv
     replace;
     getnames=yes;
run;

*adding some computed columns (diameter_constant, predicted grinding energy);
data grinding_properties_ii;
      set grinding_properties_i;
      kick_diameter = log((11.62/particle_size_dwg_dry));
      rittinger_diameter = ((1/(particle_size_dwg_dry)) - (1/(11.62)));
      bond_diameter = ((1/((particle_size_dwg_dry**0.5))) -
      (1/((11.62**0.5))));

      centered_SS = screen_size - 4.7625;
```

```
      centered_MC = moisture_content - 30.4;
      centered_MC_ii = centered_MC**2;
      MC_SS = centered_MC * centered_SS;
run;
proc print data=grinding_properties_ii;
run;

*ANOVA for effect of moisture content, storage time, and hammer mill screen
size on moisture loss during grinding, bulk density, particle size
distribution and specific drying energy;
proc mixed data=grinding_properties_i;
      class storage_time moisture_content screen_size rep;
      model moisture_loss = storage_time moisture_content|screen_size;
      random rep(storage_time);
      lsmeans storage_time moisture_content|screen_size/cl alpha = 0.05;
run;
proc mixed data=grinding_properties_i;
      class storage_time moisture_content screen_size rep;
      model bulk_density_dry = storage_time moisture_content|screen_size;
      random rep(storage_time);
      lsmeans storage_time moisture_content|screen_size/cl alpha = 0.05;
run;
proc mixed data=grinding_properties_i;
      class storage_time moisture_content screen_size rep;
      model dwg_dry = storage_time moisture_content|screen_size;
      random rep(storage_time);
      lsmeans storage_time moisture_content|screen_size/cl alpha = 0.05;
run;
proc mixed data=grinding_properties_i;
      class storage_time moisture_content screen_size rep;
      model swg_dry = storage_time moisture_content|screen_size;
      random rep(storage_time);
      lsmeans storage_time moisture_content|screen_size/cl alpha = 0.05;
run;
proc mixed data=grinding_properties_i;
      class storage_time moisture_content screen_size rep;
      model CV_dry = storage_time moisture_content|screen_size;
      random rep(storage_time);
      lsmeans storage_time moisture_content|screen_size/cl alpha = 0.05;
run;
proc mixed data=grinding_properties_i;
      class storage_time moisture_content screen_size rep;
      model grinding_energy_db = storage_time moisture_content|screen_size;
      random rep(storage_time);
      lsmeans storage_time moisture_content|screen_size/cl alpha = 0.05;
run;

*Comparing wet and oven-dried bulk densities;
proc sort data=grinding_properties_i;
      by storage_time screen_size moisture_content;
run;
proc ttest data=grinding_properties_i;
      paired bulk_density_dry*bulk_density_wet;
      by storage_time screen_size moisture_content;
run;

*fitting a non-linear model for grinding energy using the Kick equation;
```

```
ODS GRAPHICS ON;
proc nlin data=grinding_properties_ii plots=diagnostics(stats=all)alpha=0.05;
      parms alpha=0.45 gamma=0.25 omega=0.08 phi=1.00 rho=0.05;
      model grinding_energy_db = alpha + (gamma*centered_MC + omega*MC_SS
            + phi*centered_MC_ii + rho*centered_SS) * kick_diameter;
run; ODS GRAPHICS OFF;

*fitting a non-linear model for grinding energy using the Rittinger equation;
ODS GRAPHICS ON;
proc nlin data=grinding_properties_ii plots=diagnostics(stats=all)alpha=0.05;
      parms alpha=0.45 gamma=0.25 omega=0.08 phi=1.00 rho=0.05;
      model grinding_energy_db = alpha + (gamma*centered_MC + omega*MC_SS
            + phi*centered_MC_ii + rho*centered_SS) * rittinger_diameter;
run; ODS GRAPHICS OFF;

*fitting a non-linear model for grinding energy using the Bond equation;
ODS GRAPHICS ON;
proc nlin data=grinding_properties_ii plots=diagnostics(stats=all)alpha=0.05;
      parms alpha=0.45 gamma=0.25 omega=0.08 phi=1.00 rho=0.05;
      model grinding_energy_db = alpha + (gamma*centered_MC + omega*MC_SS
            + phi*centered_MC_ii + rho*centered_SS) * bond_diameter;
run; ODS GRAPHICS OFF;

*preparing data for observations and predictions on grinding energy using
estimates from the developed non-linear models;
data grinding_properties_iii;
      set grinding_properties_ii;
      kick_constant = (330.1 + (3.0591*centered_MC - 1.5455*MC_SS
- 0.0993*centered_MC_ii - 50.1765*centered_SS)* kick_diameter)/kick_diameter;
      rittinger_constant = (285.8 + (6.5606*centered_MC - 3.0018*MC_SS
- 0.2940*centered_MC_ii - 140.4*centered_SS) *
rittinger_diameter)/rittinger_diameter;
      bond_constant = (309.7 + (9.6389*centered_MC - 4.7002*MC_SS
                  - 0.3589*centered_MC_ii - 178.9*centered_SS)
                  * bond_diameter)/bond_diameter;

      Kick_predicted = 330.1 + (3.0591*centered_MC - 1.5455*MC_SS
- 0.0993*centered_MC_ii - 50.1765*centered_SS) * Kick_diameter;
      rittinger_predicted = 285.8 + (6.5606*centered_MC - 3.0018*MC_SS
- 0.2940*centered_MC_ii - 140.4*centered_SS) * rittinger_diameter;
      bond_predicted = 309.7 + (9.6389*centered_MC - 4.7002*MC_SS
- 0.3589*centered_MC_ii - 178.9*centered_SS) * bond_diameter;
run;

proc print data=grinding_properties_iii;
run;

*ploting 3D graphs for Kick, Rittinger, and Bond constant versus moisture
content and hammer mill screen size;
proc g3d data=grinding_properties_iii;
      plot moisture_content*screen_size=kick_constant/yticknum=5 xticknum=3
zticknum=4 zmin=0 zmax=300;
      plot moisture_content*screen_size=rittinger_constant/yticknum=5
xticknum=3 zticknum=4 zmin=0 zmax=600;
      plot moisture_content*screen_size=bond_constant/yticknum=5 xticknum=3
zticknum=5 zmin=0 zmax=1000;
run;
```

```
quit;

*comparing observed and predicted values using regression model;
ODS GRAPHICS ON;
proc reg data=grinding_properties_iii;
      model grinding_energy_db = kick_predicted/noint;
      model grinding_energy_db = rittinger_predicted/noint;
      model grinding_energy_db = bond_predicted/noint;
run; ODS GRAPHICS OFF;

*###CHAPTER FOUR###;
*importing csv file containing the data on specific toughness and bending
stress;
proc import datafile="C:\Users\Oluwafemi Oyedeji\Google Drive\MSc Research\Data
and Analysis\Analysis\Objective 2\Data Bending_test_toughness.csv"
     out=bending_test
     dbms=csv
     replace;
     getnames=yes;
run;
*Adding some column for variables and calculating toughness;
data bending_test_toughness;
      set bending_test;
      moisture_content_2 = moisture_content**2;
      radius = radius*100;
      radius_2 = radius**2;
      height_2 = height**2;
      height_radius = height*radius;
      toughness = ((toughness*1000)/(5*10)); *specific toughness;
run;
proc print data=bending_test_toughness; run;
*Toughness data summary;
proc tabulate data=bending_test_toughness;
      class moisture_content tree radius height;
      var toughness;
      table moisture_content*height*radius, toughness*(N Mean Min Max);
run;
*Analysis of variance for the effects of moisture, radius, and height on
toughness;
proc anova data=bending_test_toughness;
      class moisture_content radius height tree;
      model toughness_trans = tree moisture_content radius height;
      means tree moisture_content radius height /tukey;
run;
*Regression analysis for toughness data;
ODS GRAPHICS ON;
proc reg data=bending_test_toughness plots(label)=(RStudentByLeverage CooksD
DFFITS DFBETAS) corr;
      model toughness = centered_moisture_content centered_height
            centered_radius centered_moisture_content_2 centered_height_2
            centered_radius_2 centered_height_radius/ selection=stepwise
            slstay=0.05 slentry=0.05 vif stb;
run; ODS GRAPHICS OFF;
ods graphics on;

*principal component regression;
proc princomp data = bending_test_toughness out = bending_test_toughness_ii;
```

```
   var moisture_content height radius moisture_content_2 height_2 radius_2;
run;

proc reg data= bending_test_toughness_ii;
      model toughness = prin1 prin2 prin3 prin4 prin5 prin6/stb vif;
run;
proc reg data= bending_test_toughness_ii;
      model toughness = comp1 comp2 comp3 comp4 comp5 comp6/stb;
run;
*Adding some column for mean centralized variables and calculating bending stress;
data bending_test_stress;
      set bending_test;
      moisture_content_2 = moisture_content**2;
      radius = radius*100;
      radius_2 = radius**2;
      height_2 = height**2;
      height_radius = height*radius;
      stress = ((1.5*maxforce*9.81*0.0195/1000)/(0.01*0.005*0.005))/1000000;
run;
proc print data=bending_test_stress; run;
*Bending stress data summary;
proc tabulate data=bending_test_stress;
      class moisture_content tree radius height;
      var stress;
      table moisture_content*height*radius, stress*(N Mean Min Max);
run;
*Analysis of variance for the effects of moisture, radius, and height on bending stress;
proc anova data=bending_test_stress;
      class moisture_content radius height tree;
      model stress = tree moisture_content radius height;
      means tree moisture_content radius height /tukey;
run;
*Regression analysis for bending stress data;
ODS GRAPHICS ON;
proc reg data=bending_test_stress plots(label)=(RStudentByLeverage CooksD DFFITS DFBETAS) corr;
      model stress = centered_moisture_content centered_radius
            centered_moisture_content_2 centered_height
            centered_radius_2 centered_height_2/ selection=stepwise
            slstay=0.05 slentry=0.05 stb vif;
run; ODS GRAPHICS OFF;

*principal component regression;
proc princomp data = bending_test_stress out = bending_test_stress_ii;
   var moisture_content height radius moisture_content_2 height_2 radius_2;
run;

proc reg data= bending_test_stress_ii;
      model toughness = prin1 prin2 prin3 prin4 prin5 prin6/stb vif;
run;
*###CHAPTER FIVE###;
proc import datafile="C:\Users\oao0003\Google Drive\MSc Research\Data and Analysis\Analysis\Objective 3\Data.csv"
      out=sequence
```

```
    dbms=csv
    replace;
    getnames=yes; run;
*Bulk density analysis;
ODS GRAPHICS ON;
proc anova data=sequence;
      class sequence;
      model DryBulk = sequence;
      means sequence/tukey;
run; ODS GRAPHICS OFF;
*Aspect ratio analysis;
ODS GRAPHICS ON;
proc anova data=sequence;
      class SequenceAspect ParticleSizeAspect;
      model AspectRatio = SequenceAspect ParticleSizeAspect;
      means SequenceAspect/tukey; run; ODS GRAPHICS OFF;
proc anova data=sequence;
      class Sequenceii;
      model cumulative = Sequenceii;
      means Sequenceii/tukey; run;
*adding some computed columns (diameter_constant, predicted grinding energy);
data sequence_i;
      set sequence;
      K_rittinger = energy/((1/dwgdry) - (1/(11.62)));
      K_bond = energy/((1/dwgdry**0.5) - (1/((11.62)**0.5)));
      K_kick = energy/(log(11.62/dwgdry)); run;
proc anova data=sequence_i;
      class Sequence;
      model K_rittinger = Sequence;
      means Sequence/tukey; run;
proc anova data=sequence_i;
      class Sequence;
      model K_bond = Sequence;
      means Sequence/tukey; run;
proc anova data=sequence_i;
      class Sequence;
      model K_kick = Sequence;
      means Sequence/tukey; run;
proc anova data=sequence_i;
      class Sequence;
      model energy = Sequence;
      means Sequence/tukey; run;
proc anova data=sequence_i;
      class Sequence;
      model mloss = Sequence;
      means Sequence/tukey; run;
```

Table B.1. ANOVA result for effect of storage time on the chemical properties of woodchips.

Volatile Matter Content

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	1	3.06908	3.06908	21.75	0.0009
Error	10	1.41094	0.14109		
Corrected Total	11	4.48003			

Root MSE	0.37563	**R-Square**	0.6851
Dependent Mean	85.9925	**Adj R-Sq**	0.6536
Coeff Var	0.43681		

Variable	DF	Parameter Estimate	Standard Error	t Value	Pr > |t|
Intercept	1	86.671	0.18144	477.67	<.0001
Storage Time	1	-0.22617	0.04849	-4.66	0.0009

Ash Content

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	1	0.03456	0.03456	19.74	0.0012
Error	10	0.01751	0.00175		
Corrected Total	11	0.05207			

Root MSE	0.04184	**R-Square**	0.6638
Dependent Mean	0.39333	**Adj R-Sq**	0.6301
Coeff Var	10.63753		

Variable		Parameter Estimate	Standard Error	t Value	Pr > |t|
Intercept	1	0.32133	0.02021	15.9	<.0001
Storage Time	1	0.024	0.0054	4.44	0.0012

Energy Content

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	1	0.07676	0.07676	20.1	0.0012
Error	10	0.03819	0.00382		
Corrected Total	11	0.11495			

Root MSE	0.0618	**R-Square**	0.6677
Dependent Mean	20.22567	**Adj R-Sq**	0.6345
Coeff Var	0.30556		

Variable		Parameter Estimate	Standard Error	t Value	Pr > |t|
Intercept	1	20.33297	0.02985	681.11	<.0001
Storage Time	1	-0.03577	0.00798	-4.48	0.0012

Table B.2-1. ANOVA result for moisture loss during grinding.

Type 3 Tests of Fixed Effects				
Effect	**Num DF**	**Den DF**	**F Value**	**Pr > F**
Storage Time	2	6	13.4	0.0061
Moisture Content	4	72	459.82	<.0001
Screen Size	1	72	436.07	<.0001
Moisture Content*Screen Size	4	72	38.5	<.0001

Table B.2-1. LS-means for moisture loss during grinding (confidence interval, CL = 95%)

Effect	Storage Time	Moisture Content	Screen Size	Estimate	CL	
					Lower	Upper
Storage Time	0			12.011	11.254	12.769
	2			13.564	12.807	14.322
	4			14.217	13.460	14.975
Moisture Content		12		2.600	1.803	3.397
		20		7.918	7.121	8.715
		30		13.447	12.651	14.244
		40		17.557	16.761	18.354
		50		24.798	24.002	25.595
Screen Size			6.350	9.532	9.028	10.036
			3.175	16.997	16.493	17.501
Moisture Content * Screen Size		12	6.350	1.555	0.429	2.682
		12	3.175	3.645	2.518	4.772
		20	6.350	6.228	5.101	7.355
		20	3.175	9.608	8.481	10.735
		30	6.350	9.178	8.052	10.305
		30	3.175	17.716	16.590	18.843
		40	6.350	13.164	12.037	14.291
		40	3.175	21.951	20.824	23.078
		50	6.350	17.533	16.407	18.660
		50	3.175	32.063	30.937	33.190

Table B.3-1. ANOVA result for effects of moisture content, storage time, and screen size on bulk density of oven-dried grinds.

Type 3 Tests of Fixed Effects				
Effect	**Num DF**	**Den DF**	**F Value**	**Pr > F**
Storage Time	2	6	3.95	0.0806
Moisture Content	4	72	494.16	<.0001
Screen Size	1	72	6.44	0.0133
Moisture Content*Screen Size	4	72	23.41	<.0001

Table B.3-2. LS-means for effects of moisture content, storage time, and screen size on bulk density of oven-dried grinds (confidence interval, CL = 95%).

Effect	Storage Time	Moisture Content	Screen Size	Estimate	CL	
					Lower	Upper
Storage Time	0			181.27 a	175.87	186.68
	2			172.62 a	167.21	178.02
	4			178.17 a	172.77	183.57
Moisture Content		12		262.39 *1*	256.71	268.07
		20		224.25 *2*	218.57	229.94
		30		156.61 *3*	150.93	162.29
		40		124.79 *4*	119.11	130.48
		50		118.72 *4*	113.04	124.40
Screen Size			6.350	174.12 *β*	170.53	177.71
			3.175	180.59 *α*	177.00	184.18
Moisture Content * Screen Size		12	6.350	251.14 *b*	243.10	259.18
		12	3.175	273.64 *a*	265.61	281.68
		20	6.350	205.49 *c*	197.46	213.53
		20	3.175	243.01 *b*	234.98	251.05
		30	6.350	147.89 *e*	139.85	155.92
		30	3.175	165.34 *d*	157.30	173.37
		40	6.350	134.67 *e*	126.63	142.70
		40	3.175	114.92 *f*	106.88	122.96
		50	6.350	131.41 *e*	123.37	139.45
		50	3.175	106.03 *f*	97.99	114.07

** Estimates followed with the same letter (for storage time), italicized number (for moisture content), Greek letter (screen size), and italicized letter (for moisture content*screen size) are not significantly different at 0.05 level of significance.

Table B.4-1. ANOVA result for effects of moisture content, storage time, and screen size on geometric mean diameter of oven-dried grinds.

Type 3 Tests of Fixed Effects				
Effect	**Num DF**	**Den DF**	**F Value**	**Pr > F**
Storage Time	2	6	58.8	0.0001
Moisture Content	4	72	19.01	<.0001
Screen Size	1	72	1688.26	<.0001
Moisture Content * Screen Size	4	72	9.24	<.0001

Table B.4-2. LS-means for effects of moisture content, storage time, and screen size on geometric mean diameter of oven-dried grinds (confidence interval, CL = 95%).

Least Squares Means						
Effect	**Storage Time**	**Moisture Content**	**Screen Size**	**Estimate**	**CL**	
					Lower	**Upper**
Storage Time	0			1.3705	1.3269	1.4142
	2			1.0981	1.0544	1.1418
	4			1.2113	1.1676	1.2549
Moisture Content		12		1.1212	1.0753	1.1671
		20		1.3793	1.3334	1.4253
		30		1.2697	1.2238	1.3156
		40		1.1797	1.1337	1.2256
		50		1.1833	1.1373	1.2292
Screen Size			6.350	1.6501	1.6210	1.6791
			3.175	0.8032	0.7742	0.8323
Moisture Content * Screen Size		12	6.350	1.4742	1.4092	1.5392
		12	3.175	0.7682	0.7032	0.8332
		20	6.350	1.7378	1.6728	1.8028
		20	3.175	1.0209	0.9559	1.0859
		30	6.350	1.7630	1.6980	1.8279
		30	3.175	0.7764	0.7115	0.8414
		40	6.350	1.5945	1.5295	1.6595
		40	3.175	0.7648	0.6998	0.8298
		50	6.350	1.6809	1.6159	1.7458
		50	3.175	0.6857	0.6207	0.7507

Table B.5-1. ANOVA result for effects of moisture content, storage time, and screen size on geometric standard deviation of oven-dried grinds.

Type 3 Tests of Fixed Effects				
Effect	**Num DF**	**Den DF**	**F Value**	**Pr > F**
Storage Time	2	6	32.53	0.0006
Moisture Content	4	72	36.85	<.0001
Screen Size	1	72	964.59	<.0001
Moisture Content * Screen Size	4	72	9.26	<.0001

Table B.5-2. LS-means for effects of moisture content, storage time, and screen size on geometric standard deviation of oven-dried grinds (confidence interval, CL = 95%).

Least Squares Means						
Effect	**Storage Time**	**Moisture Content**	**Screen Size**	**Estimate**	**CL**	
					Lower	**Upper**
Storage Time	0			1.109	1.0716	1.1464
	2			0.9384	0.9010	0.9758
	4			1.0545	1.0171	1.0919
Moisture Content		12		0.8563	0.8170	0.8956
		20		1.1643	1.1250	1.2036
		30		1.0947	1.0553	1.1340
		40		1.0772	1.0379	1.1165
		50		0.9773	0.9380	1.0166
Screen Size			6.350	1.3079	1.2830	1.3327
			3.175	0.7601	0.7352	0.7849
Moisture Content * Screen Size		12	6.350	1.0826	1.0270	1.1382
		12	3.175	0.6299	0.5743	0.6855
		20	6.350	1.3579	1.3023	1.4135
		20	3.175	0.9707	0.9151	1.0263
		30	6.350	1.4237	1.3681	1.4793
		30	3.175	0.7656	0.7101	0.8212
		40	6.350	1.3903	1.3348	1.4459
		40	3.175	0.7641	0.7085	0.8197
		50	6.350	1.2847	1.2291	1.3403
		50	3.175	0.6699	0.6143	0.7255

Table B.6-1. ANOVA result for effects of moisture content, storage time, and screen size on coefficient of variation of oven-dried grinds.

Type 3 Tests of Fixed Effects				
Effect	**Num DF**	**Den DF**	**F Value**	**Pr > F**
Storage Time	2	6	9.29	0.0146
Moisture Content	4	72	17.48	<.0001
Screen Size	1	72	142.16	<.0001
Moisture Content * Screen Size	4	72	3.35	0.0142

Table B.6-2. LS-means for effects of moisture content, storage time, and screen size on coefficient of variation of oven-dried grinds (confidence interval, CL = 95%).

Least Squares Means						
Effect	**Storage Time**	**Moisture Content**	**Screen Size**	**Estimate**	**CL**	
					Lower	**Upper**
Storage Time	0			83.8779	81.2075	86.5482
	2			87.0415	84.3711	89.7119
	4			90.5263	87.8560	93.1967
Moisture Content		12		77.7299	74.9214	80.5385
		20		86.6493	83.8407	89.4579
		30		89.9016	87.0930	92.7102
		40		93.6132	90.8046	96.4218
		50		87.8489	85.0403	90.6574
Screen Size			6.350	79.6362	77.8599	81.4125
			3.175	94.6609	92.8846	96.4372
Moisture Content * Screen Size		12	6.350	73.6496	69.6777	77.6215
		12	3.175	81.8102	77.8383	85.7821
		20	6.350	78.9688	74.9969	82.9407
		20	3.175	94.3298	90.3579	98.3017
		30	6.350	81.2651	77.2932	85.2370
		30	3.175	98.5381	94.5662	102.5100
		40	6.350	87.4026	83.4307	91.3745
		40	3.175	99.8238	95.8519	103.8000
		50	6.350	76.8950	72.9231	80.8669
		50	3.175	98.8027	94.8308	102.7700

Table B.7-1. ANOVA result for effects of moisture content, storage time, and screen size on specific grinding energy requirement of loblolly pine woodchips.

Type 3 Tests of Fixed Effects				
Effect	**Num DF**	**Den DF**	**F Value**	**Pr > F**
Storage Time	2	6	1.61	0.2756
Moisture Content	4	72	363.31	<.0001
Screen Size	1	72	3494.15	<.0001
Moisture Content * Screen Size	4	72	252.44	<.0001

Table B.7-2. LS-means for effects of moisture content, storage time, and screen size on specific grinding energy requirement of loblolly pine woodchips (confidence interval, CL = 95%).

Least Squares Means						
Effect	**Storage Time**	**Moisture Content**	**Screen Size**	**Estimate**	**CL**	
					Lower	**Upper**
Storage Time	0			310.94	298.00	323.88
	2			323.44	310.50	336.38
	4			321.42	308.48	334.36
Moisture Content		12		129.90	116.29	143.51
		20		241.80	228.19	255.41
		30		366.02	352.41	379.63
		40		417.40	403.79	431.01
		50		437.87	424.26	451.48
Screen Size			6.35	138.12	129.51	146.72
			3.175	499.08	490.47	507.69
Moisture Content * Screen Size		12	6.350	65.86	46.62	85.11
		12	3.175	193.94	174.69	213.19
		20	6.350	162.27	143.02	181.51
		20	3.175	321.34	302.10	340.59
		30	6.350	182.45	163.20	201.70
		30	3.175	549.59	530.34	568.83
		40	6.350	142.92	123.67	162.16
		40	3.175	691.88	672.63	711.13
		50	6.350	137.08	117.84	156.33
		50	3.175	738.65	719.40	757.90