COMPACTON OF SWITCHGRASS FOR VALUE ADDED UTILIZATION

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COMPACTON OF SWITCHGRASS FOR VALUE ADDED UTILIZATION

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THESIS ABSTRACT

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There are increasing concerns about energy prices, availability and utilization in the world. This has led to many governmental and privately sponsored studies and research on the potential of renewable energy from biomass. Switchgrass (*Panicum virgatum* L.), a potential energy crop, has been evaluated and is being developed as an alternative to fossil fuels. Most lightly dense biomass such as switchgrass are easier to store, transport, and handle after they are densified/pelleted. The optimal pelletization method of switchgrass was obtained by carrying out fundamental studies on the effect of process parameters (moisture content, temperature, and die size) on pelletability, density and specific energy. Pellets were manufactured in a single die apparatus attached to a texture analyzer. Results showed that with a compaction force of 3924 N, the density of the switchgrass pellets increased with decreasing die size (4.8 mm to 7.9 mm). Density also increased with temperature from 60 to 90°C. The density of the compacts was also affected by the moisture content of the feed material. The density varied from 850 kg/m^3 to 1250 kg/m^3. There was no significant effect of temperature on the specific energy used to make switchgrass pellets. Switchgrass used for investigation of moisture effect on physical properties was pelleted through a 4.8 mm diameter die. It was found that the bulk density, particle density, durability and hardness of the pellets were significantly affected by moisture content. The maximum values of bulk density and particle density were 708 kg/m^3 and 1462 kg/m^3 respectively. The force required to rupture the pellets varied from 32N at 6.32% to 22N at 17.4% moisture content. Durability of the pellets was also affected by moisture content and was the highest at 8.62% moisture content. The pellets absorbed moisture at rates that were significantly affected by relative humidity of the surrounding air ($P<0.05$). The equilibrium moisture content and equlibrium relative humidity (EMC-ERH) relationships for the pellets were sigmoidal in shape and were best predicted by the Chung-Pfost equation. Results from compositional analyses showed significant differences between lignin and ash for ground and pelleted switchgrass.

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INTRODUCTION

There is a growing need in the United States to decrease dependence on fossil fuel. The dependence on fossil fuel compromises the nation's security in terms of vulnerability to supply disruptions and to price volatilities. In addition, fossil fuels are non-renewable and there are environmental problems associated with the extraction, transportation and utilization of fossil fuels. Energy from biomass is an alternative energy source that can replace some the energy obtained from fossil fuels.

One crop that has been identified to have potential as a bioenergy crop is switchgrass – a high yielding perennial grass that has excellent conservation attributes with a relatively high energy value - about 8000 BTU/lb (McLaughlin et al., 1999; McLaughlin and Kszos, 2005). Similar to other perennial grasses, switchgrass has a low bulk density and therefore cannot be economically transported from place of production to where it can be effectively utilized. Densification, mostly by pelleting, has been used to increase the bulk density of grasses such as alfalfa. Pelleting decreases transportation and storage costs and makes the biomass easier to handle. Characterization of the mechanical properties of these pellets is required for design and selection of handling, processing and storage equipment and facilities.

Switchgrass is hygroscopic by nature. It will therefore exchange moisture with the surrounding environment. Knowledge of the moisture exchange rate for pellets under various conditions (temperature and relative humidity) is important for product integrity.

The quality of pellets obtained from a pellet mill is affected by parameters such as moisture content, temperature, particle size, die size and pellet mill speed. Therefore, a study of the effect of these parameters on pellet quality during pelleting (at pilot scale or industrial level) is needed to provide data for the design of biomass handling and processing facilities. Compaction studies are a good way to investigate the effect of these parameters on the densification of switchgrass and can be carried out by means of a single-pellet apparatus system.

Therefore, the goal of this project was to study the compaction behavior of switchgrass. This goal was achieved by the following specific objectives:

1. Quantify the effect of moisture content on the physical properties of compacted (pelleted) switchgrass;
2. Evaluate the effect of process parameters on the compaction behavior (density and specific energy) of switchgrass and,
3. Evaluate the effect of pelleting on the composition of switchgrass.

CHAPTER 1 –LITERATURE REVIEW

BIOENERGY

Increasing concerns about rising oil prices are creating interest in the development of economical and convenient renewable energy fuels such as biomass (Samson et al., 2000). The utilization of energy crops as a source of renewable fuels is a concept with enormous relevance to the current ecological and economic issues at both national and global scales (Vinterback, 2004). Recent advances in biomass feedstock development and conversion technologies have created new opportunities for using agricultural land as a means of producing these renewable fuels in larger quantities instead of relying on wood and agricultural residues alone (Samson et al., 2000). Renewable energy from biomass (i.e. bioenergy) has the potential to reduce dependency on fossil fuels thereby reducing the emission of greenhouse gases to the environment (Jannasch et al., 2001c; Mani et al., 2004a; Parikka, 2004). In addition, if bioenergy crops are grown in ecologically appropriate locations, their incorporation into agricultural systems could possibly provide extensive grassland bird habitat and address soil and water quality concerns (Roth et al., 2005).

Bioenergy in the form of heat, electricity, and liquid fuels represents 14% of the world's primary energy supply. About 25% of bioenergy usage occurs in industrialized countries with the remaining 75% used in developing countries. A comparison between the available potential with current use indicates that on a worldwide scale two-fifths of the existing biomass potential is used which leads to the conclusion that current biomass use is below the available potential in most of the world (Parikka, 2004). In the United States, bioenergy production is about 6%. A recent report by the U. S. Department of Energy indicates the country has the potential of generating over a billion tons of biomass annually for bioenergy use. The energy from this amount of biomass is sufficient to replace more than 30 % of the current petroleum consumption in the country (Perlack et al., 2005).

Biomass stores energy during the process of photosynthesis. This energy can be recovered by the combustion process or by conversion into usable forms such as ethanol, bio-oils, producer gases, and pellets/briquettes (Mani et al., 2003a; Mani et al., 2004a). The net energy available from biomass ranges from 20 MJ/kg for dry plant matter to 55MJ/kg for methane, compared with about 27 MJ/kg for coal (Mani et al., 2003a). Biomass co-firing with coal is another way of utilizing renewable technology. Co-firing entails combusting in an existing coal-fired unit, a combination of biomass and coal. The use of existing facilities reduces the capital investment and subsequently, the potential cost of the resulting renewable energy. Feedstock quality has a significant impact on determining the optimal conversion processes. One requirement of large-scale biofuel production is the ability to store biomass feedstock for 6-12 months to ensure continuous availability during off-season and winter months. For herbaceous feedstocks, unprotected outside storage can result in losses that have a negative impact on the economics of the conversion process. Losses include decrease in mass and changes in composition of structural and non-structural components and arise from weathering and biochemical reactions (Wiselogel et al., 1996). In addition to storage losses there are unavoidable losses of dry matter during field operations (e.g. cutting baling, transport) and during field curing of the plant material (Sanderson et al., 1997). Effective utilization of lignocellulosic feedstock can be deemed impractical because of its seasonal availability, scattered stations and the high costs of transportation of large amounts of organic matter (Szczodrak and Fiedurek, 1996).

SWITCHGRASS

Switchgrass (*Panicum virgatum L.*) is viewed as a major future energy crop in the United States (McLaughlin et al., 1999; Mani et al., 2004a), Canada (Samson et al., 2000; Christian et al., 2002) and Europe (Sharma et al., 2003). In the1980's the Bioenergy Feedstock Development Program (BFDP) at Oak Ridge National Laboratory (ORNL) started screening more than 30 herbaceous crop species with the goal of developing a renewable energy source that can be used to produce transportation fuel and to generate electricity (Sanderson et al., 1996).

In 1991, a decision was made by the ORNL to focus future work on switchgrass. This was because switchgrass is a high yielding, tall grass prairie species with relatively modest ash levels that has excellent conservation attributes and good compatibility with conventional farming practices (McLaughlin et al., 1999; Samson et al., 2000). Switchgrass also has a high fiber content and high biomass yield (Roth et al., 2005) and has good quality for heat and electricity production through thermal conversion (Sharma et al., 2003). Switchgrass tolerates diverse growing conditions, ranging from arid sites in short grass prairie to brackish marshes and open woods (Sanderson et al., 1996; McLaughlin et al., 1999) and requires little fertilization and herbicide (Boylan et al., 2000). The ecological diversity of switchgrass can be credited to three principle characteristics which include its open pollinated reproductive mode, a very deep, well-developed rooting system, and efficient physiological metabolism (Sanderson et al., 1996; McLaughlin et al., 1999). The crop can also be used for bedding under animals, for the mushroom industry and for paper pulp production to replace hardwoods (Sharma et al., 2003). Due to the high productivity of the grass it can be grown by farmers on marginal land offering a cash crop and a boost to the farm economy (Boylan et al., 2000; Alizadeh et al., 2005).

Zhan et al (2005) examined the feasibility of producing ethanol using switchgrass as the primary raw material. They found that while switchgrass is not currently grown commercially as a feedstock for energy production, it appears viable in the Southeast, Midwest, and Plains states. To support ethanol production, switchgrass can be grown at regional farms, cut, field dried, baled and transported by truck to a switchgrass-to – ethanol conversion facility. The bulky nature of switchgrass results in high transportation cost for the raw material (Zhan et al., 2005). Pelleting switchgrass raises the bulk density of the feedstock eliminating high transportation costs when enroute to a conversion facility. A study carried out by Jannasch et al (2001a) found that the calorific value of switchgrass pellets (19.01 MJ/kg) is similar to that of wood (19.60 MJ/kg).

PHYSICAL PROPERTIES

Knowledge of physical properties of biological materials is important for handling, storage, and transportation purposes. Some of the moisture dependent physical properties of biological materials are shape, size (diameter and length), bulk and particle densities, porosity, hardness and durability (Balasubramanian, 2001). These physical properties can be used to determine whether a pelleted biological material will maintain its integrity during handling and transportation from the place it is manufactured to the place of utilization (Thomas and van der Poel, 1996).

The dimensions of pellets in terms of length and diameter are important for combustion processes. Thinner pellets give rise to a more uniform combustion rate especially in small furnaces. The length of the pellets affects the fuel feeding qualities, where, shorter pellets allow for an easier continuous flow (Lehtikangas, 2001). Bulk density affects the transportation costs and efficiency of handling and storage, thus the need to densify most biomass (Lehikangas, 2001; Sokhansanj and Turhollow, 2004).

Strength properties include hardness and durability. Hardness describes the force needed to rupture the pellet (Thomas and van der Poel, 1996; Lehikangas, 2001). Fragmentation or rupture of inhomogeneous materials usually occurs near or at the point of inhomogeneities due to local stresses and strains being higher near points of imperfection. Fragmentation of homogenous materials can be errors in crystalline structure or small holes in or below the surface. Within pellets, particles of differing sizes, shapes, and hardness are aggregated. The point of aggregation is called a crack tip where stresses will accumulate. When the local stress at the tip of a crack becomes higher than the cohesive or adhesive stresses the crack begins to grow and fracture occurs (Thomas and van der Poel, 1996).

Durability describes the amount of fines produced after being exposed to mechanical or pneumatic agitation (Thomas and van der Poel, 1996; Lehtikangas, 2001). Fines are formed when pellets are dropped from the conveyor down to a pile. The fines can accumulate under transport conveyors resulting in an explosion. There is an increased tendency of fines to absorb moisture, which can leave the biomass susceptible to microbial attack. Therefore, high durability of pellets is advantageous when transporting pellets at the plant or to the end user (Lehikangas, 2001). Durability is considered high when above 80%, medium when between 70% and 80% and low when below 70% (Tabil and Sokhansanj, 1996; Tabil and Sokhansanj, 1997; Adapa et al., 2003a). Pellets are more sensitive to shearing at the places where they are cut off after leaving the die (Thomas and van der Poel, 1996). Improper cooling can increase the pellet's sensitivity due to stresses in the pellet between the cooled outer layer and the warmer center (Thomas and van der Poel, 1996; Lehikangas, 2001). During the cooling process, the cooling air is used to take up moisture and heat from the pellets. In a steady state situation, the same amount of water is transported through capillaries to the surface of the pellet. When the speed of air is increased more water and heat are removed from the pellet surface than can be delivered by the capillaries. This leads to a brittle outer layer with different physical properties than the interior of the pellet. These differences create stress, which cause the outer layer to crack under less than optimal conditions. The cracks allow for more fine formation (Thomas and van der Poel, 1996).

The effect of moisture on physical properties has been studied for different types of biological materials. Fasina et al (1994) investigated the effect of increasing moisture on pellets made from alfalfa. The bulk density of the alfalfa pellets decreased with increase in moisture content. This was because the increase in mass due to moisture gain was lower than the increase in volume. Similar results have been reported for other biological materials (Deshpande et al., 1993; Nimkar and Chattopadhyay, 2001; Balasubramanian, 2001; McMullen et al., 2005). When this occurs, the amount of storage space required per unit mass of material increases with increasing moisture content. The opposite can also occur in biological materials indicating the change in volume is less than the corresponding change in mass with increase in moisture, (Joshi et al., 1993; Chandrasekar and Viswanathan, 1999; Jha 1999). Bulk and particle densities are indicators of the pelletability of a material. Higher initial values may lead to good quality pellets and easier pelletability (Jannasch et al., 2001c).

Moisture Sorption Isotherms

Biological materials are hygroscopic in nature. These materials therefore, have the ability to exchange moisture with the atmosphere (Singh, 2004). Knowledge of the equilibrium moisture content (EMC) - equilibrium relative humidity (ERH) relationship is essential to designing and optimizing post harvest operations such as storage, drying, aeration, handling, packaging, and processing of biological materials (Singh, 2004; Durakova et al., 2005; Pangano and Mascheroni, 2005; Erbas et al., 2005). The equilibrium moisture content is the moisture content at which a hygroscopic material is in equilibrium with a particular environment. The relative humidity of that environment is the equilibrium relative humidity (ERH). A plot of the EMC-ERH at a particular temperature is the moisture sorption isotherm.

The moisture isotherm curve is often used to determine the storage stability of biological materials when exposed to varying environmental conditions during transportation and storage (Erbas et al., 2005). For example, most alfalfa pellets and cubes produced in Canada are exported overseas. While in transport, these products are exposed to high humidity and low temperature conditions which cause moisture uptake. This increase in moisture during exposure to humid conditions reduces the durability and storage stability of the alfalfa pellets and cubes (Fasina and Sokhansanj, 1992; Fasina and Sokhansanj, 1993). EMC data can also determine the lower and upper limit up to which biomass should be dried. Information about the mechanism of water binding during sorption can also be attained. Limited amounts of moisture are beneficial as the steam generated causes steam gasification reaction leading to better gas quality where high moisture can result in swelling, disintegration, and prevention of application for thermo-chemical conversion (Singh, 2004).

In drying, the difference between the product moisture content and the EMC is often used as a measure for the driving force (Jenkins, 1989). EMC data are also used for thermodynamic analysis of water sorption. Thermodynamic properties that can be obtained from equilibrium moisture studies include heat of sorption, free energy, and entropy. The heat of sorption is beneficial in estimating the heat requirement during drying and the state of absorbed water in the solid materials. The level of moisture at which the heat of sorption approaches the heat of vaporization of water is taken to be indicative of the amount of bound water in the material of interest. At moisture contents higher than this level, water is free in the void spaces of the system and readily available for microorganisms (Fasina and Sokhansanj, 1993).

More than 200 equations (theoretical, semi-empirical and empirical) have been developed to determine the relationship between the EMC and ERH of agricultural and biological materials. However, none of these equations describe the sorption process for the entire range of water activity (Soysal and Oztekin, 2001). The best known isotherm equations are Brunauer-Emmet-Tetler (BET), Langmuir, Halsey, Henderson, Chung-Pfost, Chen-Clayton, Iglesias-Chirife, and Guggenheim-Anderson-de-Boer (GAB). Soysal and Oztekin (2001) concluded that the BET equation is suitable for most materials, especially for hydrophilic polymers below water activity of 0.5. The Hasley equation is appropriate for hydrophilic polymers and rubbers, plastics, synthetic fibers and foods rich in soluble components. The Chung-Pfost equation is suitable for cereal and other field crops, while the Iglesias-Chirife equation has been suitable for foods rich in soluble components. The GAB equation is considered the most versatile model for various materials over a wide range of water activity. Models adopted as standard equations by the American Society for Agricultural and Biological Engineers (ASABE) (ASABE Standard D245.5) includes Modified Chung-Pfost, Modified Henderson, Modified Halsey, and Modified Oswin equations (ASABE, 2001; Durkova et al., 2005). The Halsey equation was developed for high protein and oil content food products while the Modified Henderson and Chung-Pfost equations have been suitable for starchy foods (Fasina and Sokhansanj, 1993; Soysal and Oztekin, 2001). These equations have been used to model the moisture sorption isotherms of various agricultural and biological materials such as alfalfa pellets (Fasina and Sokhansanj, 1993), cotton plant components

(Barker, 1996), 10 medicinal and aromatic plants (Soysal and Oztekin, 2001), amaranth grains (Pagano and Masheroni, 2005), chickpea flour (Durakova and Menkov, 2005) and semolina and farina (Erbas et al., 2005). Usually in these studies, the researchers determined the equation that best fit the experimental data for various biological materials.

In general, one or more of the following five parameters have been used to quantify the goodness of fit of the equations to moisture isotherm data: the coefficient of determination (R^2), the residual sum of squares (RSS), the standard error of the estimate (SEE), the mean relative deviation (MRD) and the plots of residuals (Fasina and Sokhansanj, 1993; Barker, 1996; Soysal and Oztekin, 2001; Erbas et al., 2005; Durkova et al., 2005; Pagano and Mascheroni; 2005). If the model is acceptable the residuals should be random independent errors with a zero mean, constant variance, and arranged in a normal distribution (Pagano and Mascheroni, 2005). If the residuals show a clear pattern, the model is unacceptable. Because all the information is contained in the residuals, the analysis of residuals is a valuable tool for diagnosis (Fasina and Sokhansanj, 1993; Pagano and Mascheroni, 2005). Models can be found unsuitable by more than one statistical criterion. Pagano and Masheroni, (2005) found that at first glance R^2 values were relatively high for all models (R^2>0.9803) showing that all models can be considered valid. However, the value of R^2 is not by itself a solid or robust analysis index. For example in the same study by Pagano and Masheroni (2005), it was found that the modified Halsey equation gave higher values of RSS, SEE and MRD and the smallest values of R^2. This suggests that the Modified Halsey equation is not the most appropriate model for describing the experimental data. In short, low values of R^2, high values of RSS, SEE, and MRD, and clear patterns in residual plots are indicative of the inability of the model to explain the variation in experimental data (Barker, 1996; Pagano and Masheroni, 2005).

Rate of Moisture Sorption

In the previous section, it was mentioned that biological material will sorb or desorb moisture until reaching equilibrium with the environment. This section deals with the rate at which the moisture sorption process takes place. As expected, several investigators have reported that the moisture sorption rate is a function of temperature and relative humidity of the environment and the type of material (i.e. chemical composition and the physical form – e.g. cubes versus pellets (Zink, 1935; Dexter et al., 1947; Fasina and Sokhansanj, 1992). Fasina and Sokhansanj (1992) concluded that the moisture absorption rate for alfalfa cubes and pellets was affected by temperature and relative humidity.

Chhinnan (1984) in designing an experimental dryer for thin layer studies investigated drying models to find the most appropriate for the application of drying pecans. The exponential model was found to be one of the simplest models to describe moisture movement in solids. This model assumes negligible internal resistance which means there is no resistance to moisture movement from within the material to surface. It only considers the surface resistance implying all the resistance is concentrated in a layer at the surface of the material. Other researchers have been successful at employing this model to describe moisture sorption rates in biological materials (Fasina and Sokhansanj, 1992; McMullen et al., 2005). Other mathematical models used to predict moisture sorption rates include: the Peleg model (Dadgar et al., 2004; Mali et al., 2005), Fick's diffusion model (Chhinnan, 1984; Dadgar et al., 2004; Bello et al., 2005), Page (Dadgar et al., 2004) two term exponential (Dadgar et al., 2004). Dadgar et al (2004) found that the Page and two-tem experimental model best predicted moisture adsorption data for field peas.

COMPACTION

Biomass has relatively low bulk density, which causes problems during storage, handling and transportation for further processing. The bulk density of some biomass range from around 40 kg/m^3 for loose straw and bagasse to 250 kg/m^3 for some wood residues (Mani et al., 2003a). Densification of biomass into pellets or briquettes increases the volumetric energy content, reduces transportation costs and makes a variety of applications available. Densification involves using some form of mechanical pressure to reduce the volume of biological matter (Bruhn, 1989; Mani et al., 2003a). The mechanism of particulate bonding further explains the densification process.

During the first stage of compression, particles that are preheated through dry blending or wet granulation, rearrange themselves to form a closely packed mass. During this phase, the original particles retain most of their properties and energy is dissipated due to inter-particle and particle-to-wall friction. At high pressures, the particles are forced against each other even more and undergo elastic and plastic deformation, increasing inter-particle contact. When the particles approach each other closely, short range bonding forces like van der Waal's forces, electrostatic forces and sorption layers become effective (Chin and Siddiqui, 2000; Mani et al., 2003a). Under stress, brittle particles may fracture leading to mechanical interlocking. Mechanical interlocking is the only bonding mechanism that does not involve atomic forces and therefore contributes very little to the overall strength of the pellet. At higher pressures, the volume is reduced further until the density of the pellet approaches the true densities of the component ingredients. There is a close correlation between the increase in density and the increase in applied pressure in the early stages of compression but the rate of increase in density falls off rapidly as the density of the pressed material approaches the density of water (Mani et al., 2003a).

The overall goal during pelleting is to create a more fluid process, where a lower friction coefficient is created between the die extrusion surface and the fiber. This can be done during conditioning step that has been proven to improve pellet durability and production rates, decreasing the energy consumption of the pellet mill where steam commonly used for conditioning acts as a lubricant reducing friction during pelleting (Gilpin et al., 2002). The pellet is bound together by the lignin component of the feedstock. This process results when fiber passes through the extrusion holes, heating up the die and creating elevated temperatures. Lignin within the material starts to flow from the fiber cell walls and has the effect of binding with other fibers during extrusion (Jannasch et al., 2001a). During the process some moisture is driven off as stream. The resulting product is a uniform flowing material with a bulk density 2-4 times higher than that of the starting raw material. Since the composition of biological materials are not the same, material quality and type have been found to affect the compaction (or densification) of agricultural materials (Tabil and Sokhansanj, 1997; Samson et al., 2000).

Process Variables Affecting the Compaction Process

The properties of a biological material that mostly contribute to the amount of densification that can be achieved in a material are moisture content of material, grind characteristics and conditioning temperature (Jannasch et al., 2001a; Mani et al., 2003a). The effect of these properties on the densification efficiency is described next.

Moisture

Moisture content plays a large role in determining density and strength of densified biomass. The production of stable and durable pellets or briquettes requires the material to be conditioned to an optimum moisture content before pelleting (Wamukonya and Jenkins, 1995; Mani et al., 2003a). Density is influenced by moisture in two ways. First, by the change in mass with changing moisture content and second, by the change in volume of the particles as the moisture content changes below the fiber saturation point. The fiber saturation point is the moisture content of the material at which the cell walls are completely saturated while the cavities are liquid free (Jenkins, 1989). In the pelleting/briquetting process, moisture acts as a film type binder that strengthens the bonds between particles. In the case of organic and cellular products, moisture helps in promoting bonding by van der Waal's forces by increasing the true area of contact of the particles (Lehtikangas, 2001). The right amount of moisture therefore, helps in developing the self-bonding properties in lignocellulosic substances especially at elevated temperatures and pressures that are typically used in briquetting and pelleting machines. However, a high moisture content of the raw material can make the feedstock slippery. This can make the material slide easily through the holes of the die in the pellet mill thereby reducing pellet quality. Similarly, Lehtikangas (2001) concluded that when materials are too dry they clog the die because the resistance rol the die holes exceeds the roller force. When this occurs, the pelleting operation stops and significant down time results due to the unclogging of the dies Therefore, the optimum moisture content that will give high quality pellets varies according to the type of biomass. Most of the studies that have been reported on the effect of moisture on biomass densification have been carried out on briquettes.

Mani et al (2003a) found that the density of hay briquettes decreased as moisture content increased from 28 to 44%. The authors found that when the feed moisture content was between 8 and 10%, the briquettes were strong and free of cracks and the briquetting process was smooth. At higher moisture levels (> 10%), the briquetting process was erratic and the briquettes were weak.

Chin and Siddiqui (2000) investigated the characteristics of biomass including sawdust, rice husks, peanut shells, coconut fibers, and palm fruit fiber. The biomass was densified at 5-7 MPa and tested to evaluate relaxation behavior, mechanical strength and burning characteristics. The moisture content prior to compaction ranged from 5-30%. Briquettes with moisture content of approximately 20% showed the least relaxation. Wamukonya and Jenkins (1995) produced relatively high quality briquettes from wood residue and wheat straw with an optimum moisture content range of 12-20% (wet basis). Since the wheat straw had lower moisture content, the durability of the manufactured briquettes was lower than that of the wood residue briquettes. In the feed industry, the general consensus is that high levels of heat and moisture in the conditioner achieve proper pelleting for grain-based diets high in starch (Chin and Siddiqui, 2000). Moisture is important because the right amount develops self-bonding properties in lignocellulosic substances at the elevated temperatures and pressures prevalent in briquetting and pelleting machines.

Particle Size

The size (average and distribution) of particles is very crucial to the quality of pellets obtained during densification (Lehtikangas, 2001; Mani et al., 2003a; Adapa et al., 2004). Because of the nature of biomass, it has to be ground (usually by means of a hammer mill) to achieve the particle size that is optimum for pellet production. Reduction of particle size increases the total surface area, pore size of material, and the number of contact points for inter-particle bonding in the compaction process. Particle size of grinds will therefore have an effect on the quality of the densified masses.

Grinding of biomass was found to contribute to the production of higher density briquettes with better durability, lower water absorption rates, and an increase in binding properties of the feedstock (Jannasch et al., 2001b; Lehtikangas, 2001; Mani et al., 2003a). It was reported, however, that wider particle size distribution is more suitable for compaction because the smaller (fine) particles can rearrange and fill the void spaces of larger (coarse) particles thereby producing denser and more durable compacts. Coarsely ground materials tend to give less durable pellets because they may create natural fissures in the pellet, which are susceptible to breakage. A combination of fine and medium grind is therefore essential for compaction (Mani et al., 2004a).

In a study of 34 feed mills in the United States, it was reported that pellet quality appeared to be the highest in feed mills equipped with hammer mills having the 3.2-4.0 mm split screens. Plants using a hammer mill with 4.0 mm screen appeared to have the lowest pellet quality (Mani et al., 2004a). Similarly, it was found by Jannasch et al (2001b) that a reduction in screen size from 3.2 mm to 2.8mm for the fine grinding process for switchgrass appeared to produce an increase in pellet hardness. Adapa et al (2004) reported results that indicated a decrease in hammer mill screen size (3.20 mm to1.98 mm) gave rise to higher durability of fractionated sun-cured and dehydrated alfalfa pellets.

In agricultural and biological process engineering, the size (average and distribution) of particles is often determined according to the ASABE Standard S319.3 (ASABE, 2003). Projector and image analysis, gravitational sedimentation, centrifugal sedimentation and scanning electron microscopy (SEM) have also been used for determining particle size distribution (Yang et al., 1996). .

Temperature

Temperature plays a major role in stability and durability of the product, and in the amount of energy required for compaction. The addition of heat to the densification system can be by means of wet steam, preheating feed materials or using heated die apart from the frictional heat generated due to compression (Mani et al., 2003a). In the case of alfalfa, the addition of high temperature steam enhanced pellet durability and reduced energy consumption in the pelleting process. Tabil and Sokhansanj, (1996) conducted a study for improving the physical quality of alfalfa pellets by controlling and optimizing manufacturing parameters and found that pellet durability increased when the conditioning temperature was raised from 65 to 95°C. The conditioning step is very important because the overall goal is to create a more fluid pelleting process, by minimizing the friction coefficient between the die extrusion surface and the fiber making the fiber more pliable. The combination of these effects results in optimal extrusion of the feedstock through the dies. Since switchgrass is made up of at least 20% lignin Wiselogel et al., 1996; Esteghlalian et al., 1997; Alizadeh et al., 2005), it is necessary for the pelleting process to include conditioning of the ground switchgrass to at least 75°C before extrusion through the die (Samson et al., 2000).

COMPOSITION ANALYSIS

It must be noted that the composition of switchgrass can vary according to the time of year it was harvested and how it was stored (Dale et al., 1996; Wiselogel et al., 1996). It has been reported that the cellulose and lignin content of switchgrass increases as the plant ages during the growing season, while the hemicellulose and other soluble components decrease (Dale et al., 1996). Changes in any feedstock constituents can have negative effects on the profitability of both thermochemical and biochemical conversion processes (Wiselogel et al., 1996).

Components of interest include: structural carbohydrates, ash and lignin. Ash in biomass feedstocks promotes char formation during pyrolysis, and forms fusible ash at high temperatures in combustion units used for steam generation. Lignin is the component that has the highest carbon and energy component. Changes in lignin can impact thermochemical and biochemical conversion processes. Increased lignin content upon storage can be expected at the expense of carbohydrate components (Wiselogel et al., 1996). In this study the heat treatment during conditioning of the feedstock may give rise to higher lignin content in the pelleted sample. However in biochemical processes, the loss of lignin is viewed as a pretreatment to make the structural carbohydrates more susceptible to hydrolysis (Saddler et al., 1993; Dale et al., 1996; Szczodrak and Fiedurek, 1996; Lynd, 1996; Wiselogel et al., 1996; Esteghlalian et al., 1997; Soderstrom et al., 2003; Liu and Wyman, 2005; Kim and Lee, 2005a; Kim and Lee, 2006).

SUMMARY

The potential for switchgrass to be used widely as an alternative to current energy resources is very promising. The densification of this important, lightly dense, biomass can be optimized by investigating processing parameters which in turn can lead to lower transportation costs and easier storage and handling. Understanding the effect of moisture on the physical properties of these densified masses is also important for maintaining quality. Knowing the composition of the feedstock is valuable for optimizing conversion and pretreatment techniques for ethanol production.

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CHAPTER 2 – EFFECT OF MOISTURE ON THE PHYSICAL PROPERTIES OF SWITCHGRASS PELLETS

INTRODUCTION

Switchgrass (*Panicum virgatum*) is a high yielding perennial grass species that is native to North America. The crop has been designated as an energy crop by the US Department of Energy because of its high biomass production from which renewable sources of fuel and electricity can be generated (Missaoui et al., 2005; McLaughlin and Kszos, 2005) and because it has excellent conservation attributes and good compatibility with conventional farming practices (McLaughlin et al., 1999).

Similar to other forage crops, switchgrass is lightly-dense (< 150 kg/m^3) when harvested (Sokhansanj and Turhollow, 2004) and therefore cannot be efficiently and economically transported over long distances to areas where effective utilization can occur. Densification by pelletizing is one of the effective methods used to increase the value of agricultural and biological materials (Fasina and Sokhansanj, 1996; Barger, 2003).

Pellets are manufactured by grinding, conditioning (application of heat and/or moisture) and forcing the ground sample through dies that range in diameter from 4 to 12 mm or even larger (Fasina and Sokhansanj, 1993). Physical properties of switchgrass pellets will be needed for the proper design and selection of systems and equipment to store, handle and transport the pellets (Mohsenin, 1986). Several published studies have revealed that moisture content has a significant influence on the physical properties of biological materials such as switchgrass pellets (Nelson, 2002; McNeill et al., 2004; McMullen et al., 2005).

Some common physical properties of interest include: size (length and diameter) bulk and particle density, durability, hardness, and moisture exchange. Length and diameter are important for combustion processes where thinner (small diameter) pellets give rise to a more uniform combustion rate, especially in small furnaces. The length of the pellets affects the fuel feeding qualities, where, shorter pellets allow for an easier continuous flow (Lehtikangas, 2001). Bulk density affects the transportation costs and efficiency of handling and storage, thus the need to densify most biomass (Lehikangas, 2001; Sokhansanj and Turhollow, 2004).

Hardness describes the force needed to rupture the pellet (Thomas and van der Poel, 1996; Lehikangas, 2001). Fragmentation or rupture of inhomogeneous materials usually occurs near or at the point of inhomogeneities due to local stresses and strains being higher near points of imperfection. Fragmentation of homogenous materials can be errors in crystalline structure or small holes in or below the surface. Within pellets particles of differing sizes, shapes, and hardness are aggregated. The point of aggregation is called a crack tip where stresses will accumulate. When the local stress at the tip of a crack becomes higher than the cohesive or adhesive stresses the crack begins to grow and fracture occurs (Thomas and van der Poel, 1996).

Durability describes the amount of fines produced after pelleted materials are exposed to mechanical or pneumatic agitation (Thomas and van der Poel, 1996; Lehtikangas, 2001). It is therefore a measure of the rate at which fines will be generated during the handling, storage and transportation of pelleted materials. It is generally desired that the integrity of pellets (i.e. minimal fines generation) be maintained until the time of utilization of the pellets (Aarseth and Prestlokken, 2003). This is because fines have been known to contribute to explosion and fire hazards (Woodcock and Mason, 1987). In addition, fines from pelleted materials generally absorb moisture from the environment, which has been found to increase the susceptibility of the bulk material to microbial attack (Lehikangas, 2001).

Biomass materials are hygroscopic and will therefore exchange moisture with the environment. Knowledge of the rate of moisture uptake and equilibrium moisture relationships are important for characterizing product quality during storage and for defining optimal storage conditions (Jenkins, 1989). High moisture content in storage may lead to loss in fuel values, growth of microorganisms and spontaneous combustion of biomass material intended for thermal conversion (Jenkins, 1989).

The objective of this chapter is to investigate the effect of (a) pelleting on the composition of switchgrass and (b) increasing moisture on the physical properties of switchgrass pellets. The physical properties of interest include size (length and diameter), bulk and particle density, porosity, hardness and durability.

MATERIALS AND METHODS

Pelleting

The switchgrass samples that were used in this study were collected from the E.V. Smith Experiment Station (Auburn University), Tallassee, Alabama. Before pelleting, the switchgrass was ground through a 3.18 mm screen using a hammer mill (New Holland Grinder, Model 358, New Holland, PA) shown in Figure 2.1. A laboratory scale pellet mill (Model CL5, California Pellet Mill Co., San Francisco, CA) was used to manufacture the pellets used in this study (Figure 2.2).

The pellets were made by extruding the ground switchgrass through round cross-sectional dies. Before passing through the pellet die, the feed material was conditioned by increasing the temperature to 75°C and increasing the moisture content from 15.6 to 30.1%. Frictional heating of the die during pelleting further increased the temperature of the pellets exiting the die to 85°C. The die size used was 4.76 mm. After pelleting, the pellets were cooled in an environmental chamber set at 22°C and 40% relative humidity.

Figure 2.1 – Hammer mill

Figure 2.2 – Pilot scale pellet mill

Figure 2.3 – Ground (3.18 mm screen) and pelleted switchgrass (4.76 mm diameter)

Composition Analysis

Composition analysis was performed on ground and pelleted switchgrass according to National Renewable Energy Laboratory (NREL) Laboratory Analytical Procedure - Determination of Structural Carbohydrates and Lignin in Biomass (NREL, 2004). The pelleted and ground switchgrass was ground using a coffee grinder (Smart Grind, Black and Decker Corp., Towson, MD).

Structural Carbohydrate Analysis

Structural carbohydrates (glucose, xylose, arabinose, galactose, and mannose) were determined by using a two stage sulfuric acid hydrolysis. Samples were prepared in duplicate. In the first stage, each sample (ground and pelleted) was incubated at 30 ± 3°C in a constant temperature shaking bath (Model BT-25 Yamato, Lorton, VA) with 3.0 mL of 72% sulfuric acid for 1 hour. After 1 hour the acid was diluted to 4% with 84 ± 0.04 g of dionized water using a balance (Model AE-160, Mettler Toledo Co., Columbus, OH) accurate to 0.01 g. In the second stage the diluted samples were autoclaved (Model 2540E, Tuttnauer Brinkmann, Ronkonkoma, NY) at 121 °C for 1 hour. Sugar recovery standards (SRS) were prepared and taken through the second stage. The SRS were used to correct for losses due to the destruction of sugars during dilute acid hydrolysis. The SRS included D-(+) glucose and D-(+) xylose.

Samples were neutralized with calcium carbonate to pH 5-6 (this was monitored with pH paper). After neutralization, the samples were centrifuged for ten minutes at 16,000g (17308-Series Micro-centrifuge, Cole Parmer Instrument Co., Vernon Hills, IL). HPLC was used to quantify the amount of released sugars in the feedstock by a Bio-Rad Aminex HPX-87P column (Bio-Rad Laboratories, Hercules, CA). The HPLC conditions were as follows:

Injection volume – 10-50 µL
Mobile phase – HPLC grade dionized water, .2 µm and degassed
Flow rate – 0.55 mL/minute
Column temperature – 80-85°C
Detector temperature – close to column temperature
Detector – refractive index
Run time – 35 minutes

Acid Insoluble Lignin

To determine the acid insoluble lignin (AIL) content, the samples (from the two stage hydrolysis) were vacuum filtered through a filter crucible using dionized water to transfer all remaining solids. The filtrate was captured in a filtering flask and approximately 50 mL of filtrate was collected in a storage bottle to determine acid soluble lignin and carbohydrates. The crucibles containing acid insoluble residue were dried in an oven (ESP-400C, StabilTherm, Blue Mountain Electric Co., Blue Island, IL) at 105 ±3 °C until a constant weight was achieved. After drying in the oven, crucibles were placed in a muffle furnace (Type-1500, Thermolyn Sybron Corp., Milwaukee, WI) at 575 ± 25°C for 24 ± 6 hours after which the crucibles with ash were cooled and constant weight was recorded. Samples were prepared in duplicate. Acid insoluble residue (AIR) and lignin (AIL) were calculated using Equations 2.1 – 2.3.

$$ODW = \frac{WA \times \%TS}{100} \tag{2.1}$$

$$\%AIR = \frac{CR - C}{ODW} \times 100 \tag{2.2}$$

$$\%AIL = \frac{(CR - C) - (CA - C)}{ODW} \times 100 \tag{2.3}$$

Where, ODW = oven dried weight (g)

C = weight of crucible (g)

CR = weight of crucible plus acid insoluble residue (g)

WA = weight of air dried sample (g)

CA = weight of crucible plus ash (g)

TS = Total solids in sample (%)

Acid soluble Lignin

To analyze for acid soluble lignin (ASL), the absorbance of the hydrolysis liquor was measured at a wavelength of 320 nm with a UV-Visible spectrophotometer (Model Synergy HT, Biotek Inc., Woburn, MA).

$$\%ASL = \frac{UVabs \times V_{filtrate} \times Dilution}{\varepsilon \times ODW} \quad (2.4)$$

$$Dilution = \frac{V_s + V_{ds}}{V_s} \quad (2.5)$$

Where, UVabs = average UV-Vis absorbance for the sample at 320 nm

$V_{filtrate}$ = volume of filtrate (87 mL)

V_s = volume of sample (mL)

V_{ds} = volume of diluting solvent (mL)

ε = absorbance of biomass at specific wavelength

Ash

For ash analysis, approximately 0.900 g of switchgrass (ground and pelleted) was placed in crucibles which were put in the aforementioned muffle furnace at 575 ± 25°C for 24 ± 6 hours according to NREL Laboratory Analytical Procedure - Determination of ash in biomass (NREL, 2005). After the allotted time, crucibles and ash were removed from the furnace and put directly into a dessicator and constant weight was recorded. Total solid (TS) analysis was performed at the same time using a moisture analyzer (Model IR-30, Denver Instruments, Arvada, CO). The percent of ash in the sample was calculated from Equation 2.6:

$$\%Ash = \frac{CA - C}{ODW} \quad (2.6)$$

Particle size Distribution

Particle size distribution was determined according to ASABE Standard S319.3 (ASABE, 2003). This involves placing 100 grams of material on the top sieve of a nest of successively smaller sieves and recording the weight of material retained on each sieve after the test is complete. For this analysis, 7 U.S. Series test sieves plus a pan with aperture sizes ranging from 1.70 to 0.212 mm were used. The nest of test sieves was shaken for ten minutes in a Sieve shaker (Model CL 340, Soil Test Engineering Test Equipment Co., Evanston, IL), after which, the nest of sieves were removed from the autoseiver unit and the mass of material retained on each sieve was recorded. The determination of the geometric mean diameter (d_{gw}) of the sample and the geometric standard deviation of particle diameter (S_{gw}) was carried out according to the ASABE Standard S319.3 (ASABE, 2003) (Equations 2.7 – 2.9):

$$d_{gw} = \log^{-1}\left[\frac{\sum_{i=1}^{n}(W_i \log \bar{d}_i)}{\sum_{i=1}^{n} W_i}\right] \tag{2.7}$$

$$S_{\log} = \left[\frac{\sum_{i=1}^{n} W_i (\log \bar{d}_i - \log d_{gw})^2}{\sum_{i=1}^{n} W_i}\right]^{1/2} \tag{2.8}$$

$$S_{gw} = \frac{1}{2} d_{gw}\left[\log^{-1} S_{\log} - \left(\log^{-1} S_{\log}\right)^{-1}\right] \tag{2.9}$$

Where, d_{gw} = geometric mean diameter or median size of particles by mass (mm)

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

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

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

n = number of sieves plus one pan

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

Moisture Adjustment

The evaluation of the effect of moisture on the physical properties of the pellets was carried out at five moisture levels (6.3, 8.6, 11.0, 14.8, and 17.0%, (wet basis)). The initial moisture content of the pellets was 6.3% (wet basis). To adjust the moisture content of the pellets to the desired levels, the pellets were placed in an environmental chamber (Model AA-5460A, Espec Corp., Hudsonville, MI) set to 30°C and 90% relative humidity. The pellets were removed from the chamber after gaining the desired amount of moisture. Conditioned pellets were then allowed to equilibrate for a period of 24 hours before testing. Before experimentation, the moisture content of the conditioned pellets was verified with a moisture analyzer (Model IR-200, Denver Instruments, Arvada, CO)

Size

Fifty random pellets were selected for size evaluation. The pellets were weighed using a digital balance accurate to 0.001 grams (Model AR3130, Ohaus Corp., Pinebrook, NJ). The length (L) and diameter (D) were measured using a digital caliper (Model CD-56C, Mitutoyo Corp., Kawasaki, Japan) accurate to 0.01 mm.

Particle Density

The particle density of the pellets was measured by gas comparison pycnometry (Model AccuPyc 1330, Micromeritics Instrument Corp, Norcross, GA) where a known quantity of helium under pressure is allowed to flow from a previously known reference volume into a sample cell containing the material. Based on the pressure difference between the sample cell and the reference cell, the pycnometer calculates the volume of the material in the sample cell. Particle density (ρ_p) was taken as the ratio of the mass of material in the sample (m_p) cell to the volume (V_p) measured by the pycnometer (Equation 2.10). Sample mass was obtained with a digital balance accurate to 0.001 grams (Model AR3130, Ohaus Corp., Pinebrook, NJ). This procedure was performed in triplicate and the average value was reported.

$$\rho_p = \frac{m_p}{V_p} \tag{2.10}$$

Bulk Density

Bulk density was determined by a bulk density measurement apparatus (Burrows Co., Evanston, IL) and according to ASABE Standard S269.4 (2002). This method involves filling the container of the apparatus (volume of 947 mm^3) via a funnel. The material was leveled with the top surface of the container and weighed. The bulk density (ρ_b) of the pellets was taken as the mass of sample in the container (m_c) over the volume of the container (V_c) (Equation 2.11). This procedure was performed in duplicate.

$$\rho_b = \frac{m_c}{V_c} \tag{2.11}$$

Porosity

Porosity is the percentage of the total container volume occupied by air spaces when particulate solids are placed in a container (Fasina and Sokhansanj, 1993). Porosity is mathematically defined by Equation 2.12 and was calculated using the average bulk and particle densities that were obtained in the previous sections.

$$\varepsilon = 1 - \frac{\rho_b}{\rho_p} \tag{2.12}$$

Durability

The durability (Du) of the pellets was determined according to ASABE Standard S269.4 (2002). A 100 gram sample of pellets was tumbled at 50 rpm for 10 minutes, in a dust-tight enclosure (see Figure 2.4). A No. 5 U.S. Sieve with an aperture size of 4.0 mm was used to retain crumbled pellets after tumbling. Durability is expressed by the percent ratio of mass of pellets retained on the sieve after tumbling (m_{pa}) to mass of pellets before tumbling (m_{pb}) (Equation 2.13). Durability is said to be high when the computed value is above 80%, medium when between 70% and 80%, and low when below 70% (Tabil and Sokhansanj, 1996; Tabil and Sokhansanj, 1997; Adapa et al., 2003).

$$Du = \frac{m_{pa}}{m_{pb}} * 100 \tag{2.13}$$

Figure 2.4 – Durability Tester

Hardness

The hardness for the switchgrass pellets was determined using a texture analyzer (Model TA-HD, Stable Micro Systems, Surrey, UK). Fifty pellets that were randomly selected from each sample lot were used in this test. A single pellet was placed on the platform of the texture analyzer in its natural position (the radial dimension was in the same direction as that of the compressive force). A flat plate (50.8 mm diameter) plunger was pressed onto each pellet at a speed of 10 mm/s. The maximum force required to rupture the pellet was determined from the force-deformation curve recorded by the software provided by the manufacturer. This force was taken as a measure of pellet hardness (Thomas and van der Poel 1996; Adapa et al., 2003).

Moisture Sorption

An air-tight environmental chamber (1.8 m x 0.9 m x 0.9 m) was used to investigate the moisture sorption properties of the pellets. Air that was fed into the chamber was conditioned by a temperature-humidity conditioner (Model AA-5460A, Parameter Generation and Control Inc., Black Mountain, NC). Combinations of four air temperatures (15, 25, 35, and 45°C (± 1 °C)) and three air relative humidities (50, 65 and 80% (± 3%)) were used to investigate moisture sorption properties of switchgrass pellets. To conduct a test, a thin single layer of pellets (300-340 g) was placed in a wire mesh basket that hung from a digital balance accurate to 0.01 grams (Model, PM4600, Mettler-Toledo, Columbus, OH). The weight of the sample was monitored and recorded at ten minute intervals during exposure to the conditioned air by using Windmill RS232 communication software (Windmill Software Ltd., Manchester, UK) to interface the balance to a personal computer. A data acquisition system (OMB-Daq-56, Omega Engineering Inc., Stamford, CT) was used to record and monitor the temperature and relative humidity of the conditioned air at ten minute intervals.

Depending on the temperature and relative humidity of the conditioned air, the pellets took between 22 and 68 hours to equilibrate with the conditioned air. An experiment was stopped when the weight of the sample did not change by more than 0.01g within a span of one hour. A moisture analyzer (Model IR-200, Denver Instruments, Arvada, CO) was used to cross check the final moisture content of the pellets. The schematic diagram of the system is shown in Figure 2.5. Each temperature and relative humidity combination was performed in duplicate and average values were reported.

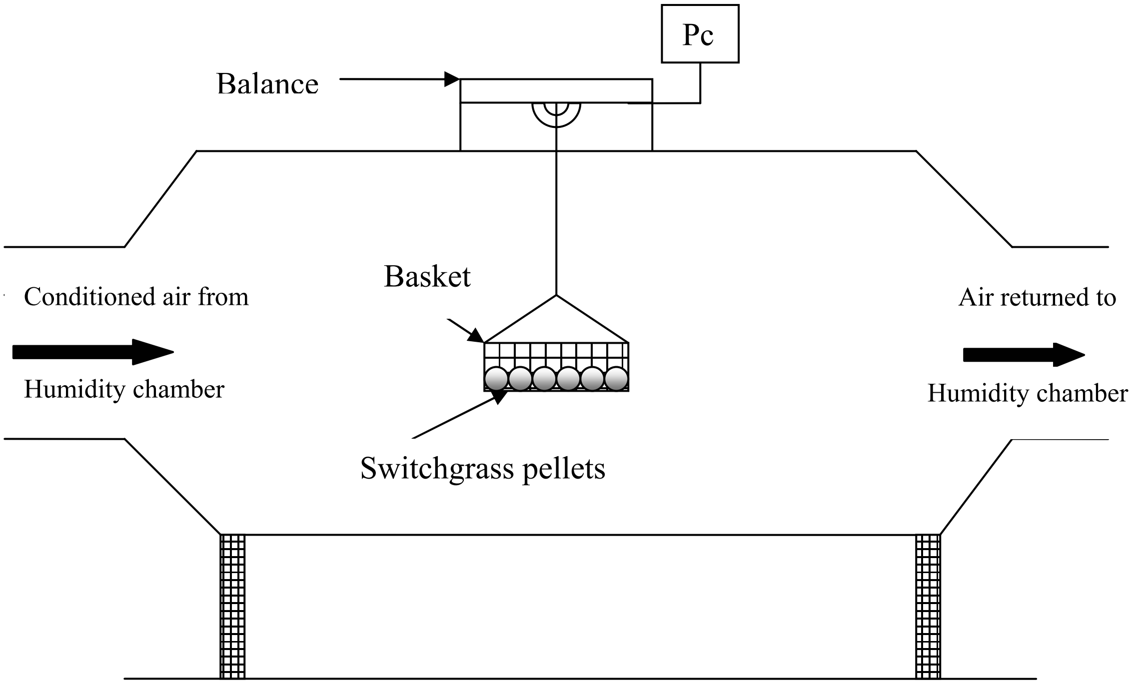


Figure 2.5 – Diagram of environmental chamber used for moisture sorption studies

Moisture Sorption Isotherms

The equilibrium relative humidity (ERH) method was employed to determine the equilibrium moisture relationship of the switchgrass pellets. This method consists of bringing air into equilibrium with a material of fixed moisture content. This method has been found to be simpler and faster than bringing a sample to equilibrium with air at a fixed temperature and relative humidity (Fasina and Sokhansanj, 1992). The pellets were adjusted to desired moisture levels (4.5, 7.2, 9.0, 11.0, and 15.0%, wet basis) in an environmental chamber (Model AA-5460A, Espec Corp., Hudsonville, MI) set at 30°C and 90% relative humidity. Each sample was allowed to equilibrate for 24 hours before each test. The moisture content was verified using a moisture analyzer (Model IR-200, Denver Instruments, Arvada CO).

A water activity instrument (HygroLab 2 - H3, Rotronic Instrument Corp., Huntington, NY) was used to measure the equilibrium relative humidity of preconditioned samples. To conduct a test, the conditioned sample was placed in the sample holder of the water activity instrument. A sealed measurement system was formed by placing the water activity probe on top of the sample holder. The probe is equipped with a small fan that circulates air within the sample container, a thin film capacitance sensor that is capable of measuring relative humidity from 0 to 100% with an accuracy of ± 1.5% and a platinum RTD (resistance temperature detector) temperature probe with an accuracy of ±0.3°C. The measurement system was then transferred into the temperature controlled-chamber set at the desired temperature of 6, 20, 35 or 50˚C. The relative humidity and dry bulb temperature output from the water activity meter was continuously recorded on a personal computer until equilibrium was reached (usually less than 4 hrs). Water activity/moisture sorption analysis was carried out in triplicate. EMC and ERH were taken as the average of the three moisture contents and relative humidities for each sample.

Data Analysis

Statistical analysis was performed on all data sets using SAS statistical software package (Version 9.1, SAS Institute Inc., Cary, NC, 2002-2003) and Microsoft Excel (Windows XP, 2003).

RESULTS AND DISCUSSION

Compositional Analysis

The composition of ground and pelleted switchgrass was similar (Table 2.1). Comparable results were reported by other researchers for the compositional analysis of ground switchgrass (Wiselogel et al., 1996; Esteghlalian et al., 1997; Alizadeh et al., 2005). Alizadeh et al (2005) reported results of 34.2% glucan, 22.1% xylan, and 3.1% arabinan and galactan from NREL laboratories.

At first glance, the pelleted switchgrass had higher values than the ground samples for all properties evaluated except for ash. This could be due to the thermal and mechanical treatment which occurs during the pelleting process. Statistically there were no differences in carbohydrate levels, but there were significant differences in ash, acid soluble lignin and acid insoluble lignin at a 95% confidence level. However, there is no scientific explanation for the increase in the proportions of the variouscomponents of the sample due to pelleting.

Table 2.1 – Composition of ground and pelleted switchgrass

Component	Ground	Stdev	Pellet	Stdev
AIL_a (%)	25.85	1.02	26.70	1.42
ASL_b (%)	13.02	0.75	14.93	0.01
Glucan (%)	35.76	1.94	36.00	0.04
Xylan (%)	21.26	1.29	21.61	0.04
Galactan (%)	0.67	0.94	0.82	0.05
Arabinan (%)	2.21	0.47	2.39	0.05
Mannan (%)	0.99	0.15	0.84	0.06
Ash (%)	1.36	0.74	4.05	3.68

Note: a = Acid insoluble lignin, b = Acid soluble lignin

Particle Size Distribution

The particle size distribution of the ground switchgrass that was used to make the pellets is shown in Figure 2.6 and Table 2.2. Most of the particles (29.51 and 38.55%) were retained on sieves with aperture sizes of 0.595 mm and 0.850 mm. The geometric mean diameter (d_{gw}) and the geometric standard deviation (S_{gw}) of the ground switchgrass were calculated to be 0.867 mm and 0.357 mm respectively (Equations 2.7-2.9). This result, according to other researchers, indicates the particles are not highly compressible. Particles with sizes below 0.400 mm are considered fine and highly compressible. An increase in the amount of fine particles is usually associated with decreased flowability. The more compressible a powder is, the less flowable it will be and vice versa (Tabil and Sokhansanj, 1997; Mani et al., 2003a).

Table 2.2 – Particle size distribution of ground switchgrass

U.S. Sieve No.	Sieve Aperture Size (mm)	Distribution (%)
12	1.700	10.75
20	0.850	38.55
30	0.595	29.51
40	0.425	7.50
50	0.297	6.28
60	0.250	2.07
70	0.212	1.55
pan	0	4.29

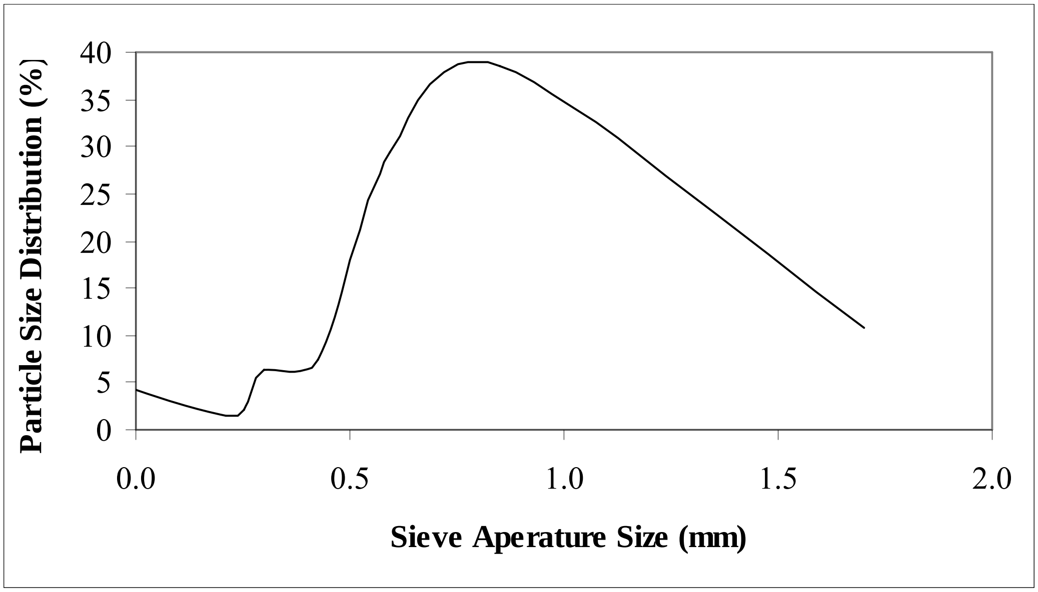


Figure 2.6 – Particle size distribution of switchgrass grind at various sieve apertures

Size

The effect of moisture content on the length (L) and diameter (D) of the pellets is illustrated in Figure 2.7. There was a slight initial increase in the length of the pellets, reaching a maximum of 35.27 mm at 8.62% moisture content. Further increase in moisture reduced the length of the pellets to 33.61 mm. The diameter of the pellets varied from 4.85 mm to 5.25 mm and indicated an increase in diameter with increase in moisture content. The change in length and diameter of the pellets was due to water filling the void spaces of the pellets and disrupting bonds formed during the compaction process. The effects of moisture content on the length and diameter of the pellets are given in the equations below:

For length:

$$L = 26.77 + 1.7024M - 0.0943M^2, \ R^2 = 0.872 \tag{2.14}$$

For diameter:

$$d = 5.19 - 0.0855M + 0.0052M^2\text{, R}^2 = 0.978 \qquad (2.15)$$

Where, M is moisture content (%, wet basis).

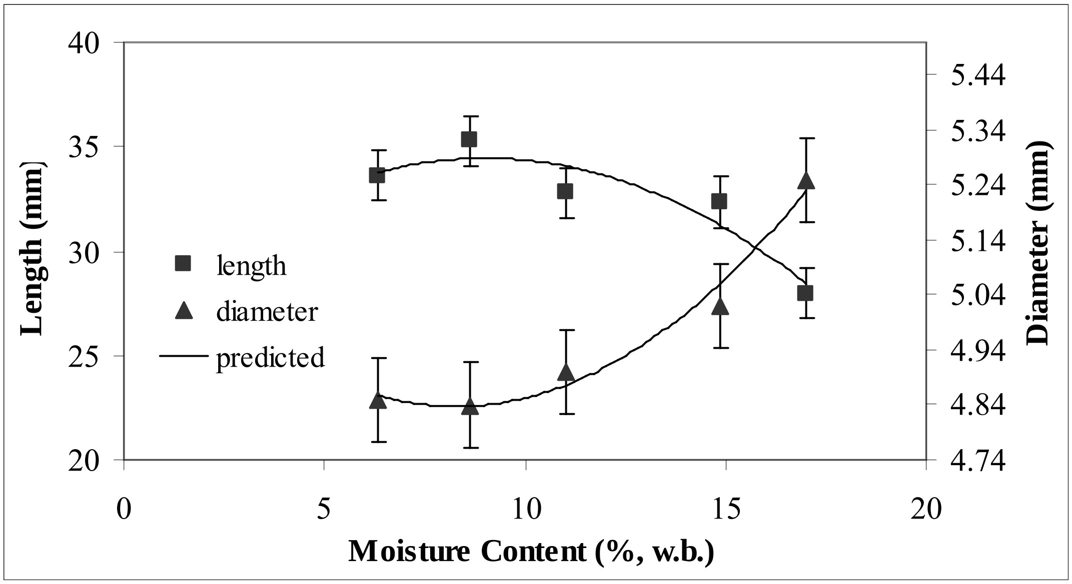


Figure 2.7 – Effect of moisture content on length and diameter of switchgrass pellets

Particle Density

In general, particle density of the pellets decreased with increase in moisture content (Figure 2.8). A maximum value of 1462 kg/m^3 was obtained at moisture content of 8.62% (w.b.). The decrease in particle density is due to the expansion of the pellet, hence, an increase in the volume of the pellets with increase in moisture content. Similar trends were reported for other biological materials (Joshi et al., 1993; Deshpande, 1993). Equation 2.16 shows the effect of moisture content on the particle density of the pellets

$$\rho_{p_{ync}} = 1412.8 + 9.435M - 0.484M^2\text{, R}^2 = 0.940 \qquad (2.16)$$

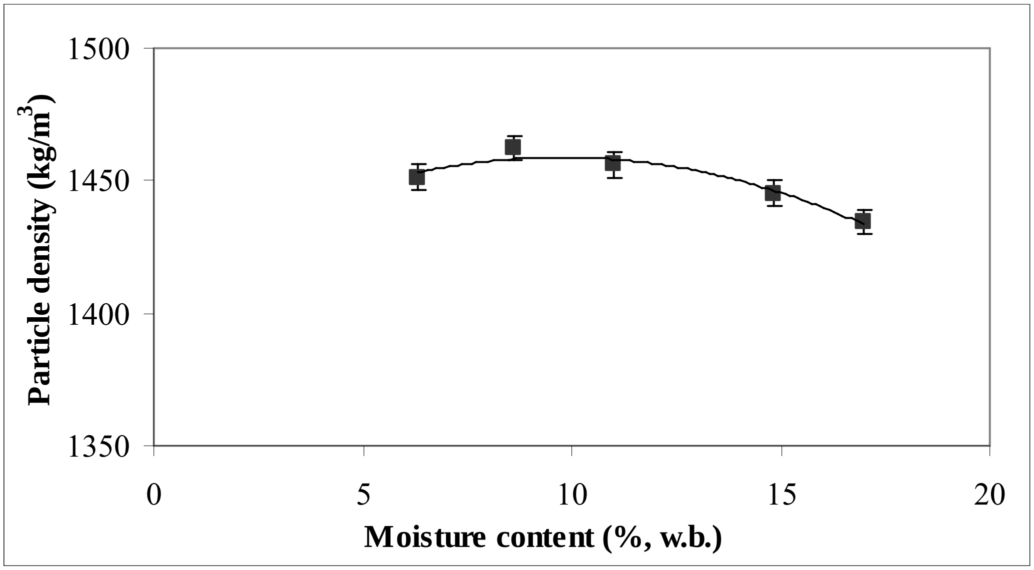


Figure 2.8 - Effect of moisture content on particle density of switchgrass pellets

Bulk Density

The bulk density of the pellets for different moisture levels ranged from 536 kg/m^3 to 708 kg/m^3. Pelleting therefore, reduced the amount of space required to store switchgrass by more than three fold (the bulk density of ground switchgrass was 169.5 kg/m^3). Initially, bulk density of the pellets slightly increased with moisture content, reaching a maximum of 708 kg/m^3 at 8.62% moisture content, and then decreased with further increase in moisture content (Figure 2.9). This is a result of the increase in mass due to moisture gain being lower than the accompanying volumetric expansion of the bulk. Therefore, the amount of storage space required for a given sample will increase with moisture addition. Similar results have been reported for other biological materials (Deshpande et al., 1993; Fasina and Sokhansanj, 1994; Nimkar and Chattopadhyay, 2001; Balasubramanian, 2001; McMullen et al., 2005). The following equation describes the relationship between bulk density and moisture content:

$$\rho_b = 538.15 + 37.518M - 2.2094M^2 \text{ , } R^2 = 0.988 \qquad (2.17)$$

Figure 2.9 compares the bulk and particle densities of the pellets. Bulk density displayed a higher sensitivity to the change in moisture content with a percent change of 24% compared to the percent change in particle density of 16%. Both showed a decrease as moisture content increased which is due to volumetric expansion being the dominant effect. Because bulk and particle densities are indicators of the pelletability of the material, high values result in good quality pellets (Tabil and Sokhansanj, 1997).

Porosity

The porosity of the pellets ranged from 51.61 to 62.62% with varying moisture contents. There was an initial decrease in porosity and a minimum of 51.61% at 8.62% moisture content. As moisture content continued to increase the porosity began to increase. Figure 2.9 and Equation 2.18 show the non-linear relationship between porosity and moisture content. A comparison of porosity of switchgrass pellets with that of other biological materials revealed that the effect of moisture was similar. However, the other biological materials had no minimum value and displayed a linear relationship (Nimkar and Chattopadhyay, 2001; Balasubramanian, 2001; McMullen et al., 2005).

$$\varepsilon = 61.756 - 2.3015M + 0.1381M^2 \text{, } R^2 = 0.990 \qquad (2.18)$$

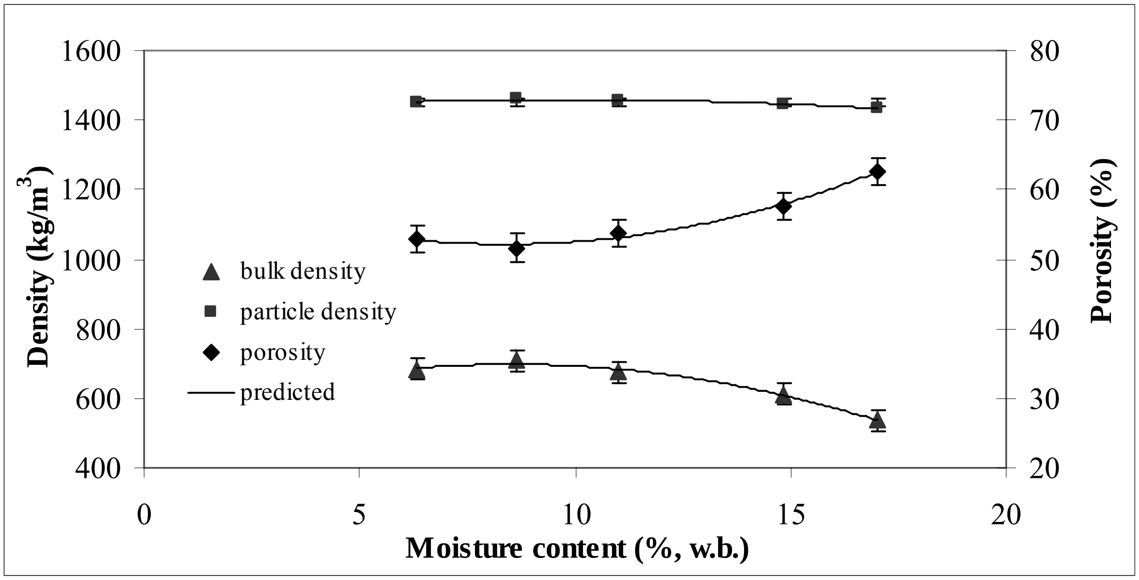


Figure 2.9 – Effect of moisture content on porosity and particle and bulk densities of switchgrass pellets

Durability

Durability (Du) of the pellets increased initially with moisture content and reached a maximum of 96.65% at 8.62% moisture content. Further increase in moisture content reduced durability (Figure 2.10) to 78.44%. Similar trends have been reported for alfalfa pellets (Fasina and Sokhansanj, 1992) and poultry litter pellets (McMullen et al., 2005). It is suspected that initially, the binding forces of the water molecules strengthened the bond between individual particles in the pellets. Further increase in moisture caused the disruption of the particulate bonds (Fasina and Sokhansanj, 1992). A two phase system of results for particles and water with no capillary force present to maintain the pellet structure. This leaves the pellet with cracks which makes the pellets susceptible to breakage (Thomas and van der Poel, 1996).

According to the durability rating cited by Adapa et al (2003), the switchgrass pellets in this study can be classified to have high durability (89.06% to 95.91%) at moisture contents between 6.32% and 14.84%; and medium durability at moisture content values greater than 14.84%. Equation 2.19 shows the relationship between durability and the moisture content of the pellets

$$Du = 78.218 + 4.3179M - 0.25M^2, R^2 = 0.976 \qquad (2.19)$$

It can be noted that durability is highly correlated with bulk and particle density (Fig. 2.11) with correlation coefficients of 0.933 and 0.989 respectively

Hardness

Similar to durability, pellet hardness (H) generally decreased (30.21 N to 21.6 N) with increase in moisture content (Figure 2.10). This is again, due to the moisture disrupting particulate bonds leaving the pellets weak and susceptible to breakage. The relationship between pellet hardness and moisture content is shown in Equation 2.20.

$$H = 32.852 - 0.2377M - 0.024M^2, R^2 = 0.928 \qquad (2.20)$$

Pellet hardness (H) displayed a higher sensitivity to moisture with a percent change of 28.1% from 6 to 17% moisture compared to durability (Du) with a percent change of 18.2% also from 6 to 17% moisture as was seen with poultry litter pellets (McMullen et al., 2005). Several studies examined the effect of moisture on durability and hardness (Fasina and Sokhansanj, 1992; Khoshtaghaza et al., 1999) which found that both properties were stable at moisture contents up to 10%, but decreased rapidly as moisture increased. In this study there was a rapid decrease in pellet hardness and durability after moisture content of 8.62%. A summary of all properties is given in Table A.1 in Appendix A.

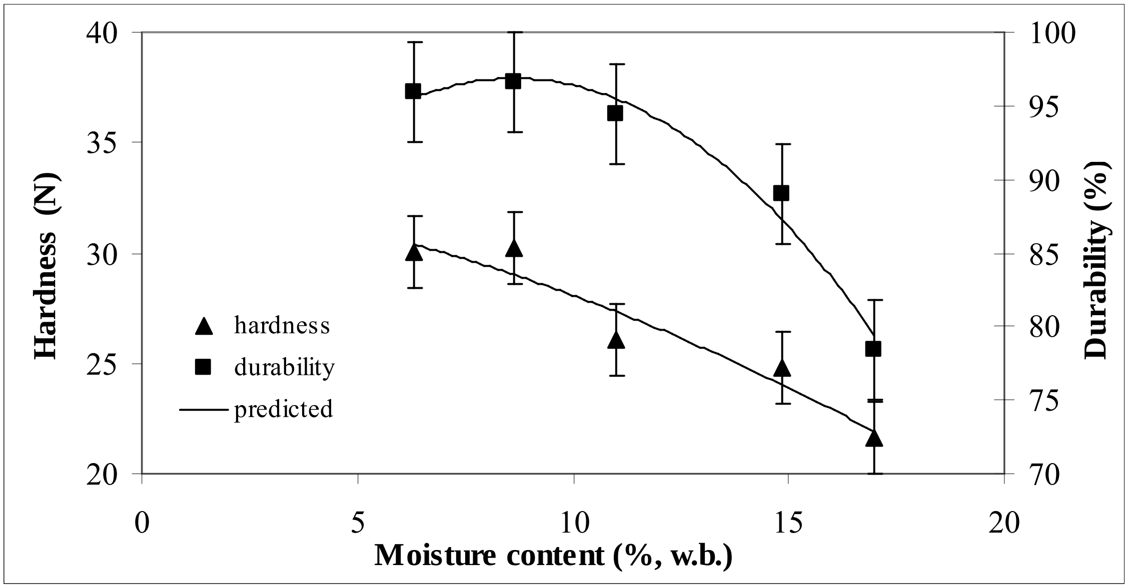
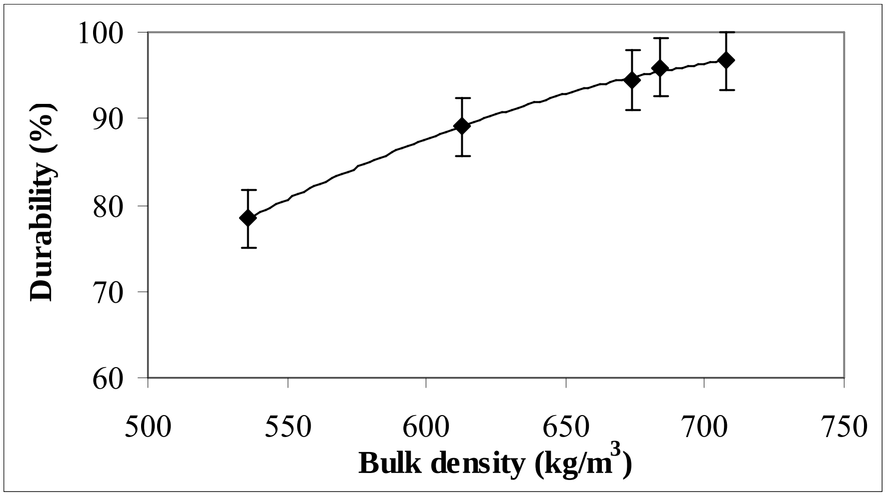


Figure 2.10 – Effect of moisture content on durability and hardness of switchgrass pellets



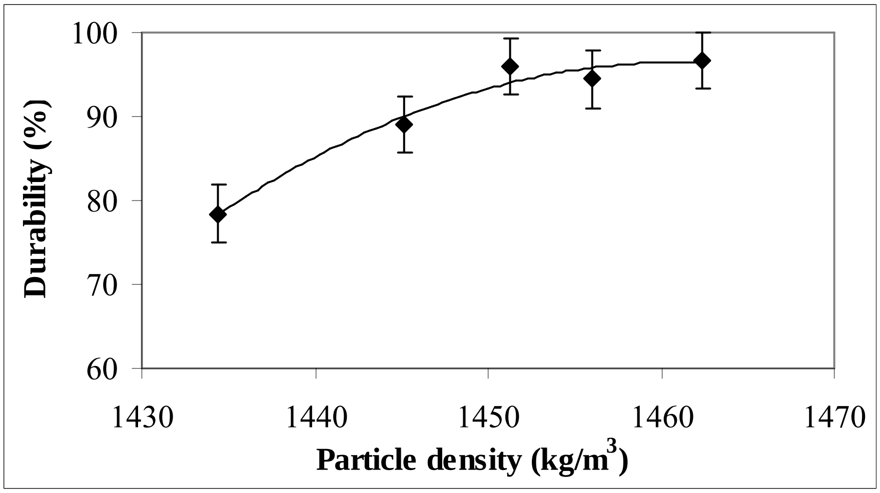


Figure 2.11 – Relationship between durability and particle and bulk density of switchgrass pellets

Moisture Sorption Rate

The typical effect of air temperature and relative humidity on rate of moisture absorption by the pellets is shown in Figures 2.12 and 2.13. In general, rate of moisture absorption varied with relative humidity and temperature of the air to which the pellets were exposed. A 15% increase in relative humidity (from 65 to 80%) at constant temperature of 15°C gave an increase of 3.25% in final moisture content. This was an indication that increased moisture in the air can lead to a substantial increase in the final moisture content of the pellets. The increase in final moisture content with increase in relative humidity is also displayed in Figure 2.12.

At 50% relative humidity, the final moisture contents of the pellets at temperatures of 15, 25 and 35°C were 7.84, 7.92 and 7.94%, respectively. However, at 45°C, the final moisture content was much lower than that obtained at other temperatures (Figure 2.13). It is postulated that the elevated temperature (45°C) is causing the moisture to evaporate near the surface of the pellets before it can be absorbed.

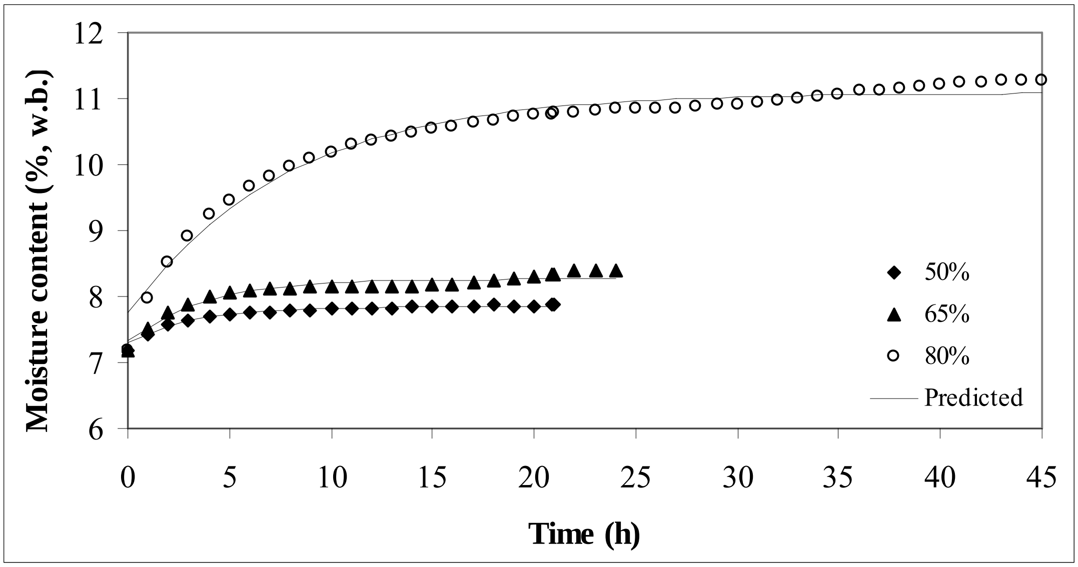


Figure 2.12 – Moisture change in switchgrass pellets exposed to air at 15°C and relative humidity of 50, 65, and 80%. Initial moisture content was 7.19% (w.b.).

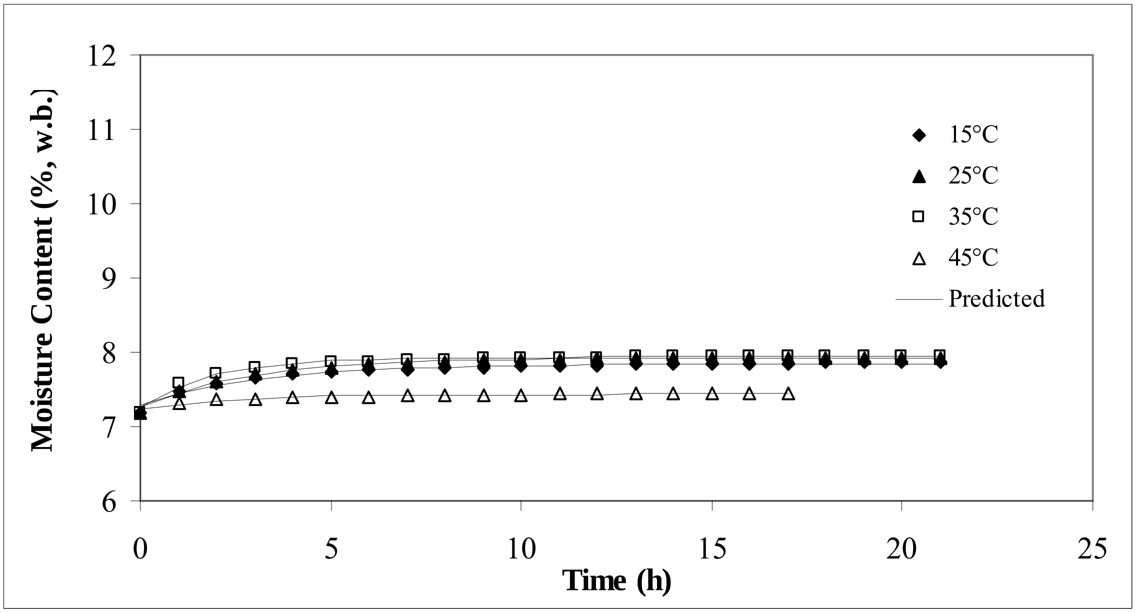


Figure 2.13- Moisture change in switchgrass pellets exposed to air at 50% relative humidity at temperatures of 15, 25, 35, and 45°C. Initial moisture content was 7.19% (w.b.)

The non-linear estimation procedure (NLIN) in SAS 9.1 Statistical package (2002) was used to fit an exponential model (Equation 2.21) to the moisture sorption data. The estimated values for moisture sorption rate constant (k) and equilibrium moisture content (M_q) are given in Table 2.3. Equation 2.21 is one of the simplest models that has been used to describe moisture sorption in biological materials (Fasina and Sokhansanj, 1992). According to Chhinnan, (1984) this model assumes negligible internal resistance which means there is no resistance to moisture movement from within the material to the surface. It only considers the surface resistance implying all the resistance is concentrated in a layer at the surface of the material.

$$\frac{M - M_q}{M_i - M_q} = \exp(-kt) \qquad (2.21)$$

Where, t = time (min)

M = instantaneous moisture content (%, w.b.)

M_i = initial moisture content (%, w.b)

Table 2.3- Estimated values of moisture sorption rate constant (k) and equilibrium moisture content (M_q) obtained from non-linear regression analysis using Equation 2.21 for switchgrass pellets

RH (%)	Temperature (°C)	k (h^{-1})	M_q (%, w.b.)
50	15	0.33	7.84
	25	0.35	7.92
	35	0.52	7.94
	45	0.42	7.44
65	15	0.28	8.26
	25	0.23	8.83
	35	0.24	9.02
	45	0.38	8.70
80	15	0.13	11.09
	25	0.18	11.18
	35	0.20	10.90

The values of the coefficient of determination, R^2, were between 0.893 and 0.994 and values of standard error were between 0.0025 and 0.00574, which showed that Equation 2.21 adequately predicted the experimental data. Further statistical analysis indicated the estimated values of k and M_q were significantly affected ($P < 0.05$) by relative humidity and not by temperature (Table 2.2). The k value was higher at lower relative humidity and higher with increasing temperature. Other researchers reported the dependency of k and M_q on relative humidity with other biological materials (Zink, 1935;

Dexter et al., 1947; Dadgar et al., 2004; McMullen et al., 2005). At higher relative humidity values, the time for pellets to equilibrate was longer. The pellets gained up to 4 percentage points of moisture at high humidity. Data for other temperature and relative humidity combinations can be found in Appendix A, Figures A.1-A.4.

Moisture Sorption Isotherms

The EMC-ERH curves were sigmoidal (Type II) in shape which is typical of equilibrium moisture content data for biological materials (Labuza 1984; Erbas et al., 2005). Generally, the relative humidity at given moisture content increased with increasing temperature until RH = 0.72 and M_e = 11%. After this point, the effect of temperature was insignificant (Figure 2.14). According to van den Berg and Bruin (1984), the isotherm curve can be divided into three regions. The first region has a range of relative humidity from 0 to 0.2 with an enthalpy of vaporization much larger than pure water. In this region the first water molecules are sorbed at the active polar sites. Consequently, the molecules are tightly bound and behave as part of the solid. In the second region with a relative humidity range of 0.23 to 0.73 the water is less tightly bound and the enthalpy of vaporization is closer to that of pure water. The water molecules sorb near to or on top of the first molecules and penetrate into newly formed holes of the swollen structure. Depending on the solids present this water enhances chemical and biochemical reactions. In the third region, with relative humidity range of 0.74-1.0, there is free water, which is mechanically trapped in the void spaces of the solid and has properties similar to those of bulk water. It is in this third region that the overlapping of isotherms is observed in this study. The isotherms show temperature has little or no effect on equilibrium moisture content at high relative humidity. Temple and van Boxtel (1999) reported similar results with black tea leaves. It can be noted that materials that follow a Type II isotherm hold less water at higher temperatures. In general the effect of temperature on increasing the relative humidity at constant moisture content is greater at low to intermediate relative humidity (Labuza, 1984). As seen in Figure 2.14, relative humidity increases as temperature increases for a given equilibrium moisture content. This can make the pellets susceptible to microbial growth.

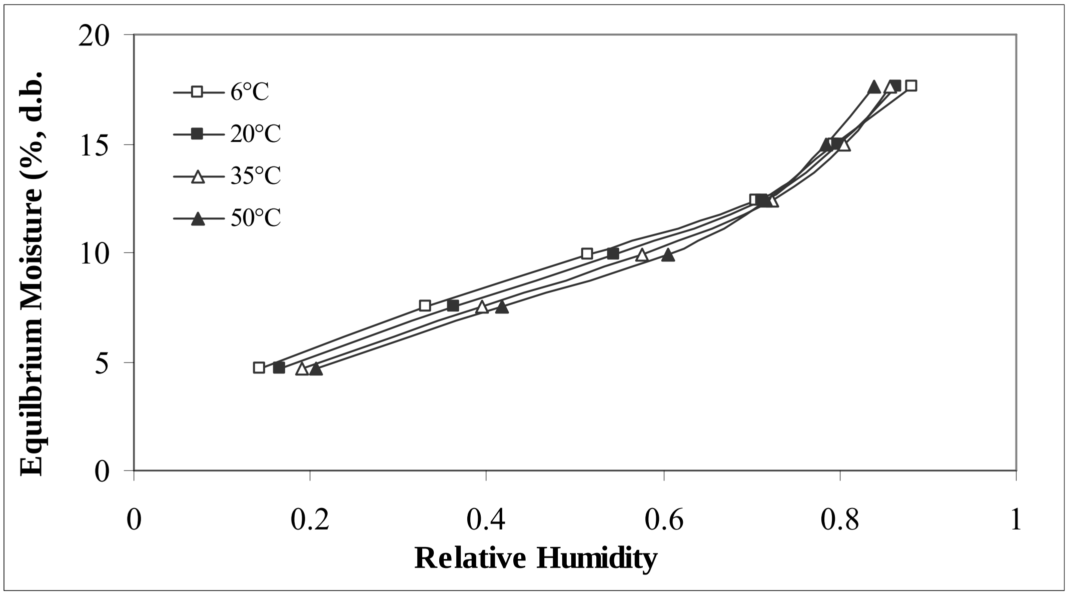


Figure 2.14 – Moisture sorption isotherms for switchgrass pellets at 6, 20, 35, and 50°C

The EMC and ERH data were analyzed using the following four equations (Fasina and Sokhansanj, 1993):

The Halsey equation:

$$RH = \exp\left[-\exp(a + bt)M^{-c}\right] \tag{2.22}$$

The Modified Henderson equation: $RH = \left[\left(\frac{a + bt}{M} \right)^{c} + 1 \right]^{-1}$ (2.23)

The Chung-Pfost equation: $RH = \exp\left[\frac{-a}{t + b} \exp(-cM) \right]$ (2.24)

The Oswin equation: $RH = 1 - \exp\left[-a(t + b)M^{c} \right]$ (2.25)

Where, RH is the relative humidity in decimal, M is the equilibrium moisture content on a dry basis (which is a typical way or reporting isotherm data) and a, b, and c are model coefficients. The coefficients of the equations were estimated using the non-linear regression procedure (NLIN) in SAS package 9.1. The goodness of fit for each model was quantified using the coefficient of determination (R^2), the standard error of the estimate (SEE) and the mean relative deviation (MRD). Several authors have reported the benefit of using multiple statistical parameters to select the model that gives the best fit to experimental data (Fasina and Sokhansanj, 1993; Barker, 1996; Soysal and Oztekin, 2001; Erbas et al., 2005, Durakova et al., 2005; Pagano and Masheroni, 2005).

The standard error of estimate (SEE) is the conditional standard deviation of the dependent variable and is defined as follows:

$$SEE = \sqrt{\frac{\sum_{i=1}^{m} \left(M_e - \hat{M}_e \right)^2}{df}} \tag{2.26}$$

Where, M_e is the measure value, $\hat{M}_e$ is the estimated value, *m* is the number of data points and '*df*' is the degrees of freedom for the model. The mean relative deviation

(MRD) is an absolute value used because it gives a clear picture of the mean divergence of the estimated data from the measured data and is defined as follows:

$$MRD = \frac{1}{m}\sum_{i=1}^{m}\left(\frac{\left|M_e - \hat{M}_e\right|}{M_e}\right) \tag{2.27}$$

In general, when evaluating the goodness of fit of a model, low values of R^2 and high values of SEE and MRD mean the model is unable to accurately describe the variation in experimental data. The model coefficients and statistics of fitting for a temperature range of 6-50°C are listed in Table 2.4. It can be observed that the Halsey equation gave the highest values for MRD and SEE and the lowest R^2 value suggesting that it is not the most appropriate model to describe the experimental data (Figure 2.15 (a)). The Chung-Pfost model gave the lowest MRD, and SEE and the highest R^2 value leading to the appearance of the most suitable model (Figure 2.15 (c)), which follows the conclusion by Soysal and Oztekin (2001) that the Chung-Pfost equation is suitable for cereal and other field crops. However, is has been noted that isotherm equations that gave values of MRD<0.05 have been considered to be a good fit (Pagano and Mascheroni, 2005). All of the equations except the Halsey equation had MRD values less than 0.05.

Table 2.4 – Model coefficients and values for the mean relative deviation (MRD) standard error of estimate (SEE) and coefficient of determination (R^2) for temperature range of 6-50 °C

	Coefficients			**Statistics of fitting**		
Equation	**a**	**b**	**c**	**MRD**	**SEE**	**R^2**
Halsey	3.6422	-0.0040	1.8172	0.1279	0.1737	0.9789
Stdev	± 0.1785	±0.00176	± 0.0760			
M. Henderson	3.70E-05	34724.0	1.7618	0.0484	0.0744	0.9882
Stdev	± 0.000017	± 163.5	± 0.0572			
Chung-Pfost	1083.9	218.60	0.2021	0.0266	0.0553	0.9935
Stdev	± 256.8	± 57.62	±0.00476			
Oswin	9.3788	-0.0169	2.6285	0.0387	0.0569	0.9931
Stdev	± 0.1570	± 0.00473	± 0.0673			

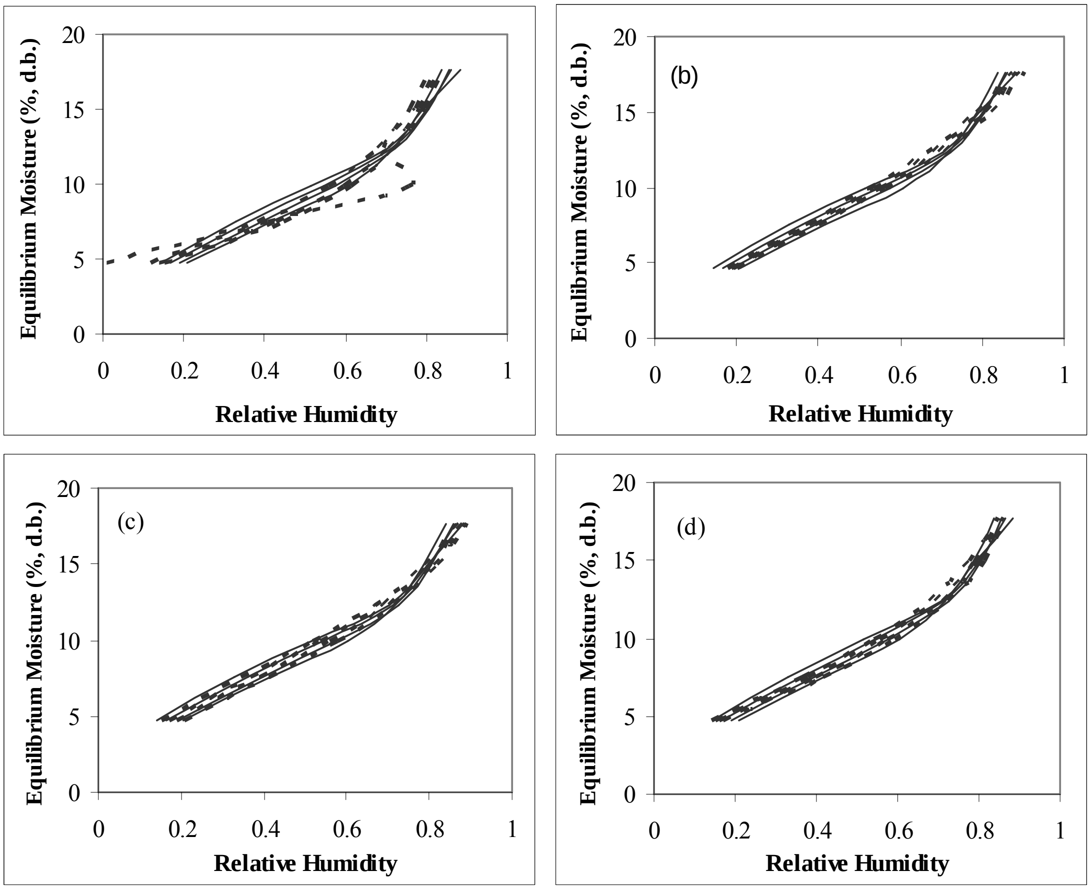


Figure 2.15 – EMC/ERH data fitted to four isotherm models: (a) Halsey equation (b) Modified Henderson equation (c) Chung-Pfost equation (d) Oswin equation. (—, experimental; ···, model)

CONCLUSION

It can be concluded from this study that moisture content significantly affects physical properties of pellets manufactured from switchgrass. Increasing moisture content increased the diameter of the pellets by 8% and decreased the length by 17%. Bulk and particle densities decreased by 24 and 16% respectively as moisture content of the pellets increased. A maximum durability rating of 95.91% was obtained when the pellets were at a moisture content of 8.62% (wet basis). Pellets also displayed high to medium durability in the moisture range evaluated. Durability and hardness decreased as a result of increasing moisture. The force required to rupture the pellets ranged from 20.60 to 30.21N. There was a maximum or minimum value of all properties at 8.62% moisture content. This moisture level could possibly be considered an optimum moisture content for switchgrass pellets upon further investigation.

Results from compositional analysis revealed that proportion values of individual components for the pelleted sample were higher than those of ground sample. However, statistically there were no differences in carbohydrate levels, but there were significant differences in ash, acid soluble lignin and acid insoluble lignin at a 95% confidence interval. There is no scientific explanation for the increase in proportion of components of the sample due to pelleting

Relative humidity had a significant effect on the moisture uptake rate constant (k) and equilibrium moisture content (M_q). At lower relative humidity, k was higher. The pellets also gained 4% in moisture at high relative humidity. The moisture sorption isotherms showed independence to temperature beyond the relative humidity of 0.72. For a temperature range between 6 and 50°C the Chung-Pfost equation was the most appropriate fit to experimental data where the Halsey equation was the least appropriate.

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CHAPTER 3 – COMPACTION BEHAVIOR OF SWITCHGRASS

INTRODUCTION

Biomass materials are renewable sources of energy. They are derived from living plants, animal manures, waste products from processing industries and other biological sources (Mani et al., 2003a). Due to the increasing cost of energy from fossil fuel and concerns about environmental pollution from fossil fuel utilization and to secure our nation's food supply, there is an urgent need to obtain energy from renewable resources such as biomass. Similar to other biological materials, low bulk density is a characteristic that makes handling difficult, and storage and transportation very expensive (Mani et al., 2005). Compaction is the most common method that has been employed to increase the bulk density thereby creating simpler handling and reduced transportation and storage costs (Smith et al., 1977; Mani et al., 2003b; Mani et al, 2005).

Compaction (or densification) involves the application of mechanical pressure to reduce the volume of biological material. Extrusion and briquetting are the most common commercially employed methods. Extrusion processes use pistons, screws, or rollers to force the material through one or more constrictive dies thus resulting in the production of pellets, cubes, or wafers (Bruhn, 1989). Extrusion processes consume high amounts of energy due to the excessive frictional resistance. This resistance causes the system to require a high power input which generates elevated temperatures in the pelleted materials (Faborode, 1990). Materials that are compacted into pellets are usually denser than other compacted forms (i.e. briquettes, cubes and wafers). Pelleted materials are therefore preferred when there will be significant handling and transportation of the compacted material. In order to manufacture high quality pellets, knowledge of compaction behavior is very important. Converting biomass to pellets or briquettes is greatly influenced by processing parameters such as moisture content, temperature and die size. Compaction studies can aid in optimizing processing parameters that will result in the production of high quality pellets at pilot and industrial scales.

Smith et al (1977) reported that an increase in temperature led to a greater degree of compaction, and recovery length of the straw briquettes was less when applied temperatures were in the range of 90 to 140°C. Tabil and Sokhansanj (1996) reported that it was important for alfalfa grinds to reach above 90°C after passing the conditioning chamber to ensure high quality pellets. The adhesive properties of thermally softened lignin are thought to contribute significantly to the strength characteristics of briquettes made from lignocellulosic materials (Grenada et al., 2002).

Tabil and Sokhansanj (1996) found that an optimum die size of 6.2 mm is needed to pellet alfalfa grind with a moisture content of 7.5-9.0% moisture. They also found that a die size of 7.9 mm could be used to pellet alfalfa grinds with moisture content of up to 12.0%. This is because alfalfa grinds are likely to be gummy when hot and moist. The smaller diameter dies offer more resistance to the grind particles due to a lower surface area, hence an increase in density. In addition, during the pelleting of alfalfa, steam is added to aid the release and to activate natural binders and lubricants thereby increasing pellet durability and reducing energy consumption during pelleting (Tabil and Sokhansanj, 1996; Samson et al., 2000).

The energy required for compaction of biomass depends highly on pressure, moisture content, physical properties of the material and method of compaction. Most densification processes encompass two main components, the energy of compression, and the energy of extrusion. The compression energy results in bulk reduction and the extrusion energy is required to overcome the friction between the material and the die, and may account for roughly 60% of the total densification energy (Bruhn, 1989; Mani et al., 2004a). There have been reported efforts to pellet switchgrass but no parametric studies to investigate the die size, moisture content, or temperature during pelleting. The aim of this study was to quantify the effects of moisture content, temperature, and die size on the degree of compaction that can be obtained from switchgrass in a single pellet apparatus and the specific energy required to achieve this degree of compaction. Limited studies were also carried out on the effect of compaction force and particle size on degree of compaction.

MATERIALS AND METHODS

The switchgrass used in this study was obtained from the E.V. Smith Experiment Station (Auburn University) in Tallassee, Alabama. Before experimentation, the switchgrass was ground in a hammer mill (New Holland Grinder, Model 358, New Holland, PA) that was fitted with a screen size of 3.18 mm. The compression test was conducted using a single pellet apparatus that consists of a cylindrical jacketed die (diameter s of 4.8, 6.4, and 7.9 mm and length of 50 mm). Parameters that were investigated in the study include die size (4.8, 6.4, and 7.9 mm), moisture content (10.4, 13.2, 16.2 and 20.0%, wet basis.), and temperature (60, 75, and 90°C).

The moisture content of the ground switchgrass was adjusted to the desired moisture levels by putting samples in an environmental chamber (Model AA-5460A, Espec Corp., Hudsonville, MI) set to 30°C and 90% relative humidity. The compression apparatus was composed of a plunger and die assembly attached to a texture analyzer (Model TA-HD, Stable Micro Systems, Surrey, UK) (Figure 3.1). Water from a water circulator (Model 9512, Polyscience, Niles, IL) was used to heat the samples by flowing through the annular space of the jacketed die.

To conduct a test, a weighed amount of ground switchgrass was placed in the die and heated for 20 minutes to the desired temperature before compacting. The time duration of 20 minutes was verified by monitoring and recording the temperature reading from a thermocouple that was placed in the sample during the heating process. The plunger was then used to compress (at a speed of 1 mm/s) the sample in the die to a force load of 3924 N. Once this force was attained, the software provided by manufacturer of the Texture Analyzer was programmed to hold the sample at this force (stress relaxation) for a period of 1 minute (Figure 3.2). After this duration of time, the pellet was removed from the die. The weight (using a balance accurate to 0.001 g ,Model AR3130 Ohaus Corp, Pinebrook, NJ) and the dimensions (length and diameter using a digital caliper accurate to 0.01 mm , Model CD-56C, Mitutoyo Corp, Kawasaki, Japan) of the pellet were then obtained. During sample compression, force-deformation-time data from the Texture Analyzer were automatically displayed and recorded by the software.

The density of each pellet was computed from Equation 3.1 using the values of the measured mass and volume of the pellet obtained from the die. The area under the force-deformation curve was taken as the specific energy required for the compression of each pellet (Figure 3.3). Limited studies were also carried out on (a) compaction of switchgrass using the different die sizes (4.8, 6.4, and 7.9 mm) with the same amount of pressure (95 MPa) and (b) on effect of particle size on compaction using hammer mill screen sizes of 0.79, 1.58, and 3.18 mm.

$$\rho_{pellet} = \frac{m_{pellet}}{V_{pellet}} \tag{3.1}$$

Figure 3.1- Single pellet compaction test set up

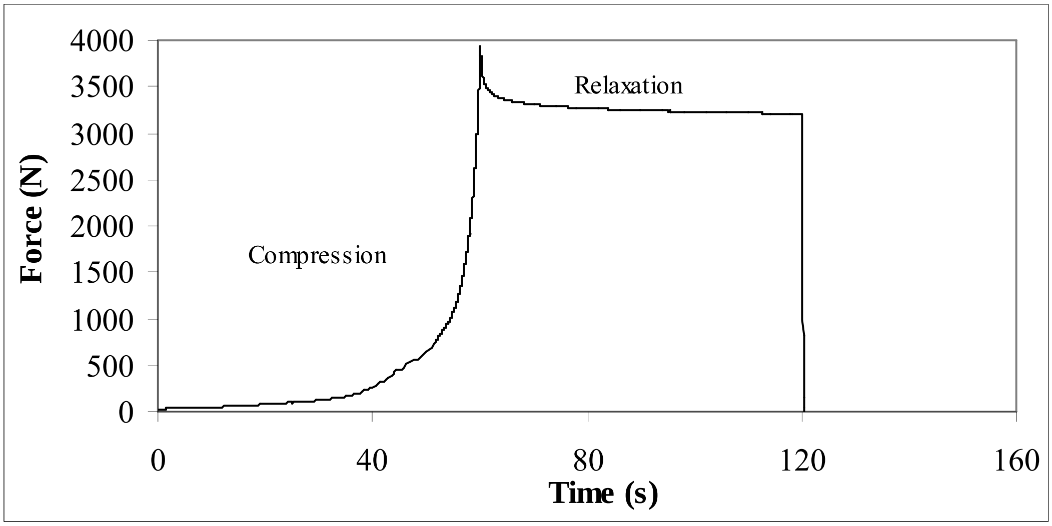

Figure 3.2 – Force-time curve for switchgrass made in single pellet apparatus (conditions: 4.8 mm, 60°C, 10.4%, w.b.)

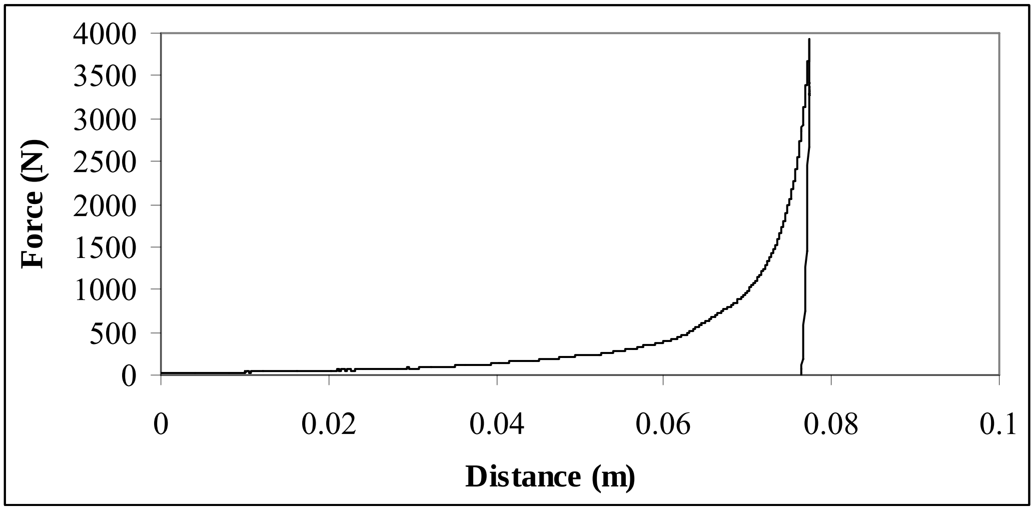

Figure 3.3 – Force-distance curve used to calculate energy required to compress the switchgrass (conditions:4.8mm, 60°C, 10.4% w.b.)

RESULTS AND DISCUSSION

Figure 3.4 shows the temperature data obtained from the thermocouple placed in the sample during the heating process. The figure shows that it took less than 5 minutes for the samples to reach each desired temperature. Increasing the temperature of the sample is crucial to the compaction process because switchgrass is a lignocellulosic biomass. Several studies on similar lignocellulosic biomass (e.g. alfalfa) have shown that the fibrous component of this biomass type has to be melted (achieved by increasing temperature) before significant compaction (or increase in density) and stability of the biomass can be achieved (Smith et al., 1977; O'Dogherty and Wheeler, 1984; Faborode 1990; Tabil and Sokhansanj, 1996; Samson et al., 2000).

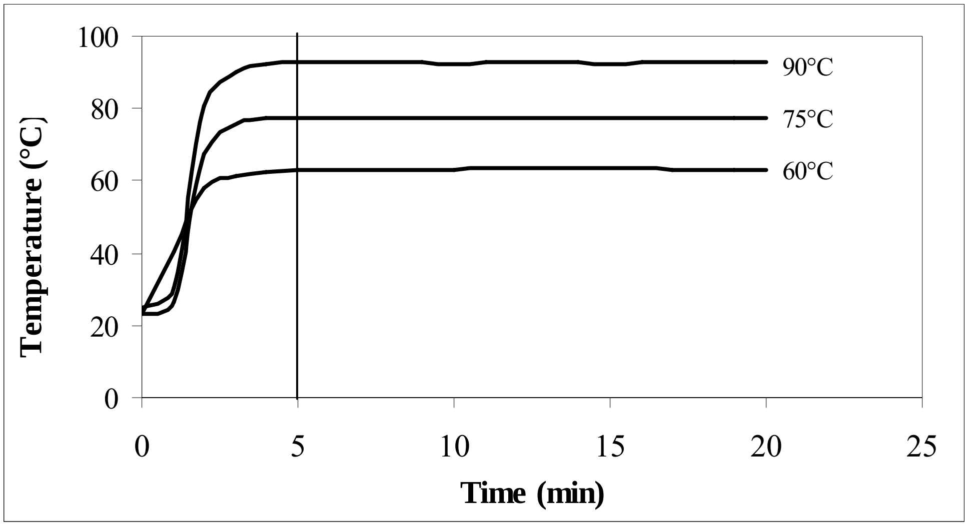

Figure 3.4 – Temperature profiles of switchgrass sample at 13.2% moisture content (w.b.) during conditioning

Density

The effect of die size, temperature and moisture content on the density of pellets obtained from the single-die apparatus is illustrated in Figure 3.5. Statistical analysis revealed that all the variables (die size, temperature and moisture content) significantly affected ($P < 0.05$) pellet density. There was however, no significant interaction between variables.

As expected, the density of the switchgrass pellets decreased as die size increased from 4.8 to 7.9 mm diameter regardless of the temperature and moisture content combination (Figure 3.5). This was primarily due to (a) increase in surface area (hence lower compaction pressure) as die size increased, and (b) greater folding and interlocking of the particles as die size decreased (Butler and McColly, 1959; O'Dogherty and Wheeler, 1984).

Density of the pellets increased within the temperature range of 60 to 90°C. It is suspected that this is due to the documented effects of temperature on the fibrous components of lignocellulosic biomass such as switchgrass. This is in agreement with Samson and Duxbury's (2000) findings which stated that between 75-85°C, the fiber within the cellulosic biomass begins to flow from the fiber cell wall and binds with other fibers during compression. Other researchers showed that heating a material between 60 and 70°C led to a more stable product than what was possible with unheated material. It was also determined that at higher temperatures, lower pressure was needed to provide a given degree of compaction for lucerne and Bermuda grass and that grass with relatively high moisture content could be stably compacted at elevated temperatures (Smith et al., 1977). O'Dogherty and Wheeler (1984) reported that increasing the temperature of straw and hay before compressing allows greater package density and durability to be achieved. Tabil and Sokhansanj (1996) found that it was important for alfalfa grinds to reach a temperature above 90°C during conditioning to ensure pellet quality and efficiency. It was shown (in the same study) that the conditioning temperature had a positive correlation with the durability of the manufactured alfalfa pellets where density is highly correlated with durability (see results in Chapter 2).

As moisture content increased, the density of the pellets decreased. For example when the moisture content of switchgrass samples increased from 10.4% to 20%, the density of pellets (made at 90°C and die size of 4.8 mm) decreased from 1215 kg/m^3 to 955.21 kg/m^3, respectively (Figure 3.4). Similar results were reported by for wheat straw briquettes (Smith et al., 1977), straw wafers (O'Dogherty and Wheeler, 1984) and corn stover briquettes (Mani et al., 2004b). The decrease in density is thought to be due to the inhibitory effect of the water molecules on particulate bonding during the compaction process. It is postulated that moisture present which cannot escape, by way of extrusion through vent holes and/or clearance between the compression chambers may limit the maximum dry matter density attainable, and may interfere with the performance of natural bonding agents (Bruhn, 1989). High moisture can also cause axial expansion of the pellets, thereby reducing density (Mani et al., 2005).

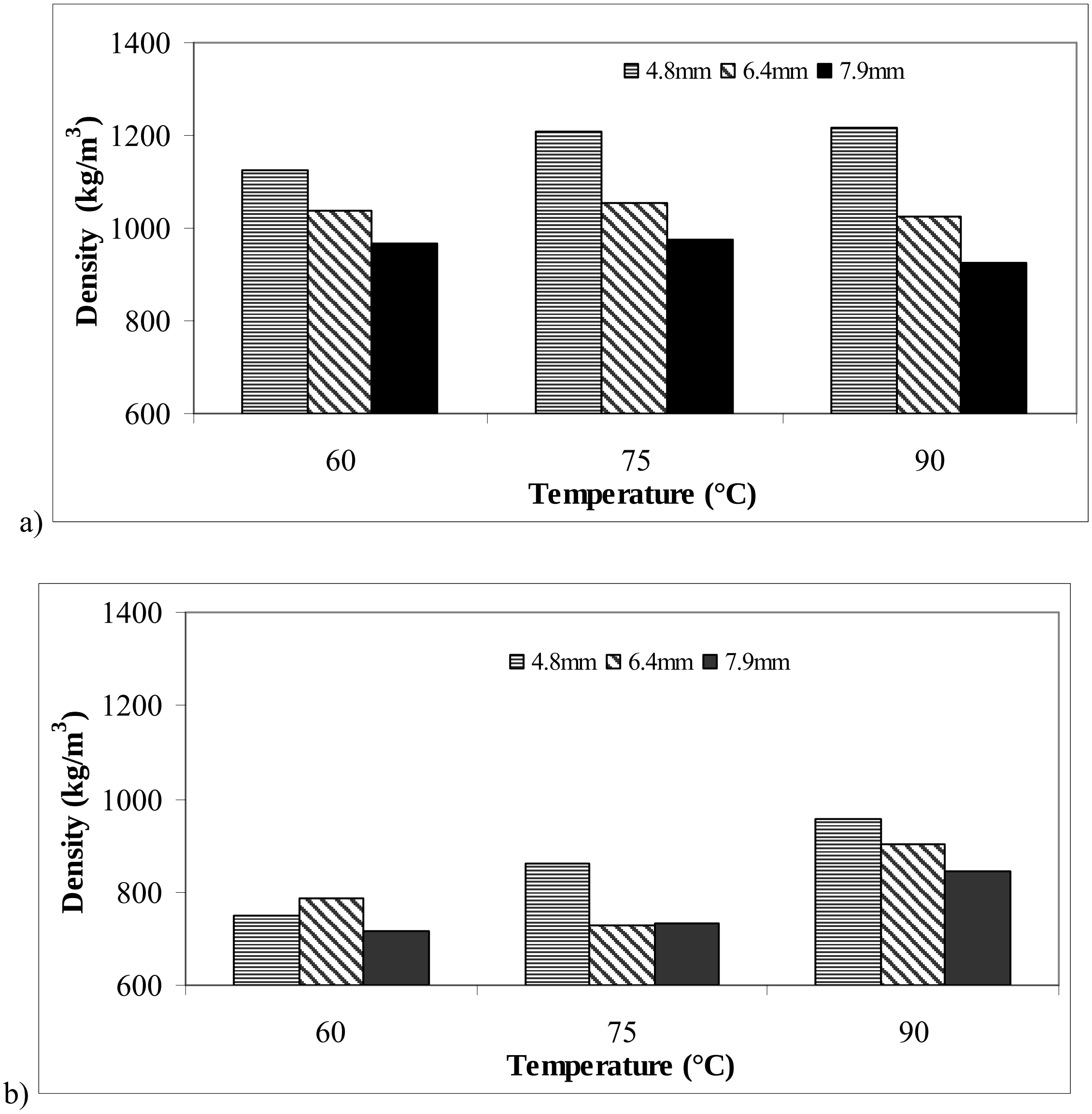

Figure 3.5 – Density of switchgrass pellets affected by temperature and die size at constant moisture contents of a) 10.4% and b) 20.0%

In this study, the maximum density attained when switchgrass was compacted to a force of 3924 N in a single pellet apparatus was 1214 kg/m^3 at 10.4% moisture content and 90°C. Mani et al (2004b) obtained a maximum corn stover briquette density of 950 kg/m^3 in the moisture range of 5-10% (wet basis). Other researchers found the optimal moisture content for lucerne and Bermuda grass to be between 16 and 23% (wet basis) (Smith et al., 1977). It is therefore obvious that the optimum moisture content required to produce high density compacts varies with the type of feedstock.

Effect of Pressure and Grind Size

Based on the studies that were carried out, it was determined that application of the same amount of pressure (95 MPa) to samples in different die sizes did not result in compacts of the same density. The density of the compacts (Figure 3.6) decreased with increase in die size thereby confirming that die size is as important as applied pressure when biological materials are compacted. Butler and McColly (1959) confirmed that at a given pressure the density of hay pellets were greater for smaller diameter chambers. Other studies reported the pressure required to form hay wafers (O'Dogherty and Wheeler, 1984) and palm fiber briquettes (Husain et al., 2002) of a given density increased exponentially with die diameter. It is stipulated that as the die becomes larger there is more relaxation of the particles resulting in less folding and interlocking of particles.

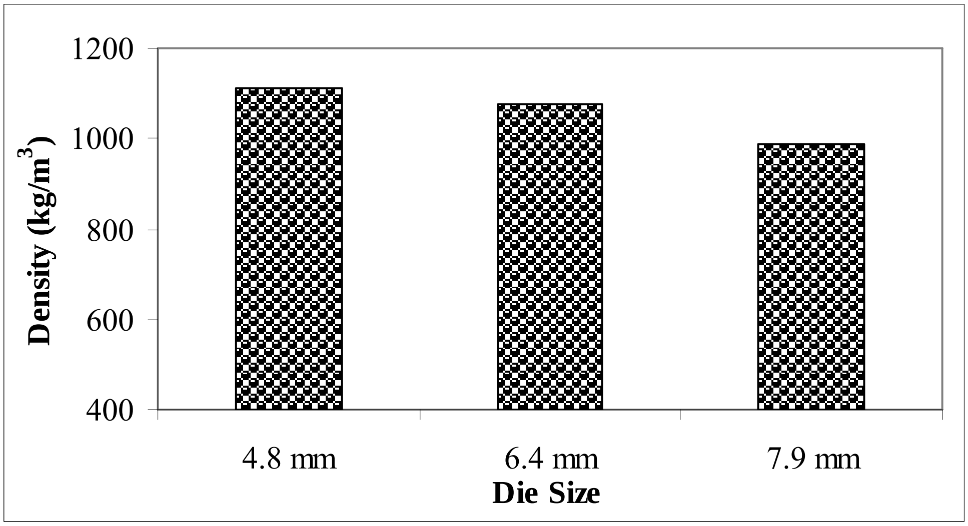

Figure 3.6 – Density of switchgrass pellets with constant applied pressure of 95 MPa, 10.4% moisture

Results from the study on effect of pellet density on grind size (Table 3.1) showed that the density of the compacts increased as the particle size decreased (Figure 3.7). Further statistical analysis (at 95% significance level) indicated that the density values from sample A (1274 kg/m^3) were not significantly different from that of sample B (1272 kg/m^3). Density values from sample C (1214 kg/m^3) were significantly lower that the density values of samples A and B. The samples used in this part of the study were obtained by using the hammer mill (referenced in Materials and Methods) to grind the samples through screen sizes of 0.79, 1.59, and 3.18 mm. The geometric mean diameter (d_{gw}) and standard deviation (S_{gw}) of the samples were obtained according to ASABE Standard S319.3 (2003) (Table 3.1).

Table 3.1 – Geometric mean diameter and standard deviation of the samples from the three hammer mill screen sizes

Sample	Screen size (mm)	d_{gw} (mm)	S_{gw} (mm)
A	0.79	0.191	0.140
B	1.59	0.231	0.127
C	3.18	0.860	0.370

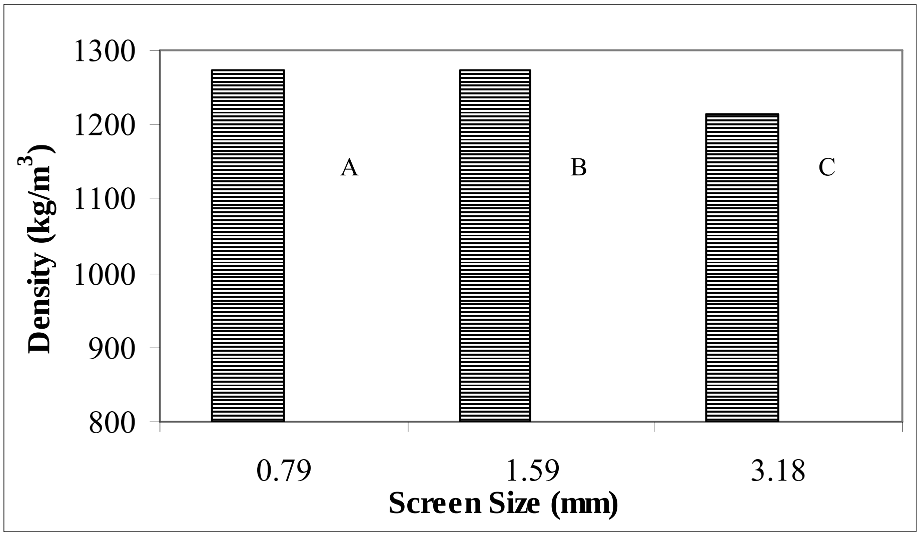

Figure 3.7 – Density of switchgrass pellets as particle size increase (conditions: 90°C, 4.8 mm)

The size of feed grind influences the final quality of densified masses. Similar results on the effect of grind size on pellet quality have been reported by other researchers (Lehtikangas 2001; Mani et al., 2003a; Adapa et al., 2004). Fine or medium ground feed constituents are desirable for pelleting because more surface area is available for moisture addition during steam conditioning (Tabil and Sokhansanj, 1996; Adapa et al., 2004). During compaction, smaller (fine) particles rearrange and fill in the void space of larger (coarse) particles producing denser and more durable compacts. Coarsely ground materials tend to give less durable pellets because they may create natural fissures in the pellet, which are susceptible to breakage. A proportion of fine and medium grind is essential, but if too little coarse material is present, quality and efficiency will suffer. It has been reported that grinding the biomass material to smaller particle size results in higher density briquettes with better durability, lower water absorption rates, and an increase in binding properties of the feedstock (Samson et al., 2000; Jannasch et al., 2001b; Lehtikangas, 2001). Mani et al (2003a) concluded that low quality alfalfa grinds from a 2.4 mm hammer mill screen had higher cohesion than the grind from a 3.2 mm screen. Also, particles with sizes below 0.400 mm are considered fine and highly compressible (Mani et al., 2003a).

Specific Energy

Statistical testing on the specific energy (Figure 3.7) required to compact switchgrass indicates that moisture and die size had significant effect on compaction energy while temperature was not significantly related to energy. The specific energy required for compression of switchgrass pellets was in the range of 19.33 - 88.81 MJ/t depending on test parameters. Tabil and Sokhansanj (1996) reported a specific energy required for alfalfa pellets of 106.92 MJ/t. Mani et al (2004b) reported specific energy for compression of corn stover briquettes in the range of 7.31-16.08 MJ/t. O'Dogherty and Wheeler (1984) reported specific energy required for straw wafers in the range of 5.5 - 21.3 MJ/t. The method of densification along with test parameters dictates the amount of energy required. Pellets are more compact than briquettes and require more energy to achieve the maximum density attainable.

Specific energy decreased by 66% as die size increased from 4.8 to 7.9 mm (Figure 3.8). Less energy was required to compress the switchgrass as the die size increased from 4.8 mm with a range of 88.81 – 44.34 MJ/t to 7.9 mm with a range of 29.26 – 19.33 MJ/t.

Specific energy required to compact samples in the 4.8 mm diameter die increased with moisture content. It is suspected that less space for relaxation gave rise to an increase of energy required to overcome inhibitory forces stemming from moisture gain. Smaller dies have been reported (O'Dogherty and Wheeler, 1984; Samson et al., 2000) to be able to handle moisture contents of <10% (wet basis) for lignocellulosic material. Anything above 10% can lead to erratic pelleting, less dense compacts, and more required energy. Similar results were reported with alfalfa hay pellets (Butler and McColly, 1959; Bellinger and McColly, 1961).

Unlike the result from the 4.8 mm diameter, specific energy required for samples densified in 6.4 and 7.9 mm diameter dies decreased as moisture content increased. This may be due to the increased surface area of the die, which leaves more available space for relaxation of the particles and less resistance to compression (Figure 3.8). For pellets made at 16.2% moisture content, results were very sporadic with no obvious trend.

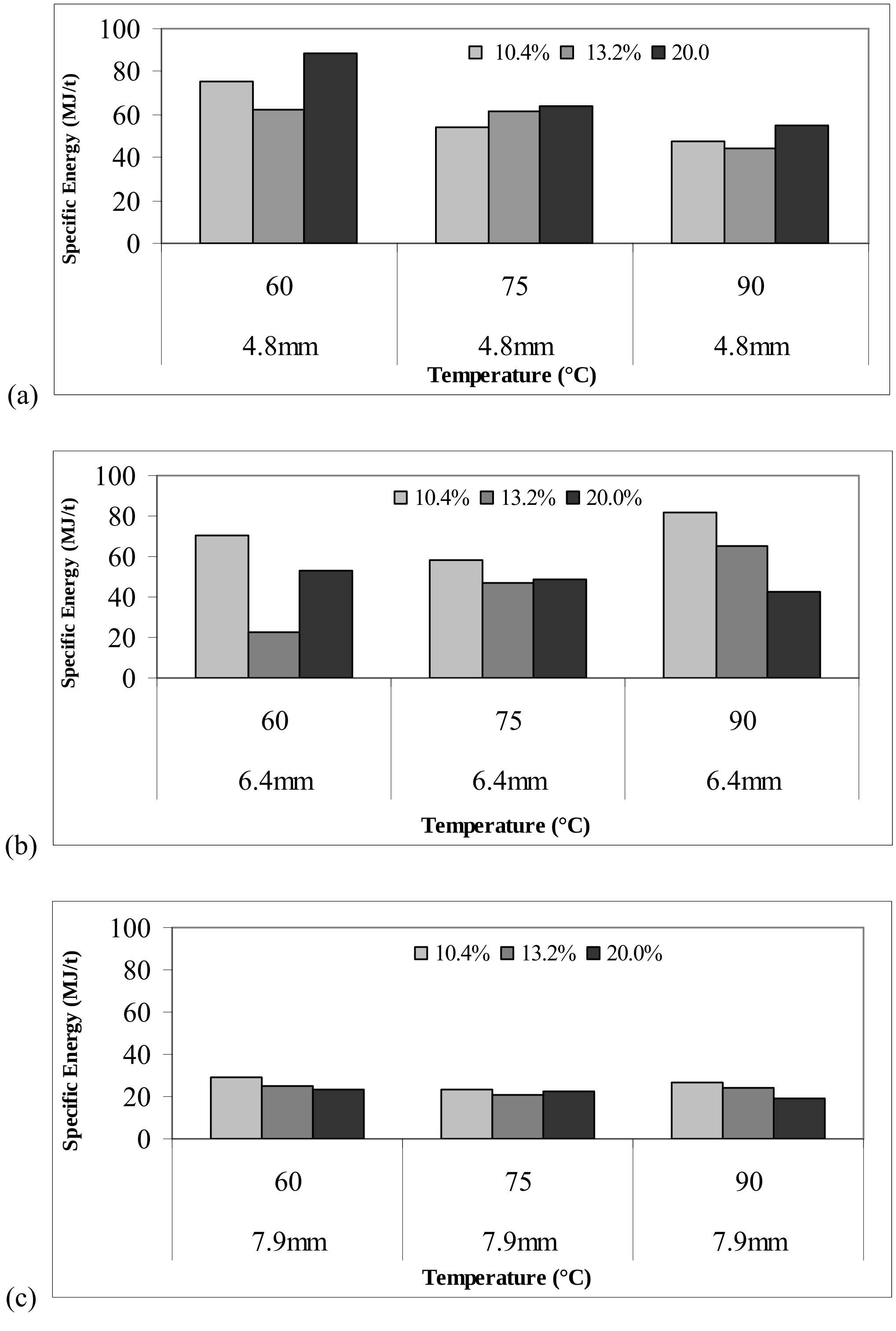

Figure 3.8 – Effect of temperature, moisture content and die size on specific energy required to make switchgrass pellets in a single pellet apparatus at constant die sizes of (a) 4.8 mm (b) 6.4mm and (c) 7.9 mm (mean values are reported)

CONCLUSION

A relationship between the density and specific energy required and processing parameters (die size, moisture content, and temperature) was established by compressing ground switchgrass using a single pellet apparatus. The density of the switchgrass compacts decreased with increasing die size resulting from the increase in surface area. There was less relaxation of the material as the die size became smaller, and therefore, a denser compact was formed in the smaller (4.8 mm) die. Application of the same amount of pressure (95 MPa) to samples in different die sizes does not result in compacts of the same density. In addition the density of the compacts increased as particle size deceased.

Specific energy required to compress the ground switchgrass decreased with increase in die diameter. For die diameter of 4.8 mm there was an increase in energy required with increasing moisture content. The opposite occurred with 6.4 and 7.9 mm die sizes where the specific energy required decreased as moisture content increased. This may be due to the increase in surface area which allows particles to relax and resistance to compression is lessened. Because results from 16.2% moisture content data were sporadic it is concluded that a moisture level of 16.2% should not be used for compaction of switchgrass.

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CHAPTER 4 – FUTURE WORK

COMPACTION BEHAVIOR

The pellets made in the single pellet apparatus have a propensity to expand once released from the die. Another way to measure density of the compacts before expansion is of interest. The density of the switchgrass pellets created in the single pellet unit will be calculated using the force-deformation data given as output by the texture analyzer. The density as the probe descends can be calculated by identifying the initial height of the sample in the die before compression begins.

At moisture contents below 20.0% there was difficulty in replicating the pellets at a given temperature and die size. The difficulty is thought to be due to the placement of the particles in the die. Because of the particle size, and size distribution, the biomass does not fill the die evenly which can lead to results that can not be easily replicated. Smaller particles will eliminate filling issues and according to the investigation on particle size in Chapter 3, will possibly lead to a more compact pellet. The effect of particle size on density and specific energy requirement should be studied on switchgrass ground with 0.79 and 1.59 mm hammer mill screen sizes.

SIMULTANEOUS SACCHARIFICATION AND FERMENTATION (SSF)

Large scale ethanol production from biomass can be used for alternative fuel, a fuel additive, or as chemical feed stocks (Seenayya et al., 2000). The use of ethanol as an alternative motor fuel has been increasing throughout the world (Badger, 2002) such as in the United States and Europe (Palmarola-Adrados, 2005). Ethanol has become important as an engine fuel, since it is easily blended with gasoline (Yu and Zhang, 2004). Ethanol can be synthetically made from petroleum or by microbial conversion of biomass materials though fermentation (Badger, 2002). The biomass materials used to produce ethanol fall into three major categories, sugar, starch and cellulosic biomass (Sen, 1989). For example, sugar cane and cane molasses are the basis of the Brazilian fuel ethanol industry (Wayman and Parekh, 1990). Starchy materials commonly used for ethanol production across the globe include, cereal grains (maize and wheat), potato, sweet potato, and cassava, which require hydrolysis to break down the starch into fermentable sugars (Badger, 2002). Currently wheat is being grown as an energy crop and the wheat starch is converted to ethanol in European plants (Palmarola-Adrados, 2005).

Because sugar and starch are in the human food chain, and are required for alternative uses they are expensive to use for ethanol production. Therefore, large scale production of ethanol will depend upon the use of a less expensive and renewable feedstock such as lignocellulosics (Seenayya et al., 2000; Badger, 2002).

Lignocellulosic materials used for ethanol production originate either as waste materials evolving from processes other than fuel production (agricultural and forest) or as energy crops grown for the purpose of fuel production (Lynd, 1996). Lignocellulosic biomass can provide a resource large enough to be considered as a renewable source of liquid transportation fuels (Alizadeh et al., 2005). Many researchers have demonstrated the ability to product ethanol from lignocellulosic biomass such as corn stover (Kim and Lee 2005b), used recycled paper sludge (RPS), (Lark et al., 1997) used sunflower hulls (Sharma et al., 2004) and switchgrass and poplar (Alizadeh et al., 2005; and Chung, et al., 2005).

In general the biomass - to - ethanol conversion process includes three main steps: 1) pretreatment to increase availability/reactivity of the substrate to the enzyme, 2) saccharification (acid or enzymatic) to hydrolyze the cellulose to simple sugars and 3) fermentation to convert the sugars produced during hydrolysis to ethanol (Chung et al., 2005). There are two ways to procure ethanol from lignocellulosics a) separate hydrolysis and fermentation (SHF) or b) simultaneous saccharification and fermentation (SSF). In the SSF process, enzymes and an ethanol producing organism (yeast) is used to carry out simultaneous hydrolysis of cellulose to glucose and the conversion of glucose to ethanol in the same reactor (Lark et al., 1997; Alizadeh et al., 2005). The advantages to the SSF process are: time for ethanol production decreases, less equipment is used, and it has been reported that less enzymes are required (Wyman and Parekh, 1990).

The goal for future work is to investigate the difference in ethanol yield between ground and pelleted switchgrass which is of high interest. All the principles and methods stated previously will allow the successful conversion of switchgrass to ethanol. As shown by the previous chapters, pelleting biomass (which has low bulk density) allows simpler handling and decreased transportation to the conversion facility and storage costs before material arrives at the conversion facility. Since the result of the compositional analysis between ground and pelleted switchgrass was similar (Chapter 2), it is postulated that the ethanol yield will be as well.

ENZYMATIC HYDROLYSIS

Enzymatic hydrolysis is commonly used as a method for breaking down cellulose and hemicellulose into monomeric sugars for fermentation purposes. Compared to acid hydrolysis, enzymatic hydrolysis is milder and more specific. Cellulase is used as the catalyst and is usually carried out at 40-50°C, reducing sugar degradation that occurs at high temperatures and to extend the life of the enzyme (Wyman and Parekh, 1990) and Sun and Cheng, 2004). Enzymatic hydrolysis also makes simultaneous saccharification and fermentation feasible making it an extensively studied process for fuel ethanol production (Sun and Cheng, 2004). Initial studies using ground and pelleted switchgrass have already been carried out by the author. Due to equipment malfunction final results were not obtained. Because this procedure is necessary for successful conversion of switchgrass to ethanol this will be included in future work.

PRETREATMENT

Pretreatment methods are an important aspect of the bioconversion process. The difficulty in enzymatically hydrolyzing lignocellulosic biomass arises from the complex nature of the material. Cellulose fibrils are embedded in a matrix of lignin and hemicellulose which make the plant tissue resistant to enzymes. Therefore, efficient bioconversion of lignocellulotic feedstock requires pretreatment (Saddler et al., 1993; Dale et al., 1996; Szczodrak and Fiedurek, 1996; Esteghlalian et al., 1997; Soderstrom et al., 2003; Liu and Wyman, 2005; Alizadeh et al., 2005; Kim and Lee, 2005a; Kim and Lee, 2006). Pretreatment methods for lignocellulosic biomass increase the specific surface area of the substrate, increase pore volume of the substrate, breakdown the cyrstallinity of the cellulose, and breakdown the lignocellulosic complex, to allow higher yields of fermentable sugars for ethanol production (Saddler, 1993; Dale et al., 1996; Szczodrak and Fiedurek, 1996; Esteghlalian et al., 1997; Kurakake et, al., 2001; Kim and Lee, 2006).

Pretreatment effectiveness has been associated with removal of hemicellulose and lignin. Lignin solubilization is beneficial for enzymatic hydrolysis, but the benefits have to be compared with the potential for fermentation inhibition by higher concentration of soluble lignin derivatives. If the lignin does not emerge from the pretreatment stage in the soluble form, it should be chemically modified. Since lignin melts at elevated temperatures (>90°C wet, >160°C dry), researchers have noted that lignin changes upon cooling and does not return to its original form (Lynd, 1996). In developing more effective pretreatments, Cowling and Kirk (1976) suggested placing an emphasis on biological, physical and chemical methods which can alter cyrstallinity and delignify the material which can in turn increase the available surface area of the substrate to the enzyme. Other researchers have a contrasting view of altering or decreasing the cyrstallinity where studies have shown examples of effective pretreatments that resulted in unchanged or increased cyrstallinity (Lynd, 1996).

The cost of the cellulase enzyme had been a concern in the recent years, therefore the most economical and effective pretreatment is desired to decrease enzyme loading (Alizadeh et al., 2005). Industrial economical pretreatment methods include utilizing acid and alkaline reagents (Kurakake et al., 2001). Typically, hydrolysis yields without pretreatment are < 20% of theoretical yields, where yields after pretreatment can exceed 90% of theoretical yields (Lynd, 1996).

Initial study on pretreatment methods have already taken place with ground and pelleted switchgrass. Further investigation of pretreatment methods (chemical and mechanical) should take place. For chemical pretreatments, treatment with hot water and the ammonia recycle percolation (ARP) process could enable increased digestibility of the switchgrass. This is because flow through methods allow lignin and hemicellulose to be removed from the system on a continual basis, which can prevent precipitation of lignin upon cooling and reactions with other components which are present (Kim and Lee, 2006). Also, residence times and temperatures for the soaking with aqueous ammonia (SAA) process should be varied. The switchgrass used in the current study was ground through a hammer mill with a screen size of 3.18 mm. The size of the particles were somewhat large (d_{gw} = 0.865 mm). Reduction of particle size disrupts the crystalline structure and breaks the chemical bonds of the long chain molecules increasing enzyme substrate contact during hydrolysis (Saddler et al., 1987). According to Mani et al (2004a) narrow range particle size distribution with more fines is suitable for enymatic hydrolysis of lignocellulosic materials because of the generation of more surface area and pore spaces during fine grinding. The draw back is that fine grinding of biomass requires high energy consumption. Ladich (1989) reports that grinding, milling and shearing biomass proves to be an effective pretreatment for enzymatic hydrolysis.

Therefore, future research objectives include:

1. Determine how the density values of switchgrass compacts created in a single pellet apparatus will differ with an alternative calculation method,
2. Determine how the density of switchgrass compacts created in a single pellet apparatus will be affected by change in grind size for all temperature, moisture, and die size combinations,
3. Determine the optimal pretreatment methods and conditions (chemical and mechanical) for switchgrass and
4. Investigate the difference in ethanol yield between ground and pelleted switchgrass by using SSF.

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CONCLUSION

The quality of pellets obtained from a pellet mill are affected by processing parameters such as moisture content, temperature, particle size, and pellet mill speed. It is important to study the effects of these parameters to provide data for the design of biomass handling and processing facilities. The goal of this investigation was to study the compaction behavior of switchgrass. The following specific objectives have been covered throughout the course of this paper :

1. Quantify the effect of moisture content on the physical properties of compacted (pelleted) switchgrass;
2. Evaluate the effect of process parameters on the compaction behavior (density and specific energy) of switchgrass and,
3. Evaluate the effect of pelleting on the composition of switchgrass.

It can be concluded from this study that moisture content significantly affected the physical properties of pellets manufactured from switchgrass. Increasing moisture content increased the diameter of the pellets by 8% and decreased the length by 17%. Bulk and particle densities decreased by 24 and 16% respectively as moisture content of the pellets increased. A maximum durability rating of 95.91% was obtained when the pellets were at a moisture content of 8.62% (wet basis). Pellets also displayed high to medium durability in the moisture range evaluated. Durability and hardness decreased as a result of increasing moisture. The force required to rupture the pellets ranged from 20.60 to 30.21N. There was a maximum or minimum value of all properties at 8.62%

moisture content. Relative humidity had a significant effect on the moisture uptake rate constant (k) and equilibrium moisture content (M_q). At lower relative humidity, k was higher. The moisture sorption isotherms showed independence to temperature beyond the relative humidity of 0.72. For a temperature range between 6 and 50°C the Chung-Pfost equation was the most appropriate model that fit to the experimental data where the Halsey equation was the least appropriate.

A relationship between the density and specific energy required and processing parameters (die size, moisture content, and temperature) was established by compressing ground switchgrass using a single pellet apparatus. The density of the switchgrass compacts decreased with increasing die size resulting from the increase in surface area. There was less relaxation of the material as the die size became smaller, and therefore, a denser compact was formed. Application of the same amount of pressure (95 MPa) to samples in different die sizes does not result in compacts of the same density. In addition the density of the compacts increased as particle size deceased the specific energy required to compress the ground switchgrass decreased with increases in die diameter. For die diameter of 4.8 mm the energy required increased with increasing moisture. The opposite occurred with 6.4 and 7.9 mm die sizes where the specific energy required decreased as moisture content increased. This may be due to the increase in surface area which allows particles to relax more and resistance to compression is lessened.

Results from compositional analysis revealed that proportions of individual components for the pelleted samples were higher than those of the ground samples. There were significant differences in ash, acid soluble lignin and acid insoluble lignin and no significant differences in carbohydrates at a 95% significance level. There is no scientific explanation for the increase proportions of individual components of the samples due to pelleting.

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APPENDICES

APPENDIX A

Table A.1 – Physical properties of switchgrass pellets (sample sizes are in parentheses).

Moisture Content (%, w.b.)	Diameter (mm) (50)	Stdev (mm)
6.32	4.85	0.05
8.62	4.84	0.06
11.00	4.90	0.07
14.84	5.02	0.11
17.00	5.25	0.20
Moisture Content (%, w.b.)	**Length (mm) (50)**	**Stdev (mm)**
6.32	33.61	3.28
8.62	35.27	3.91
11.00	32.80	3.46
14.84	32.35	3.80
17.00	27.97	4.03
Moisture Content (%, w.b.)	**Particle Density (kg/m^3) (3)**	**Stdev (kg/m^3)**
6.32	1451.27	6.97
8.62	1462.37	1.07
11.00	1455.99	5.07
14.84	1445.13	5.80
17.00	1434.38	2.86
Moisture Content (%, w.b.)	**Bulk Density (kg/m^3) (2)**	**Stdev (kg/m^3)**
6.32	683.61	0.39
8.62	707.62	0.76
11.00	673.79	6.03
14.84	612.45	2.29
17.00	536.21	4.57
Moisture Content (%, w.b.)	**Durability (%) (3)**	**Stdev (%)**
6.32	95.91	0.31
8.62	96.65	0.43
11.00	94.43	0.19
14.84	89.06	0.53
17.00	78.44	0.67
Moisture Content (%, w.b.)	**Hardness (N) (50)**	**Stdev (N)**
6.32	30.04	12.21
8.62	30.21	9.36
11.00	26.03	9.58
14.84	24.79	10.13
17.00	21.60	7.84

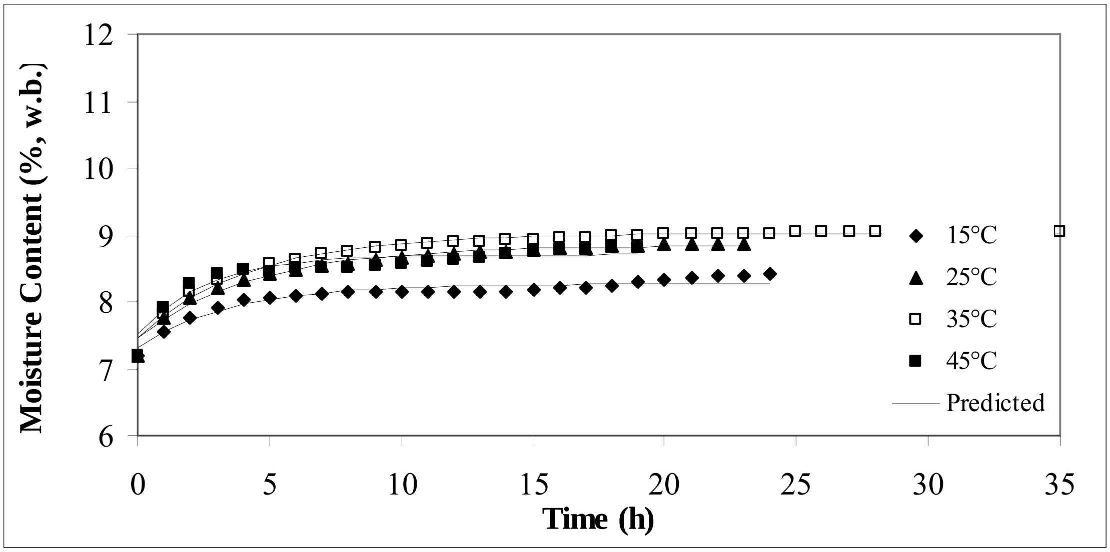

Figure A.1- Moisture change in switchgrass pellets exposed to air at 65% relative humidity with varying temperatures (15, 25, 35, and 45°C).
Initial moisture content was 7.19% (w.b.).

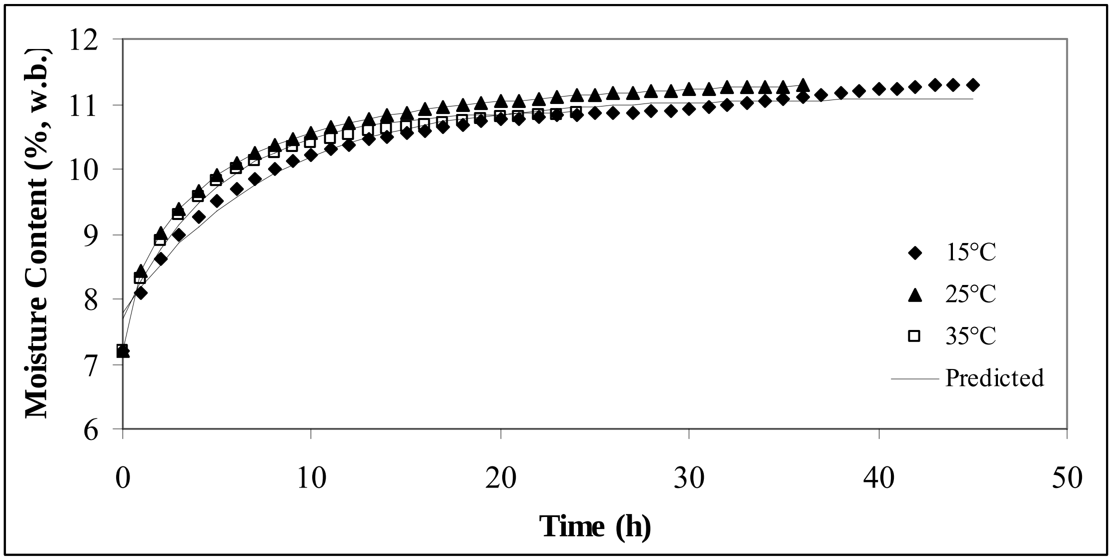

Figure A.2 – Moisture change in switchgrass pellets exposed to air at 80% relative humidity with varying temperatures
(15, 25 and 35°C).

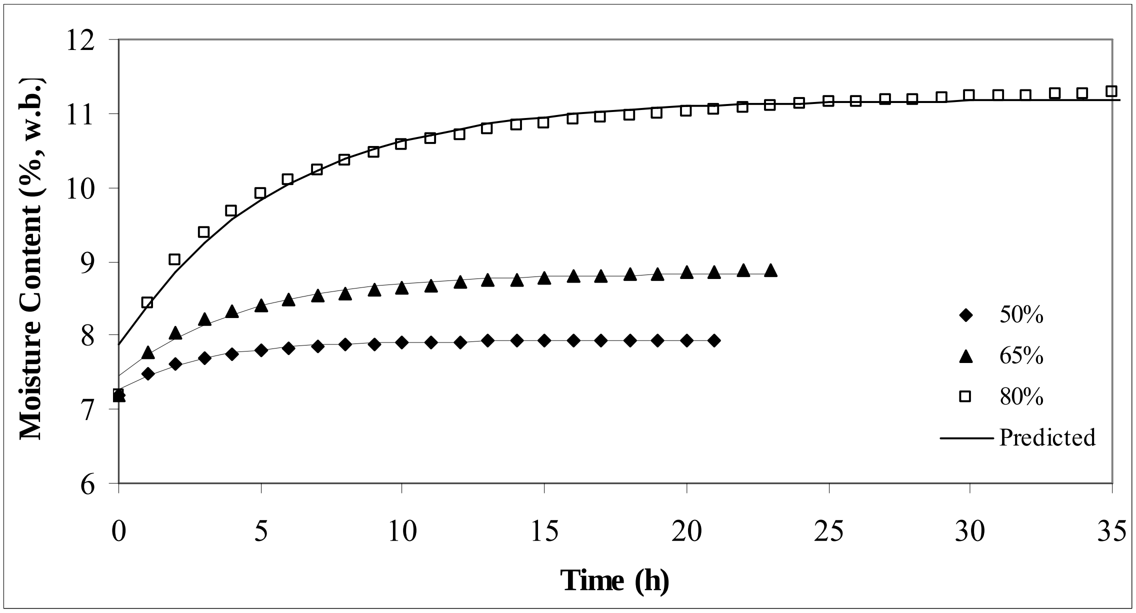

Figure A.3 - Moisture change in switchgrass pellets exposed to air at constant temperature of 25°C with varying relative humidities (50, 65 and 80%).

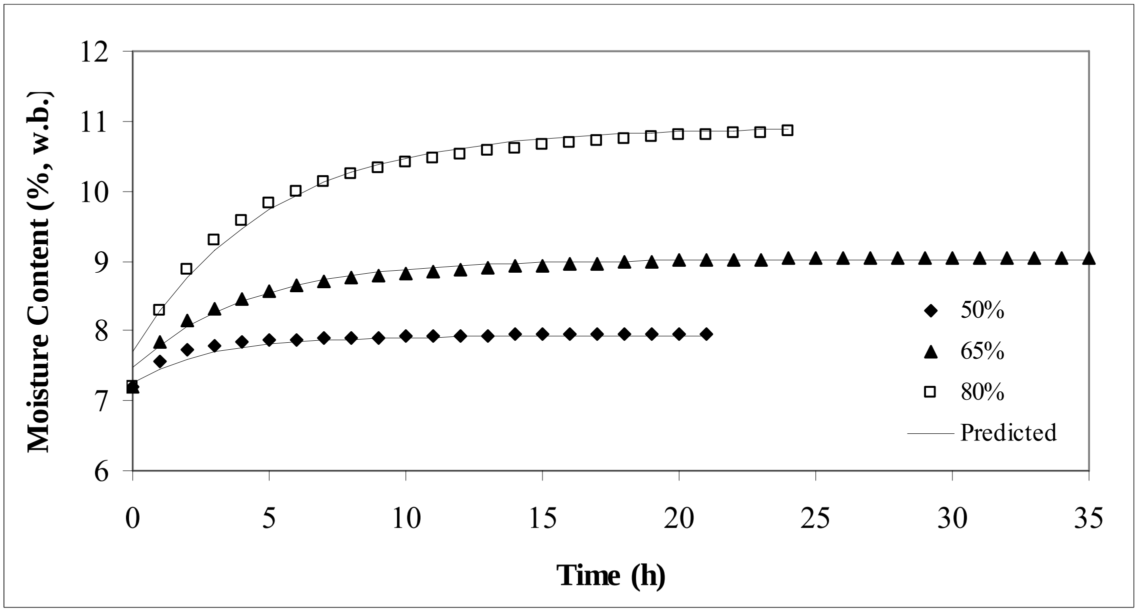

Figure A.4 - Moisture change in switchgrass pellets exposed to air at constant temperature of 35°C with varying relative humidities (50, 65 and 80%).

APPENDIX B

Table B.1 – Density of switchgrass pellets at 10.4 and 13.2% moisture content (w.b.).

Die Size	**Temp (°C)**	**Moisture (%, w.b.)**	**Density (kg/m^3)**
4.8 mm	60	10.4	1122.98
	75	10.4	1206.76
	90	10.4	1215.88
6.4 mm	60	10.4	1036.61
	75	10.4	1052.09
	90	10.4	1023.30
7.9 mm	60	10.4	966.09
	75	10.4	976.16
	90	10.4	923.31
4.8 mm	60	13.2	1122.07
	75	13.2	1151.63
	90	13.2	1206.31
6.4 mm	60	13.2	1023.30
	75	13.2	1098.42
	90	13.2	1024.27
7.9 mm	60	13.2	956.76
	75	13.2	960.56
	90	13.2	1006.86

Table B.2 – Density of switchgrass pellets at 16.2 and 20.0% moisture content (w.b.).

Die Size	**Temp (°C)**	**Moisture (%, w.b.)**	**Density (kg/m^3)**
4.8 mm	60	16.2	1049.05
	75	16.2	1048.33
	90	16.2	1075.72
6.4 mm	60	16.2	998.54
	75	16.2	936.08
	90	16.2	1045.93
7.9 mm	60	16.2	861.37
	75	16.2	898.80
	90	16.2	946.78
4.8 mm	60	20.0	750.68
	75	20.0	862.28
	90	20.0	955.21
6.4 mm	60	20.0	784.98
	75	20.0	728.61
	90	20.0	904.66
7.9 mm	60	20.0	714.73
	75	20.0	732.06
	90	20.0	844.37

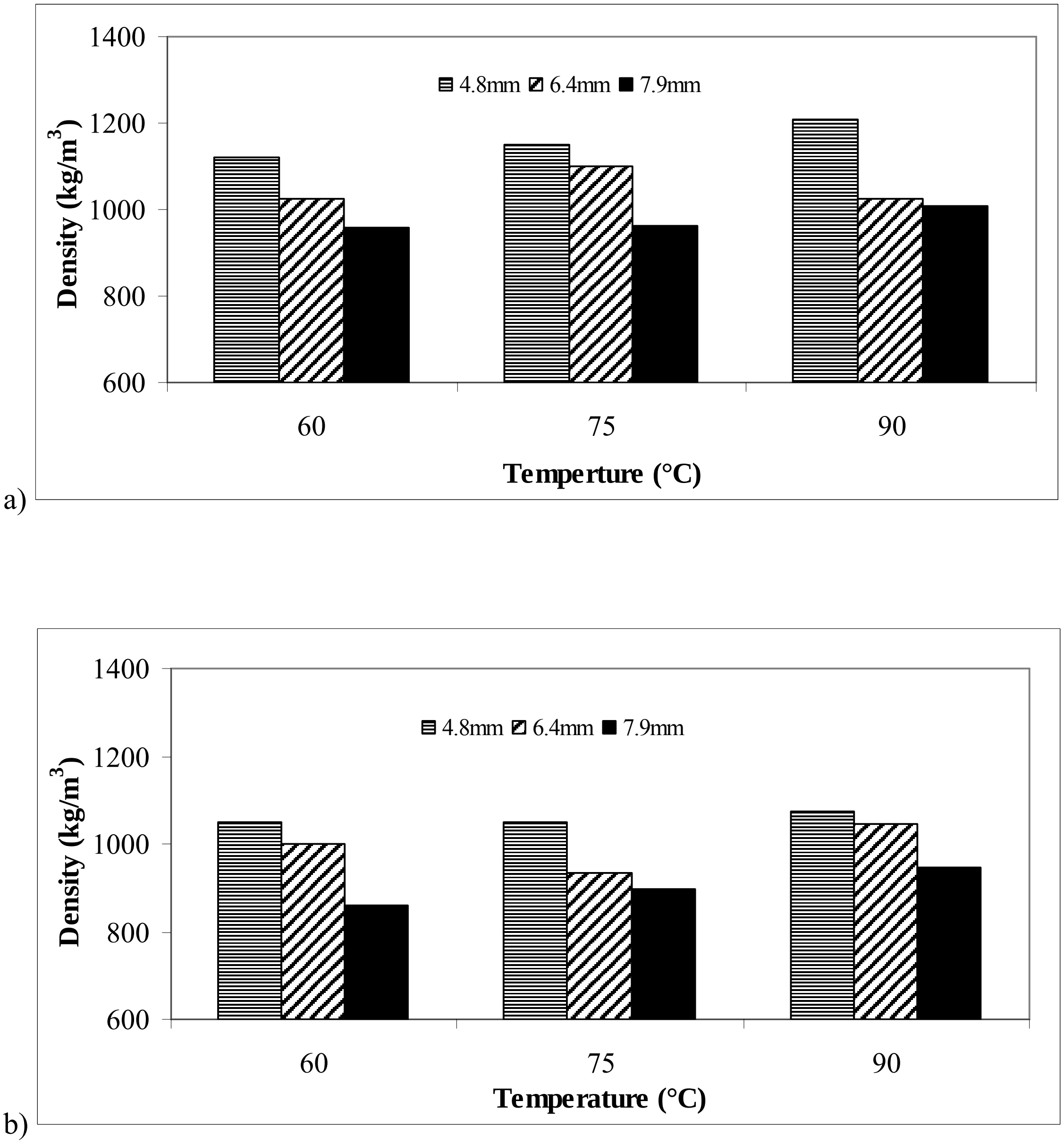

Figure B.1 – Density of switchgrass pellets affected by temperature and die size at constant moisture contents of a) 13.2% and b) 16.2% (w.b.).

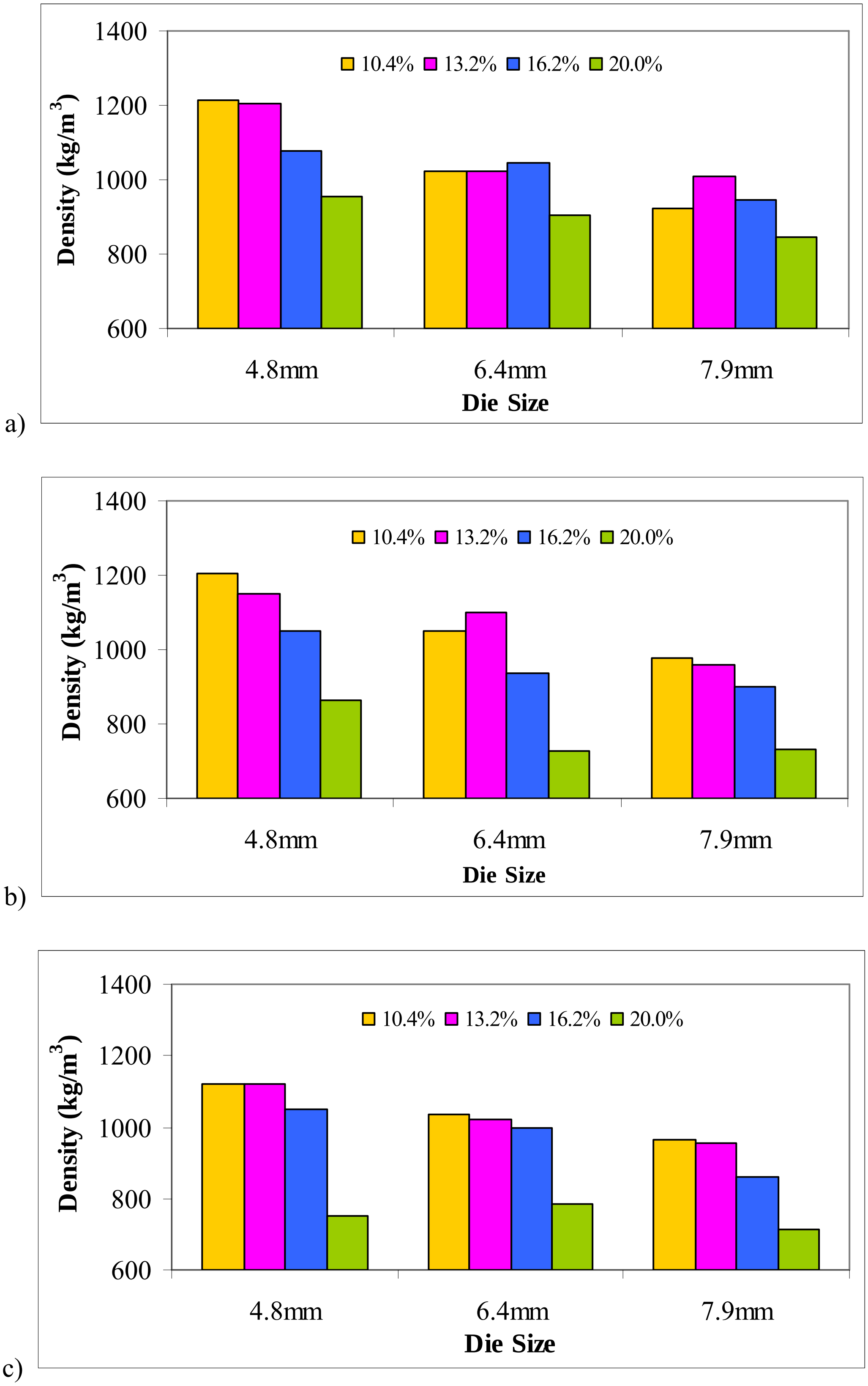

Figure B.2 – Density of pellets at constant temperature of a) 60°C b) 75°C c) 90°C.

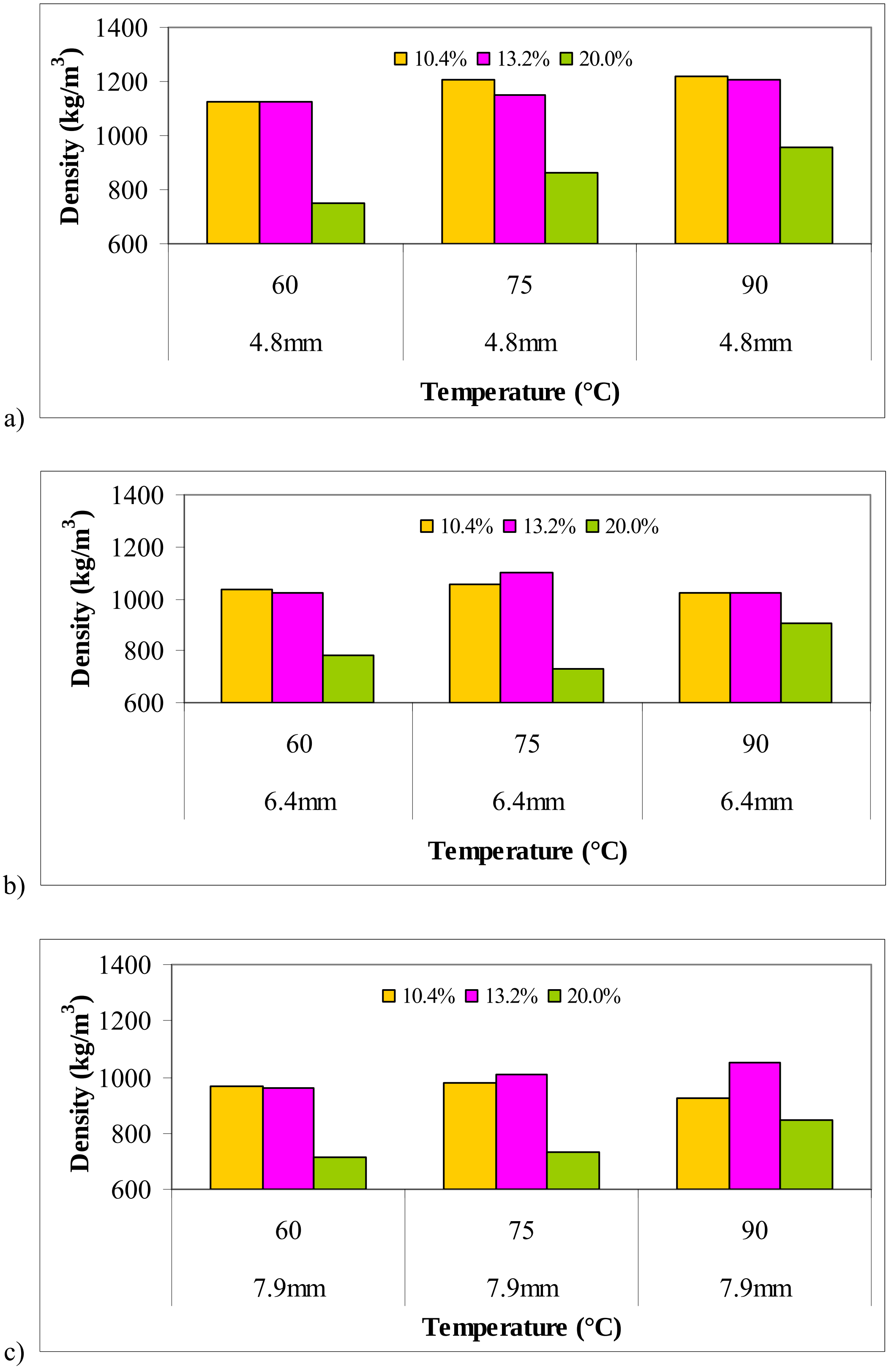

Figure B.3 – Density of pellets at constant die sizes of a) 4.8 mm b) 6.4 mm c) 7.9 mm.

Table B.3 – Specific energy used to compress ground switchgrass at 10.4 and 13.2% moisture content (w.b.).

Die Size	Temp (°C)	Moisture (%, w.b.)	Specific Energy (MJ/t)
4.8mm	60	10.4	75.18
	75	10.4	54.42
	90	10.4	47.75
6.4mm	60	10.4	70.51
	75	10.4	57.92
	90	10.4	82.07
7.9mm	60	10.4	29.26
	75	10.4	23.71
	90	10.4	26.92
4.8mm	60	13.2	62.04
	75	13.2	61.32
	90	13.2	44.34
6.4mm	60	13.2	22.48
	75	13.2	47.17
	90	13.2	65.27
7.9mm	60	13.2	25.24
	75	13.2	21.25
	90	13.2	24.15

Table B.4 – Specific energy used to compress ground switchgrass at 16.2 and 20.0% moisture content (w.b.)

Die Size	**Temp (°C)**	**Moisture (%, w.b.)**	**Specific Energy (MJ/t)**
4.8mm	60	16.2	64.41
	75	16.2	59.58
	90	16.2	81.35
6.4mm	60	16.2	60.12
	75	16.2	76.97
	90	16.2	72.18
7.9mm	60	16.2	23.00
	75	16.2	57.46
	90	16.2	20.81
4.8mm	60	20.0	**88.81**
	75	20.0	63.83
	90	20.0	54.98
6.4mm	60	20.0	53.46
	75	20.0	48.94
	75	20.0	42.57
7.9mm	60	20.0	23.72
	75	20.0	22.48
	90	20.0	19.33

Note: Unless specified all experiments were preformed in duplicate and mean values are reported.