Parameters Affecting Biomass Densification Process of The Rice Husk.

by

Muhammad Fahmi B Azme

Dissertation submitted in partial fulfilment of

the requirements for the

Bachelor of Engineering (Hons)

(Chemical Engineering)

JULY 2010

Universiti Teknologi PETRONAS

Bandar Seri Iskandar

31750 Tronoh

Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

Parameters Affecting Biomass Densification Process of The Rice Husk.

by

Muhammad Fahmi B Azme

A project dissertation submitted to the

Chemical Engineering Programme

Universiti Teknologi PETRONAS

in partial fulfilment of the requirement for the

BACHELOR OF ENGINEERING (Hons)

(CHEMICAL ENGINEERING)

Approved by,

____

(Dr. Kuzilati Ku Shaari)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

July 2010

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

(MUHAMMAD FAHMI B AZME)

ABSTRACT

Densification of biomass is often necessary to combat the negative storage and handling characteristics of these low bulk density materials. A consistent, high quality densified product is strongly desired, but not always delivered. Within the context of pelleting and briquetting, binding agents are commonly added to comminuted biomass feedstocks to improve the quality of the resulting pellets or briquettes. However in this study, author chooses to make the densification process binderless by only manipulating the parameters during the densification process. The load applied and the particle size of the rice husk is the manipulating variables in this experiment. The rice husk first is being milled by the miller and the distribution of the size ranging up to 500 μm. The rice husk is then being divided into three sizes 100 μm, 355 μm and 500 μm by using the sieving machine. For each sizes, authors set the weight of the sample to be constant which is 0.5 g. Auto pallet machine is used for densification process with the load applied starting from 500 kg to 1000 kg with the intervals of 100kg. A proper medium is necessary to hold the pallet prior to the hardness test. The pallet had undergone a hot mounting process where a resin is added to the hot mounting machine to provide a medium for pallets. After hot mounting process, the pallets with a resin as a medium are being tested by the Rockwell Hardness Machine. From the value of hardness obtained, as the load/applied pressure increased, the hardness of the rice husk also increased. The fine size of rice husk give better hardness compared to the medium and large one, but the combination of both fine and medium particle size give better hardness among all the sizes.

ACKNOWLEDGEMENT

First and foremost, I would like to thank to my supervisor of this project, Dr. Kuzilati Ku Shaari for the valuable guidance and advice. She inspired me greatly to work in this project. Her willingness to motivate me contributed tremendously to our project. I also would like to thank her for showing me some example that related to the topic of my project.

Besides, I would like to thank the authority of Universiti Teknologi Petrona (UTP) for providing me with a good environment and facilities to complete this project. Also, I would like to take this opportunity to thank to the Chemical Engineering Department of Universiti Teknology Petronas (UTP) for offering this subject, Final Year Project. It gave me an opportunity to show characteristic of being a researcher and learn about the importance in writing a journal. I also would like to thank the Mechanical Engineering Department for lending me their equipment for my project. In addition, I would also like to thank Bernas Sdn. Bhd.Kedah which provides me valuable information regarding my project and sample (rice husk).

Finally, an honorable mention goes to my families and friends for their understandings and supports on me in completing this project. Without helps of the particular that mentioned above, I would face many difficulties while doing this project.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1
INTRODUCTION

1.1 Background of Study

The demand for chemical and energy is parallel to the world population as well as the consumption and lifestyle of human being. This scenario will result in multiplied manifold for the demand of energy. Traditional inefficient energy technology can no longer be satisfied the demand of human being because its only utilizes few local resources. Therefore, a renewable energy resources is needed to occupy high demand of the human being. This renewable energy can be derived from the biomass feedstock where it exist as an abundant in nature.

Biomass is a renewable energy sources derived from the living things such as agricultural crops and waste, wood and wood wastes, animal wastes, aquatic plants, and organic fractions of municipal and industrial waste (BIOCAP and Pollution Probe, 2004). Chemical energy stored in organic matter is derived from solar energy via photosynthesis is called biomass energy or bioenergy (Hall and Rosillo Calle,1999) . The use of biomass residues was first seriously investigated during the oil embargo of the 1970s. However when oil prices dropped after the embargo, biomass residue lost its competitiveness with fossil fuel (Matsumura *et al*, 2005).

In Malaysia, rice is nominated as the important source of food and nutrition. Malaysia grows its own paddy and also imports some from neighbor countries. In Malaysia, the total area planted with paddy in peninsular is 454,917 ha, which constitutes 61.3% of total paddy plantings in the country (Ministry of Agriculture, 1993). Net average rice production in the peninsula amounted to 1, 810, 222 mt in 1992 (Ministry of Agriculture, 1993).

A major derivative of paddy is the husk or hull, a fibrous, non-digestible product that comprises approximately 20% by weight of the rough rice. For the period 1991/92, this amounted to 362,044 mt. The most common use of this residue has been the production of heat energy by burning. Due to its abrasive character, poor nutritive value, low bulk density and high ash content, only a small proportion of rice husks has been utilized for non-energy related low value applications, such as chicken litter, animal roughage (Velupillai, 1987), mulching and bedding materials (Hsu and Loh, 1980).

In many Asian countries, where the bulk of rice is produced and consumed, a major proportion of the husks are transported to open fields for disposal by burning. This practice is now strongly opposed and even prohibited in some countries under environment protection legislation. In general, rice husks are residue from the rice processing industry that costs money to dispose of in a manner that does not harm the environment. One method of turning this liability into an asset is to generate energy and profit from rice husks in a variety of ways.

Biomass exist in a low bulk densities and heterogeous in nature thus make it so difficult to handle large quantities of most feedstocks. This resulted in a large expenses during material handling (transportation, storage, etc.). A detailed study by Kumar and co-workers (2003) examined the cost to produce biomass power from direct combustion in western Canada.Out of all the factors considered, transportation cost had the second highest cost (next to capital recovery) when the biomass power plant was at full capacity (year 3). It was also stated that transportation costs will increase with increasing power plant size. In order to prevent the negative handling aspects of bulk biomass, densification process is often required.

The process of agglomeration whereby involves taking discrete and independent particles by bringing them into contact with one another,thus promoting (or allowing) interparticle adhesion to occur, and then causing structure rearrangement, usually under the action of external forces (Hogg, 1992). Densification of biomass is a form of promoted agglomeration process where pressure (along with other process variables) is utilized to force the particles to bond with one and another.

Li and Liu (2000) reported that, extrusion, roll briquetting, and pelletizing is the conventional processes for biomass densification. Extrusion involves forcing biomass through a heated die by pressure typically exerted on the product by a tapered screw. Briquetting process employs a roll press to compress a material passing between the two rolls. Depending on the surface geometry of the rolls, various shapes and sizes of compact material can be produced. Pelletizing (pelleting) is a process by which biomass is forced by an internal roller through cylindrical dies in an external ring , producing compact pellets of the charge material. Pelleting is the densification process of interest to this study.

Process of densification of biomass improves its handling characteristics, reduces transportation cost, enhances its volumetric calorific value, and produces a uniform, clean, stable fuel, or an input for further refining processes (Granada *et al*, 2002). Densification of biomass feedstocks helps improve the process of feeding the fuel into co-fired power plants (Li and Li,2000). Also, the combustion of dense granulated and uniformly sized biomass can be controlled more precisely than loose, low bulk density biomass and thus reduce emissions (Sokhansanj *et al*, 2005). With respect to animal feed, the benefits of pelleting include enhanced handling characteristics of feeds and improved animal performance. Pelleting increases bulk density and flowability and decreases spillage and wind loss (Briggs *et al*, 1999).

Production of quality pellets is a work of art rather than science as thought by the feed mill operator (Briggs *et al*, 1999).Judgement and experience of the operator is very important if there are any the changes in one or more variable or parameter that can affect the quality of the pallet (durability and hardness) (Thomas *et a*,. 1997). A method to produce high quality of densified material is required by manipulating the parameters during the densification process.

It is very essential to produce a high quality densified product in order to ensure the positive effects of densification are not mitigated. Therefore, synthetic binding agents are often added to the pre-densified biomass to improve pellet quality. Studies have demonstrated that different biomass grinds bind well without the use of artificial binding agents (Shaw and Tabil, 2005) such feedstocks possess natural binding agents that allow them to exhibit preferential qualities after densification.

Particles bind during compression has been studied, however knowledge of natural biochemical constituent behavior during densification has enormous potential to provide insight into the complex process of biomass compression. Thomas and co-workers (1998) concluded that more research efforts should be directed towards the effects of individual constituents and their respective properties, since the latter appeared to affect, to a large extent, the final hardness and durability of pellets. It was stated that the effects of raw material constituents, both their level and physico-chemical properties, may provide more information on pelleting characteristics and pellet quality than the ingredient inclusion level.

Studying the compression/compaction behavior of biomass will provide insight into the densification process. This in turn will allow the design of more efficient and cost effective densification systems, thus improving the feasibility of biomass densification for feed, chemical, and energy production.

1.2 Problem Statement

Rice husk naturally exist in the low bulk density and its existence in abundant in nature. Rice husk (Bernas Sdn. Bhd. Kedah) is use as a replacement of coal and some is for selling purposes. Author's concern in this issues is the raw rice husk exist in a low bulk density and if no proper treatment is being done to the rice husk, the cost of transportation will be higher, space for storage of rice husk and handling problem. Thus a proper method is needed to pre-treat the rice husk before selling it to the customer. Author's method is by doing the palletizing on the rice husk. Palletizing means, applying a force or load to the rice husk in order to increase the density of the rice husk.

1.3 Objectives

Prior to the densification process, a method or parameters is required for this process to be optimized. The objectives of this are stated below:

- To study the effect of the size of biomass (rice husk) to the strength of the pallet.
- To study the effect of load applied on the biomass which is rice husk to the strength of the pallet.

1.4 Scope of research

Rice husk is chosen as a biomass in this study because of the availability and easy to handle. Firstly, raw rice husk is being milled by the miller and the distribution is ranging up to 500 μm. This size is divided into three different sizes which are 100 μm, 355 μm and 500 μm by using the sieving machine. After sieving process, rice husk will be weighed for 0.5g. After weighing process, rice husk is being palletized by Auto Pallet Machine. The load applied is set at 500kg up to 1000kg with the intervals of 100kg. After palletizing, in order to test the hardness/strength of the rice husk, a proper medium is needed. This medium can be achieved by doing the hot mounting process. This material for the medium is resin and it is being heated up to 150 °C. After a proper medium is done, the sample will be tested by the Rockwell Hardness Machine to test the strength of the material.

CHAPTER 2

LITERATURE REVIEW

Densification process isa mechanical increase in density of biomass feedstocks via compression. The interest in using biomass as a source of energy is continually evolving thus providing a vast knowledge in literatures especially in recent years, which examines the many facets of biomass compression/compaction. There are over 200 publications per year on compaction alone as reported by Denny (2002). Densification of lignocellulosic material is a complex process and no coherent theory exists (Granada *et al,* 2002). Densification of biomass can be done by extrusion, roll briquetting, and pelletizing (Li and Liu, 2000). This literature review primarily focused on densification via pelletizing and closed-end die compression. In order to fully understand the physical and chemical processes occurring during biomass densification, literature from disciplines such as engineering and food/feed science were consulted

2.1 Previous biomass densification studies

A uni-axial compression via a plunger in a cylindrical die is one of the most common methods reported in literature for studying the force-deformation, relaxation, and subsequent quality characteristics of a biomass. This method allows detailed analysis of the compression/relaxation behavior of feedstocks at the laboratory scale. Plunger-die systems have been used to study the compression of alfalfa (Adapa *et al,* 2002; Hall and Hall, 1968; Tabil and Sokhansanj, 1996a; Tabil and Sokhansanj, 1996b; Tabil and Sokhansanj, 1997), straws/grasses (Demirbaş, 1999; Kaliyan and Morey, 2006; Mani *et al*, 2004; Mani *et al*, 2006a; Mani *et al*, 2006b; Ndiema *et al*, 2002; Shaw *et al* 2006; Singh and Singh, 1983; Smith *et al*, 1977; Wamukonya and Jenkins, 1995), palm fiber/shell (Husain *et al*, 2002), olive cake/refuse (Al-Widyan *et al*, 2002; Yaman *et al*, 2000), as well as wood and wood waste (Chin and Siddiqui, 2000; Demirbaş *et al*, 2004; Li and Liu, 2000; Rhén *et al*, 2005).

Firstly, raw feedstocks are typically milled or comminuted, and conditioned to an appropriate moisture content either by dehydration or moisture addition prior to the densification process. The attempt is to simulate conditions of commercial/industrial densification. The resultant products of uni-axial tests are commonly referred to in literature as pellets or briquettes. The name 'pellet' is usually given to materials less than 15 mm in diameter, while 'briquette' is generally the term used for larger units of densified material.

2.2 Parameters affecting biomass densification

The following parameters were found to influence the binderless densification experiments using a plunger-die assembly to produce single pellets/briquettes (Rehkugler and Buchele, 1969; Granada *et al*, 2002):

1. Process variables
    a. Temperature
    b. Pressure and pressure application rate (compression velocity)

2. Feedstock/material variables
    a. Moisture content
    b. Particle size, shape, and distribution
    c. Pretreatment

2.2.1 Process variables

Process variables are a set of condition (temperature and pressure) which imposed on biomass material by the mechanical densification equipment and those factors are non-material specific.

2.2.1.1 Temperature

Mani and co-workers (2003) reported that higher process temperatures require less loads to achieve a desired compact density with less power consumption. Hall and Hall (1968) found that, for a given moisture content, the pressure required to obtain a certain wafer (alfalfa and Bermuda grass) density was reduced by the addition of heat in the wafering die. Likewise, adding heat increased the moisture content at which a certain pressure was able to produce a specific wafer density. Sokhansanj and co-workers (2005) supported this observation by stating that with an increase in temperature, the resistance of the material decreases against an applied load.

Smith and co-workers (1977) found that for a given pressure, the higher the temperature (within limits of 60- 140°C), the greater the degree of compaction and stability of the briquettes (wheat straw). Also, the length of recovery (expansion) of the briquettes was less when the die temperature was between 90 and 140°C. The authors observed that wheat straw briquettes were surface charred and slightly discolored at temperatures above 110°C due to chemical degradation. In a study of densification characteristics of corn stover and switchgrass, Kaliyan and Morey (2006) used the glass transition temperature to determine the temperatures at which to study densification behavior of corn stover and switchgrass. They found that the average glass transition temperature (for moisture contents of 10, 15, and 20% wet basis (wb)) was 75°C. Increasing the moisture content generally decreased the glass transition temperature. The endpoint of the glass transition region was 100°C. Therefore, 75 and 100°C were chosen as processing temperatures for the study, along with 150°C to observe the effect of temperature beyond the glass transition. It was discovered that there was moisture migration at the highest temperature resulting in a lower durability for the 150°C briquettes than the 100°C briquettes. The durability of the 100°C briquettes was also higher than the 75°C briquettes.

2.2.1.2Pressure

It was observed by Butler and McColly (1959) that the density of chopped alfalfa hay pellets was proportional to the natural logarithm of the applied pressure. There is no doubt that an increased in applied pressure will increase the density, however, the mechanical strength of the pellets is not so easily predicted. Yaman and co-workers (2000) recommended that briquetting pressure should be selected at an optimum value. They explained that as the briquetting pressure increases, the mechanical strength of the briquettes increases as a result of the plastic deformation.

However, above an optimum densification pressure, fractures may occur due to a sudden dilation. For a given die size and storage conditions, there is a maximum die pressure beyond which no significant gain in cohesion (bonding) of the briquette can be achieved (Ndiema *et al*, 2002). With respect to pressure application rate, Li and Liu (2000) compressed oak sawdust at pressure application rates varying from 0.24 to 5.0 MPa/s. The dry density of the compacts, measured 2 min after compression, decreased with an increase in compaction speed up to 3 MPa/s. The compaction speed became negligible at rates higher than 3 MPa/s.

2.2.2 Feedstock/material variables

Feedstock/material variables are those factors which are characteristic of a particular biomass feedstock

2.2.2.1 Moisture content

In the densification process, water acts as a film-type binder by strengthening and promoting bonding via van der Waal's forces by increasing the contact area of the particles (Mani *et al*, 2003). As a general rule, the higher the moisture content, the lower the density of the pellet. Demirbaş (2004) found that increasing the moisture content (7- 15%) of pulping rejects and spruce wood sawdust resulted in stronger briquettes. Mani and co-workers (2006a) report that corn stover of a low moisture (5–10%) resulted in denser, more stable and more durable briquettes than high moisture stover (15%). Li and Liu (2000) recommend that the optimum moisture content for compacting wood in a punch-and-die assembly was approximately 8%.

Compaction of tree bark, sawmill waste, wood shavings, alfalfa hay, fresh alfalfa, and grass was reported that materials having lower moisture content and fewer long fibers (more fines) gave more stable wafers, due to limited expansion (Moshenin and Zaske, 1976). Protoplasm, liberated during the compression, acted as a binder and provided fresh alfalfa (19% moisture) with the highest durability. Sokhansanj and co-workers (2005) identified that feed material containing higher proportions of starch and protein will produce higher quality pellets than material high in cellulosic material. The optimum moisture content for pelleting cellulosic material ranges from 8 to 12% wb, while the optimum moisture content for starch and protein material (most animal feeds) can reach 20% wb. Li and Liu (2000) found that at moisture contents equal or less than 4% wb, pellets tended to absorb moisture from the air and expand significantly, becoming fragile in a few days. Starch gelatinization, protein denaturation, and fiber solubilization processes are facilitated by the presence of water, however water added as steam is far superior to conditioning with water alone, since the additional heat modifies physico-chemical properties (gelatinization of starch, denaturation of protein) to such an extent that binding between particles is greatly enhanced resulting in improved physical pellet quality (Thomas *et al*, 1997). It is evident that the optimum moisture content for densification is different for each individual feedstock and set of process conditions. Ollett and co-workers (1993) undertook a study to determine the effect of water content on the compaction behavior of food powders.

They reported that the compaction of the food powders and the effects of water content proved to be complex phenomena. Increased water content resulted in a decrease in deformation stresses, as determined by the Heckel (1961) analysis. The authors attributed this to plasticization of amorphous materials. For crystalline materials, this was explained by lubrication effects during particle rearrangement. In an experiment examining the effect of moisture on the stress relaxation of compacted powders, Peleg and Moreyra (1979) demonstrated that wet powders were more deformable than dry powders, as indicated by the longer time required to reach the preset load (Figure 2.1). This allows more stress relaxation to occur during relaxation.

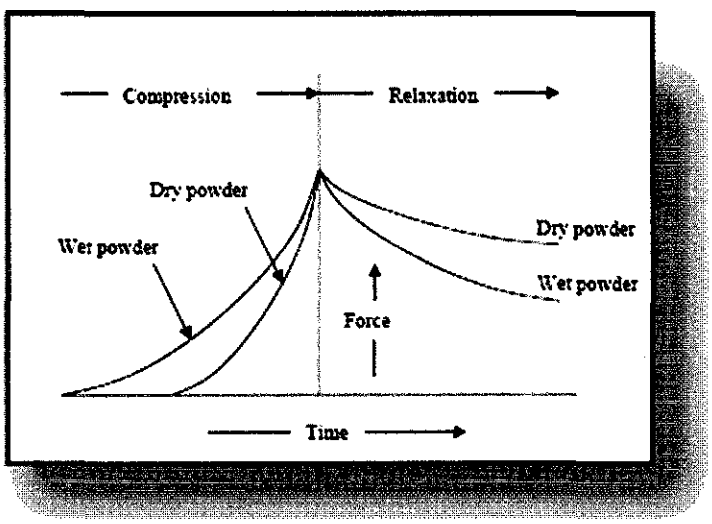

Figure 2.1: Generic compression and relaxation curve of wet and dry powders (Moreyra and Peleg 1980).

2.2.2.2 Particle size, shape, and distribution

Generally, the quality of the pellets is inversely proportional to the particle size of the material, however this is not always the case as conflicting results can be found in literature. Mani and co-workers (2003) alluded to the idea that particle size distribution has an effect on pellet quality. A proportion of fine to medium particles is required, but pellet quality and the efficiency of commercial pelleters will suffer if coarse material is not present (Payne, 1978). The effect of particle size distribution as an important material property for forage wafering when comparing leaf to stem ratios, as a higher leaf content has been reported to produce a superior densified product; this may also be due to increased protein content in the leaf. Smith and co-workers (1977) noted that the compaction and stabilization of straw may have a different mechanism than for grass due to the fact that straw is dead material and has a significantly smaller leaf content. There is a lack of information relating the effect of particle shape (i.e. roundness and surface roughness) to the quality of biomass pellets, and may be an area of future research initiatives.

2.2.2.3 Pretreatment

Pretreatment of biomass is a potential method for altering the physical structure and the structure of the chemical constituents in an attempt to enhance the binding characteristics prior to densification process. Pretreatment can be broken down into two categories: physical/mechanical and chemical.

2.2.2.3.1 Physical/mechanical pretreatment

In densification process, milling will provide a larger surface area for binding. This will also increase the porosity of the bulk material. Materials relevant to this study must be ground in order to meet the input requirements of laboratory and commercial densification equipment. For fine powders, the number of contact area between particles is higher than it would be for large particles, furthermore because the finer the powder, the larger its exposed surface area, the surface energy per unit weight (regardless of its physical-chemical character) also increases with the size reduction of the powder (Peleg 1977). In a study investigating the mechanical properties of pellets from wheat and barley straws, corn stover, and switchgrass, Mani and co-workers (2006b) concluded that particle size had a significant effect on the pellet density of all feedstocks except for wheat straw.

Compressed hot water or steam is another pretreatment approach which induces lignin removal (Liu and Wyman, 2005). Batch, partial flow, and flow-through processing techniques are available. Controlled pH methods have also been investigated (Mosier *et al*, 2005). It is postulated that by disrupting lignocellulosic biomass materials via steam explosion pretreatment, that the compression and compaction characteristics can be improved. Zandersons and co-workers (2004) stated that activation of lignin and changes in the cellulosic structure during the steam explosion process facilitate the formation of new bonds. Much of the research involving steam explosion pretreatment has focused on the alteration of the lignocellulose matrix in biomass, and subsequent improvement of enzymatic hydrolysis (Ballesteros *et al*, 2002; Nunes and Pourquie, 1996). Steam explosion has also been explored by the flax fiber industry as an upgrading step to produce high quality short fibers for the textile market (Kessler *et al*, 1998). Steam explosion pretreatment is a process by which material is introduced into a reactor and heated under steam pressure at elevated temperatures for a few minutes.

During the reaction, the hemicelluloses are hydrolyzed and become water soluble, the cellulose is slightly depolymerized, and the lignin melts and is depolymerized (Toussaint *et al*, 1991). Kaar and co-workers (1998) noted that steam explosion requires little or no chemical input and thus, is environmentally benign relative to other technologies, such as acid hydrolysis.

Anglès and co-workers (2001) explained that hydrolytic depolymerization in aqueous media is catalyzed by acidic species in wood (auto-hydrolysis) or by adding small amounts of mineral acids (pre-hydrolysis). They further explained the significant chemical reactions taking place in the wood lignocellulose:

- Partial hydrolysis of cellulose and hemicellulose into water soluble sugars and oligomers.
- Partial hydrolysis of lignin to lower molecular weight material.
- At high steam temperatures, some low molecular weight lignin melts, flows, and partially coalesces into droplets.

The latter point was supported by Murray Burke (Vice President & General Manager, SunOpta BioProcess Inc., Brampton, ON) in a personal communication. He explained that his experience demonstrates that during steam pre-treatment, the lignin breaks down and forms 'teardrop' structures. During the pelleting process, the 'teardrop' lignin remelts and forms an extremely tough outer layer. He added that biomass pellets made after pretreatment with steam auto-hydrolysis will grind the same as coal, and have been utilized to replace 10-15% of the coal in air fired power plants.

2.2.2.3.2 Chemical pretreatment

Due to the fact that lignocellulose is the most abundant chemical constituent in the biomass materials of interest, pretreatment is targeted towards the alteration of the lignocellulosic structure. Pretreated starch (i.e. pre-gelatinized starch) is commonly used in the pharmaceutical industry. However, such starch must be treated independently and then added to the tablet mixture. This study is only concerned with pretreatment of the biomass matrix as a whole.

Several recent studies have been conducted which examined the effect of chemical and hydrothermal pretreatment of lignocellulose materials. However, these studies investigated the effect of pretreatment on acid or enzymatic hydrolysis of lignocelluloses and subsequent conversion to ethanol. Such studies included the treatment of lignocellulose biomass with alkali solutions, inducing swelling and subsequent delignification. Alkali pretreatment includes aqueous ammonia in a process known as ammonia recycled percolation, or ARP (Kim and Lee, 2005). Another alkali pretreatment, known as ammonia fiber explosion (AFEX) involves the use of liquid anhydrous ammonia (Teymouri *et al*, 2005). Sodium hydroxide (Carrillo *et al*, 2005) and lime, or calcium hydroxide (Kim and Holtzapple, 2006), have also been investigated as pretreatment techniques for alteration of lignocellulose.

Sulfuric acid has been studied as a dilute acid for biomass pretreatment (Lloyd and Wyman, 2005; Saha *et al*, 2005). It has been shown that dilute acid pretreatment is successful in solubilizing the hemicellulose portion of the lignocellulose matrix (Wyman *et al*, 2005). While a majority of the aforementioned studies were intended for the pretreatment of lignocellulosic biomass for further acid or enzymatic hydrolysis, they still indicate a definitive alteration of the lignocellulose matrix.

CHAPTER 3
METHODOLOGY

3.1 Materials

Biomass choosen is rice husk because in this country, this material can be easily found and abundant especially in the northern region of Malaysia. This material author obtained from the Bernas Sdn. Bhd., Kedah. At the company, rice husk is use to replace the coal in the drying process for better cost saving. The authorities at the company claims that the rice husk give better heating capability than the coal and it last longer than coal.

Figure 3.1 : Rice husk

3.2 Method

Below is the details of the procedure taken for the experiment;

Figure 3.2: After grind rice husk

Above figure is the example of the rice husk after grinding process with the blade size of 500μm. The size distribution using the grinding machine is not uniformly distributed and the size of the particle may vary from 100μm to 500μm. In order to obtain a good range of the particle, a sieveing machine is use to divide the size of the particle. From the seiving machine setting, tray with a seive size of 100μm, 335μm, and 500μm .The bigger sized tray is place at the top of all tray and. The arrangement is 500μm, follow by 355μm, 100μm and last tray is the collected tray for the particle size below than 100 μm. Total tray used is four. The after grind rice husk is place in the tray with the size of 500μm. The operating setting for the machine are Time=10mins, Amplitude = 0.5mm and the Tray =4. During the operation, the particle with the lower size will seive through the tray depending on the particle size until it stopped. The rice husk collected at the bottom tray after 10minutes is the rice husk with the size less than 100 μm (size, s <100 μm). The rice husk collected at the 100 μm tray is the rice husk with the size of more than 100 μm and less than 355 μm (100 μm < s < 355 μm). The rice husk collected at the tray 355 μm is the rice husk with the size of more than 355 μm and less than 500 μm, (355 μm < s < 500 μm). The rice husk collected at the tray 500 μm after 10minutes of the operation is the rice husk with the size bigger than 500 μm, (s > 500 μm). The rice husk with the size bigger than 500 μm is not use because the size is too big and not uniformly distributed after grinding proceses.

Figure 3.3: Auto pallet machine

The above figure is the auto pallet machine used to apply pressure to the material according to the setting load/pressure. This machine can be operated up to 20 Tons of load. The collected rice husk after the seiving process is first need to be weight to 0.5 g for each samples. The mould use for this process is a cylindrical with the stopper and the cross section area of 0.0001327 m^2 . After the weighing process done, the rice husk is being inserted into the mould. Author use the load of the machine from 500 kg to 1000kg with the interval of 100kg. This process is done for the sample size of s <100 μm (100 μm) , 100 μm < s < 355μm (355 μm) and 355 μm < s < 500 μm (500 μm) . Figure 3.4 and 3.5 is the example of the pallet obtained after the densification process (0.5 g sample weight) . Author also included the effect of the densification of the different sample size. Author use 50% of the rice husk from 100 μm size and another 50% from the 355 μm size and apply the load from 500kg to 1000kg with the interval of 100kg.

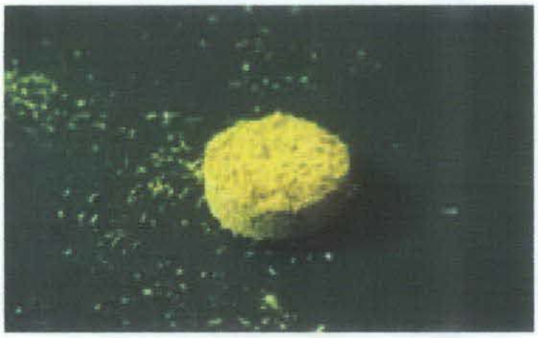

Figure 3.4 : Rice husk with the size particle of 355 μm

Figure 3.5: Rice husk with the size particle of 100 μm

After the sample undergone the palleting machine/ densification process, hot mounting machine will take place to provide a better medium for the sample. Figure 3.6 is the picture of the hot mounting machine use for mounting process. The sample is inserted in the cylindrical part of the machine and the resin is added on top of the sample covering the whole body of the sample. The operating setting for the machine are preheat = 5mins, heating = 8mins , cooling= 5mins and the temperature is 300F/150°C.

Figure 3.6: Hot mounting machine

After the sample done the mounting process, it undergone the last test which is the Rockwell Hardness test. Figure 3.7 is the sample after the mounting process. The black medium is the resin after it melted and cover the rice husk (excluding the top surface of the rice husk).

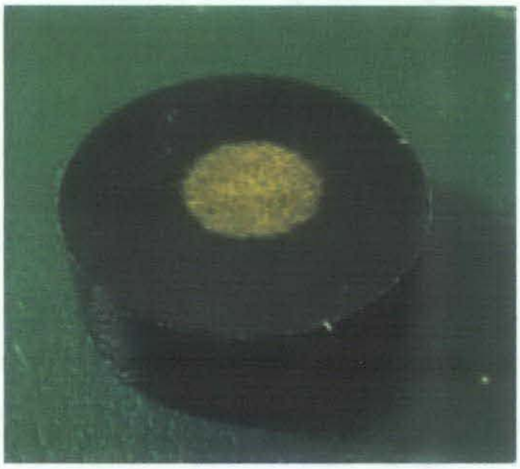

Figure 3.7 : Rice husk after the mounting process.

The resin provide the rigid medium for the rice husk as it hold the sample tightly. Figure 3.8 is the picture of the hardness testing machine. Rockwell Hardness use to measure the hardness of the rice husk. The load applied for this machine is 150 kg and the diamond type. The dented on the surface of the rice husk is the result of the hardness testing process. The deeper the dented area, the lesser the hardness of the material posses.

Figure 3.8: Rockwell Hardness Machine

CHAPTER 4

RESULTS & DISCUSSION

Below is the table for the result obtain. Table below is the data obtained throughout the process with the variation of load/pressure calculated and the hardness it posses.

Sample size = 100 μm		
Load (kg)	Pressure Calculated (Mpa)	Hardness (kg/nm^2)
500	37.68	57.2
600	45.21	58.3
700	52.75	59.8
800	60.29	60.5
900	67.82	67.5
1000	75.36	73.5

Table 4.1 : Data obtained from rice husk with size of 100 μm

Refer to table 4.1, the data obtained for the hardness is increasing as the load increased. The hardness of the rice husk is maximum at the load of 1000kg / 75.36 Mpa of pressure which is 73.5kg/nm^{2}and lowest at 500kg / 37.68 Mpa of pressure which is 57.2 kg/nm^2.

Sample size = 355 μm		
Load (kg)	Pressure Calculated (Mpa)	Hardness (kg/nm^2)
500	37.68	50.3
600	45.21	52.9
700	52.75	55.8
800	60.29	58.1
900	67.82	59.2
1000	75.36	61.7

Table 4.2 : Data obtained from rice husk with size of 355 μm

Refer to table 4.2, the data obtained for the hardness is increasing as the load increased. The hardness of the rice husk is maximum at the load of 1000kg / 75.36 Mpa of pressure which is 61.7 kg/nm^{2}and lowest at 500kg / 37.68 Mpa of pressure which is 50.3 kg/nm^2.

Sample size = 500 μm		
Load (kg)	Pressure Calculated (Mpa)	Hardness (kg/nm^2)
500	37.68	48.6
600	45.21	49.2
700	52.75	51.3
800	60.29	53.8
900	67.82	55
1000	75.36	55.6

Table 4.3: Data obtained from rice husk with size of 500 μm

Refer to table 4.3, the data obtained for the hardness is increasing as the load increased. The hardness of the rice husk is maximum at the load of 1000kg / 75.36 Mpa of pressure which is 55.6 $kg/nm^2$and lowest at 500kg / 37.68 Mpa of pressure which is 48.6 kg/nm^2.

Sample size = 50% 100μm, 50% 355μm		
Load (kg)	Pressure Calculated (Mpa)	Hardness (kg/nm^2)
500	37.68	66
600	45.21	66.4
700	52.75	68.7
800	60.29	71.2
900	67.82	74.5
1000	75.36	80

Table 4.4: Data obtained from rice husk with size of 50% 100μm, 50% 355μm

Refer to table 4.4, the data obtained for the hardness is increasing as the load increase. The hardness of the rice husk is maximum at the load of 1000kg / 75.36 Mpa of pressure which is 80 $kg/nm^2$and lowest at 500kg / 37.68 Mpa of pressure which is 66.0 kg/nm^2.

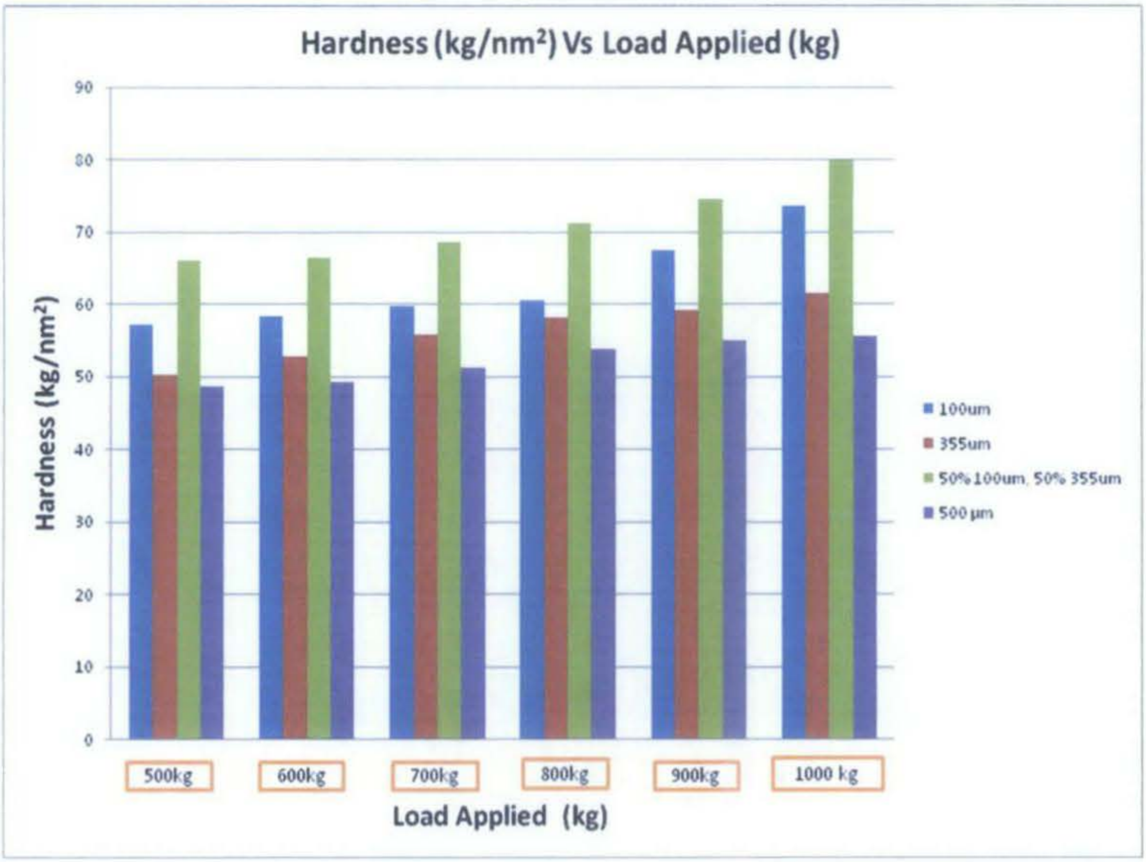

Figure 4.1: Histogram of the data obtained.

Refer to the figure 4.1, from the general view of the histogram, basically the strength of the combination of two particle size which is 50% 100µm, 50% 355µm give better hardness at all point of the load/ pressure applied. From the histogram, it is in the green bar. Rice husk of the particle size of 500 µm give lowest value of the hardness and it applies to all the load/pressure applied.

From the figure 5.1, when we increased the applied load/applied pressure of the auto-pallet machine the hardness is gradually increase as well and the characteristic of this behavior is in a linear form. The linear line is as graph below;

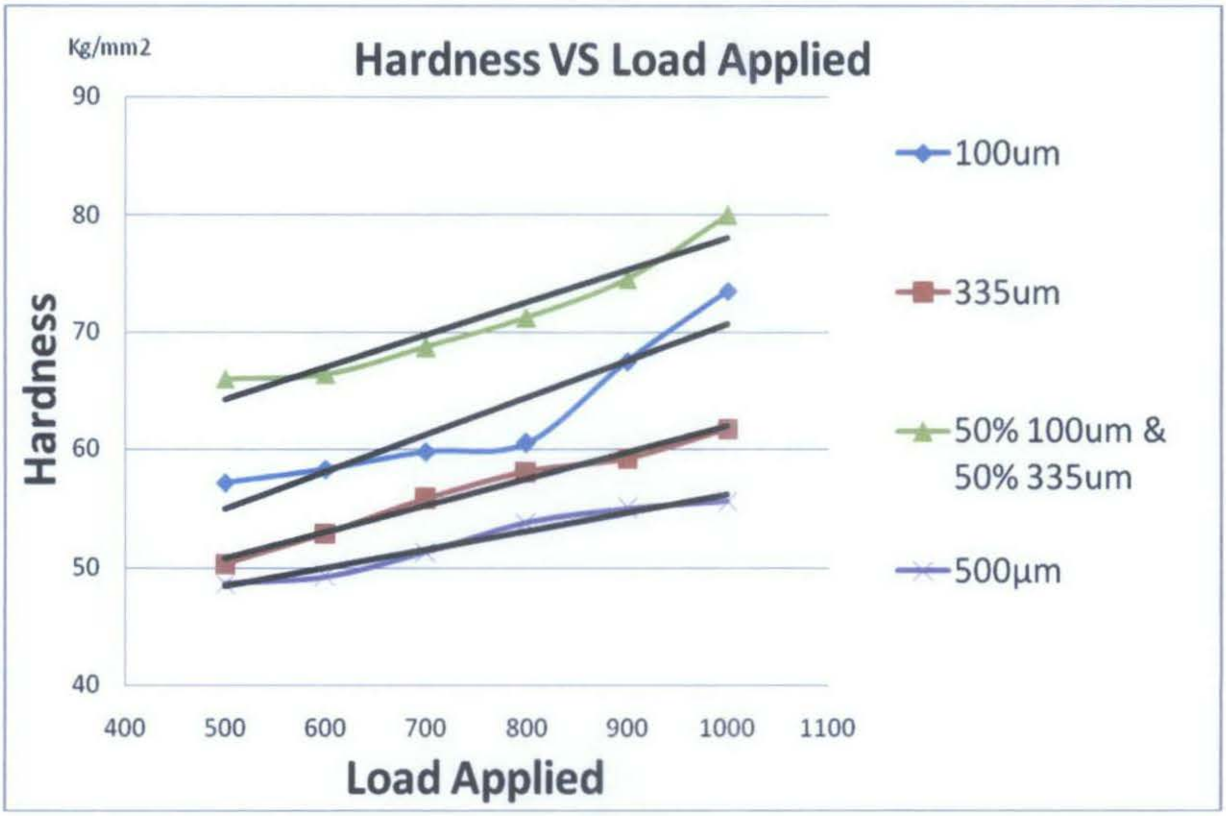

Figure 4.2: Graph Hardness vs load applied

From the graph above, the material size also plays important role in determining the hardness is posses. The small size of rice husk which is 100 µm give better hardness compare to 355 µm and 500 µm. This is because when the mass is contant which is 0.5 g, the small particle give better contact area between particle, thus make the structure more stonger. When the interaction between particle higher, the interparticle force is bigger. Generally, biomass contain large value of lignin. This lignin can naturally act as the natural binder. When the particle is smaller, the lignin can interact with each other more.

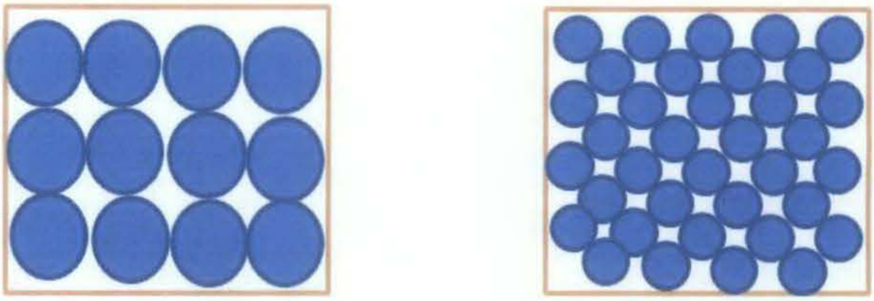

Figure 4.3: Graphical view between large and small particles

From the graph above, the mixture of both size 100 μm and 355 μm give better hardness compare to individual size hardness with respect to the load applied. This phenomena happen because the interparticle reaction is more complex and the complexity of the mixture makes the structure more rigid and hard. The small particle will fill the void between the large particle and fill the unoccupied space. The structure of the mixture cannot be ilustrated as 2 dimensions because the structure is complex and is well ilustrated in 3 dimensions.

Generally, in the market of palleting, most of the suppliers use synthetic binder to make the bonding stronger, but my this experiment no bonding material is use for palleting, just the variation of pressure applied and the sample size. Natural binding in biomass happen because the interaction between the particle's lignin. Lignin can be activated as the natural binder when the pressure applied within the region not exceeding the plastic deformation region. When the applied pressure exceed the limit of the platic region , the structure's hardness will be poor because of the dilation and may cause rupture to the structure. The more lignin exposed, the stronger hardness the structure posses.

CHAPTER 6
CONCLUSION

As the conclusion, the pressure and the size of the material do affect the strength or hardness of the material. For the size of the material, the small size gives better hardness compared to the bigger size because the smaller size of the particle give more contact area between particles and enhance the interparticle interaction(lignin interaction).

As the pressure increase, the strength of the material or hardness also increase. The increase in pressure is linear with the hardness. This is because when the pressure applied to the material which is biomass, most of the biomass act as the plastic where deformation and dilation present. The material can withstand only to some extent of pressure untill it dilates. From this experiment, author only use the pressure up to the extent of 75Mpa. From the data obtained, the value of the hardness keep increasing with respect to the pressure. Up to 75Mpa, the rice husk is still is the plastic region. If the experiment is extend to pressure higher than 75Mpa, maybe the hardness of the material will decrease because of the plastic deformation and sudden dilation/rupture.

The mixture of different size give better hardness compared to the same size rice husk. This is because the mixture of small and bigger size give more rigid structure and the small particles can easily fill up the void and empty spaces between the large particles thus give better structure and better hardness.

RECOMMENDATION

A further studies regarding this project could be done by examining the sample after the densification process by using the Semi Electron Microscopy(SEM) to study the detail of the substance that bind together during the densification process. From the literature review it is said that the lignin at a certain degree of temperature and pressure will melt down and form a connection to make the bond between the particle stronger. In this project, author did not include the effect of the temperature on the densification process because of the lack of equipment to manufacture the mounting of the sample. Further experiment could be done by using the cold mounting machine and the temperature of the sample could be varies during the densification process. Further work also could be done by varying more particle size and the mixture of it by combining starting from 10% and up to 80% because author only did for 50%.

REFERENCES

Adapa, P.K., L.G. Tabil, G.J. Schoenau, B. Crerar and S. Sokhansanj. 2002. Compression characteristics of fractionated alfalfa grinds. *Powder Handling & Processing* 14(4), pp 252-259.

Al-Widyan, M.I., H.F. Al-Jilal, M.M. Abu-Zreig and N.H. Abu-Hamdeh. 2002. Physical durability and stability of olive cake briquettes. *Canadian Biosystems Engineering* 44, pp 3.41-3.45.

Anglès, M.N., F. Ferrando, X. Farriol and J. Salvadó. 2001. Suitability of steam exploded residual softwood for the production of binderless panels. Effect of the pre-treatment severity and lignin addition. *Biomass and Bioenergy* 21, pp 211-224.

Ballesteros, I., J.M. Oliva, M.J. Negro, P. Manzanares and M. Ballesteros. 2002. Enzymic hydrolysis of steam exploded herbaceous agricultural waste (*Brassica carinata*) at different particule sizes. *Process Biochemistry* 38, pp 187-192.

BIOCAP and Pollution Probe. 2004. Primer on Bioproducts. http://www.biocap.ca/images/ pdfs/BioproductsPrimerE.pdf (2006/01/31).

Briggs, J.L., D.E. Maier, B.A. Watkins and K.C. Behnke. 1999. Effect of ingredients and processing parameters on pellet quality. *Poultry Science* 78, pp 1464-1471.

Butler, J.L. and H.F. McColly. 1959. Factors affecting the pelleting of hay. *Agricultural Engineering* 40, pp 442-446

Carrillo, F., M.J. Lis, X. Colom, M. López-Mesas and J.Valldeperas. 2005. Effect of alkali pretreatment on cellulose hydrolysis of wheat straw: kinetic study. *Process Biochemistry* 40, pp 3360-3364.

Chin, O.C. and K.M. Siddiqui. 2000. Characteristics of some biomass briquettes prepared under modest die pressures. *Biomass and Bioenergy* 18, pp 223-228.

Demirbaş, A., A. Şahin-Demirbaş and A. Hilal Demirbaş. 2004. Briquetting properties of biomass waste materials. *Energy Sources* 26, pp 83-91.

Demirbaş, A., A. Şahin-Demirbaş and A. Hilal Demirbaş. 2004. Briquetting properties of biomass waste materials. *Energy Sources* 26, pp 83-91.

Demirbaş, A. 1999. Physical properties of briquettes from waste paper and wheat straw mixtures. *Energy Conversion & Management* 40, pp 437-445.

Granada, E., L.M. López González, J.L. Míguez and J. Moran. 2002. Fuel lignocellulosic briquettes, die design and products study. *Renewable Energy* 27, pp 561-573.

Denny, P.J. 2002. Compaction equations: a comparison of the Heckel and Kawakita equations. *Powder Technology* 127, pp 162-172.

Hall, D.O. and F. Rosillo-Calle. 1999. Biological conversion of biomass to high-quality chemical carriers. In *Chemistry for the Energy Future*, ed. V.N. Parmon, H. Tributsch, A.V. Bridgwater and D.O. Hall, 121-136. Oxford, NY: Blackwell Science Ltd.

Hall, G.E. and C.W. Hall. 1968. Heated-die wafer formation of alfalfa and Bermudagrass. *Transactions of the ASAE* 11, pp 578-581.

Heckel, R.W. 1961. An analysis of powder compaction phenomena. *Transactions of the Metallurgical Society of AIME* 221, pp 1001-1008.

Hogg, R. 1992. Agglomeration models for process design and control. *Powder Technology* 69, pp 69-76.

Hsu, W.H. and B.S. Loh. 1980. *Rice Hulls in Rice Production and Utilization.* Westport, Connecticut: Avi Publishing.

Husain, Z., Z. Zainac and Z. Abdullah. 2002. Briquetting of palm fibre and shell from the processing of palm nuts to palm oil. *Biomass and Bioenergy* 22, pp 505-509.

Kaar, W.E., C.V. Gutierrez and C.M. Kinoshita. 1998. Steam explosion of sugarcane bagasse as a pretreatment for conversion to ethanol. *Biomass and Bioenergy* 14(3) , pp 277-287.

Kaliyan, N. and R. Morey. 2006. Densification characteristics of corn stover and switchgrass. Presented at the ASABE Annual International Meeting, July 9-12, 2006, Portland, OR. ASABE Paper No. 066174. ASAE, 2950 Niles Road, St. Joseph, MI 49085-9659 USA.

Kessler, R.W., U. Becker, R. Kohler and B. Goth. 1998. Steam explosion of flax – a superior technique for upgrading fiber value. *Biomass and Bioenergy* 14(3) , pp 237-249.

Kim, T.H. and Y.Y. Lee. 2005. Pretreatment and fractionation of corn stover by ammonia recycle percolation process. *Bioresource Technology* 96, pp 2007-2013.

Kim S. and M.T. Holtzapple. 2006. Effect of structural features on enzyme digestibility of corn stover. *Bioresource Technology* 97, pp 583-591.

Kumar, A., J.B. Cameron and P.C. Flynn. 2003. Biomass power cost and optimum plant size in western Canada. *Biomass and Bioenergy* 24, pp 445-464.

Li, Y. and H. Liu. 2000. High pressure densification of wood residues to form an upgraded fuel. *Biomass and Bioenergy* 19, pp 177-186.

Liu, C. and C.E. Wyman. 2005. Partial flow of compressed-hot water through corn stover to enhance hemicellulose sugar recovery and enzymatic digestibility of cellulose. *Bioresource Technology* 96, pp 1978-1985.

Lloyd, T.A. and C.E. Wyman. 2005. Combined sugar yields for dilute sulfuric acid pretreatment of corn stover followed by enzymatic hydrolysis of the remaining solids. *Bioresource Technology* 96, pp 1967-1977.

Mani, S., L.G. Tabil and S. Sokhansanj. 2003. An overview of compaction of biomass grinds. *Powder Handling & Processing* 15(3), pp 160-168.

Mani, S., L.G. Tabil and S. Sokhansanj. 2004. Grinding performance and physical properties of wheat and barley straws, corn stover and switchgrass. *Biomass and Bioenergy* 37, pp 339-352.

Mani, S., L.G. Tabil and S. Sokhansanj. 2006a. Effects of compressive force, particle size and moisture content on mechanical properties of biomass pellets from grasses. *Biomass and Bioenergy* 30, pp 648-654.

Mani, S., L.G. Tabil and S. Sokhansanj. 2006b. Specific energy requirement for compacting corn stover. *Bioresource Technology* 97, pp 1420-1426.

Matsumura, Y., T. Minowa and H. Yamamoto. 2005. Amount, availability, and potential use of rice straw (agricultural residue) biomass as an energy resource in Japan. *Biomass and Bioenergy* 29,pp 347-354.

Ministry of Agriculture 1993. *Paddy Statistics.* Kuala Lumpur: Planning and Development Division.

Moreyra, R. and M. Peleg. 1980. Compressive deformation patterns of selected food powders. *Journal of Food Science* 45: 864-868.

Moshenin, N. and J. Zaske. 1976. Stress relaxation and energy requirements in compaction of unconsolidated materials. *Journal of Agricultural Engineering Research* 21, pp 193-205.

Mosier, N., R. Henderson, N. Ho, M. Sedlak and M.R. Ladisch. 2005. Optimization of pH controlled liquid hot water pretreatment of corn stover. *Bioresource Technology* 96, pp 1986-1993.

Ndiema, C.K.W., P.N. Manga and C.R. Ruttoh. 2002. Influence of die pressure on relaxation characteristics of briquetted biomass. *Energy Conversion and Management* 43, pp 2157-2161.

Nunes, A.P. and J. Pourquie. 1996. Steam explosion pretreatment and enzymatic hydrolysis of eucalyptus wood. *Bioresource Technology* 57, pp 107-110.

Ollett, A.-L., A.R. Kirby, R. Parker and A.C. Smith. 1993. A comparative study of the effects of water content on the compaction behaviour of some food materials. *Powder Technology* 75, pp 59-65.

Payne, J.D. 1978. Improving quality of pellet feeds. *Milling Feed and Fertiliser* 161, pp 34-41.

Peleg, M. 1977. Flowability of food powders and methods for its evaluation – a review. *Journal of Food Process Engineering* 1, pp 303-328.

Peleg, M. and R. Moreyra. 1979. Effect of moisture on the stress relaxation pattern of compacted powders. *Powder Technology* 23, pp 277-279.

Rehkugler, G.E. and W.F. Buchele. 1969. Biomechanics of forage wafering. *Transactions of the ASAE* 12, pp 1-8.

Rhén, C., R. Gref, M Sjöström and I. Wästerlund. 2005. Effects of raw material moisture content, densification pressure and temperature on some properties of Norway spruce pellets. *Fuel Processing Technology* 87, pp 11-16.

Saha, B.C., L.B. Iten, M.A. Cotta and Y.V. Wu. 2005. Dilute acid pretreatment, enzymatic saccharification and fermentation of wheat straw to ethanol. *Process Biochemistry* 40, pp 3693-3700.

Shaw, M.D. and L.G. Tabil. 2005. Compression studies of peat moss, wheat straw, oat hulls and flax shives. *Powder Handling and Processing* 17(6), pp 344-350.

Shaw, M.D., L.G. Tabil, S. Panigrahi and P. Chang. 2006. Evaluation of compression, relaxation, and frictional properties of peat moss, wheat straw, oat hull, and flax shive grinds. Poster presented at the Third International Conference on Biomass for Energy, September 18-20, Kiev, Ukraine.

Singh, A. and Y. Singh. 1983. Briquetting of wheat straw. *Journal of Research Punjab Agricultural University* 20(2), pp 154-159.

Smith, I.E., S.D. Probert, R.E. Stokes and R.J. Hansford. 1977. The briquetting of wheat straw. *Journal of Agricultural Engineering Research* 22, pp 105-111.

Sokhansanj, S., S. Mani, X. Bi, P. Zaini and L. Tabil. 2005. Binderless pelletization of biomass. Presented at the ASAE Annual International Meeting, July 17-20, 2005, Tampa, FL. ASAE Paper No. 056061. ASAE, 2950 Niles Road, St. Joseph, MI 49085-9659 USA.

Sokhansanj, S., S. Mani, X. Bi, P. Zaini and L. Tabil. 2005. Binderless pelletization of biomass. Presented at the ASAE Annual International Meeting, July 17-20, 2005, Tampa, FL. ASAE Paper No. 056061. ASAE, 2950 Niles Road, St. Joseph, MI 49085-9659 USA.

Tabil, Jr., L.G. and S. Sokhansanj. 1996a. Compression and compaction behavior of alfalfa grinds – part 1: compression behavior. *Powder Handling & Processing* 8(1), pp 17-23.

Tabil, Jr., L.G. and S. Sokhansanj. 1996b. Compression and compaction behavior of alfalfa grinds – part 2: compaction behavior. *Powder Handling & Processing* 8(2), pp 117-122.

Tabil, Jr., L.G. and S. Sokhansanj. 1997. Bulk properties of alfalfa grind in relation to its compaction characteristics. *Applied Engineering in Agriculture* 13(4), pp 499-505.
Toussaint, B., G. Excoffier and M.R. Vignon. 1991. Effect of steam explosion treatment on the physico-chemical characteristics and enzymic hydrolysis of poplar cell wall components. *Animal Feed Science and Technology* 32, pp 235-242.

Teymouri, F., L. Laureano-Perez, H. Alizadeh and B.E. Dale. 2005. Optimization of the ammonia fiber explosion (AFEX) treatment parameters for enzymatic hydrolysis of corn stover. *Bioresource Technology* 96, pp 2014-2018.

Thomas, M., D.J. van Zuilichem and A.F.B. van der Poel. 1997. Quality of pelleted animal feed 2. Contribution of processes and its conditions. *Animal Feed Science Technology* 64, pp 173-192.

Thomas, M., T. van Vliet and A.F.B. van der Poel. 1998. Physical quality of pelleted animal feed 3. Contribution of feedstuff components. *Animal Feed Science Technology* 70, pp 59-78.

Velupillai, L. 1987. *Rice Hull Utilization for Energy - Review of The Technologies.* Baltimore American Society of Agricultural Engineers.

Wamukonya, L. and B. Jenkins. 1995. Durability and relaxation of sawdust and wheatstraw briquettes as possible fuels for Kenya. *Biomass and Biomass* 8(3), pp 175-179.

Wyman, C.E., B.E. Dale, R.T. Elander, M. Holtzapple, M.R. Ladisch and Y.Y. Lee. 2005. Comparative sugar recovery data from laboratory scale application of leading pretreatment technologies to corn stover. *Bioresource Technology* 96, pp 2026-2032.

Yaman, S., M. Şahan, H. Haykiri-açma, K. Şeşen and S. Küçükbayrak. 2000. Production of fuel briquettes from olive refuse and paper mill waste. *Fuel Processing Technology* 68, pp 23-31.

Zandersons, J., J. Gravitis, A. Zhurinsh, A. Kokorevics, U. Kallavus and C.K. Suzuki. 2004. Carbon materials obtained from self-binding sugar cane bagasse and deciduous wood residues plastics. *Biomass and Bioenergy* 26, pp 345-360.