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Compaction behavior, mechanical properties, and moisture resistance of torrefied and non-torrefied biomass pellets

Tianmiao Wang
West Virginia University

COMPACTION BEHAVIOR, MECHANICAL PROPERTIES, AND MOISTURE RESISTANCE OF TORREFIED AND NON-TORREFIED BIOMASS PELLETS

Tianmiao Wang

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Approved by

David DeVallance, Committee Chairperson
Gloria Oporto, Committee Member
Jingxin Wang, Committee Member

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Keywords: Biomass pellets, Pelletizing, Compaction energy, Moisture resistance, Torrefaction

Abstract

COMPACTION BEHAVIOR, MECHANICAL PROPERTIES, AND MOISTURE RESISTANCE OF TORREFIED BIOMASS PELLETS

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Biomass properties have a potential to be improved by torrefaction, a thermal pretreatment process that removes hemicellulose. The intent in using torrefaction for biomass is to increase the carbon content and calorific value, as well as reduce the hydrophilic nature of woody-biomass. To facilitate the handling and use of torrefied biomass, densification (e.g. pelletizing) is used to compact it into standard uniform shape pellets with high density and mechanical strength. In this thesis, torrefaction as pretreatment, as well as moisture content and particle size of raw biomass materials before pelletizing were studied as parameters that may affect the biomass pellet quality including: compaction behavior, gross heating value, hardness, and moisture resistance. Woody biomass red oak (*Quercus rubra*), and two species of grass-type biomass switchgrass (*Panicum virgatum*), and miscanthus (*Miscanthus giganteus*) were used in the experiment. Results of this research indicated that torrefied biomass required 50% ~ 200% more pelletization energy than non-torrefied biomass. Additionally, in general, the hardness of torrefied biomass pellets was lower than non-torrefied pellets. However, it was found that the moisture resistance of the torrefied biomass pellets was higher than the non-torrefied pellets. The moisture content and particle size also showed some relatively small effects on the biomass pellet properties, but their influence varied from species to species, and even between pretreatments within one species. Therefore, based on the results of this research, the better moisture content and particle size for pellet production was specific to each type of pretreatment and species.

Key words: Biomass pellets, pelletizing, compaction energy, moisture resistance, torrefaction

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Biomass, includs woody and agricultural plants, has become an important part of the world's energy resource. Much of the attention given to biomass has to do with it being more sustainable and renewable than comparable fuel feedstock. The U.S. Renewable Fuel Standard (RFS) is targeting and increase in biofuel use within the U.S. to 36 billion gallons by 2022 (Schnepf & Yacobucci, 2013). Nevertheless, most biomass is not convenient or economical in terms of handling, transportation, storage, and usage due to disadvantages such as low density and hydrophilic property. According to Emery and Mosier (2012), during storage, biomass loses weight and energy content, and also releases greenhouse gases. To deal with these problems, pretreatment (e.g. torrefaction, extraction) and densification (e.g. pelleting, briquetting) have been applied to improve biomass properties such as bulk density or calorific value.

In recent years, torrefaction, due to its improvement of biomass properties, has become an attractive advanced pretreatment for biomass. Torrefaction is a thermal pretreatment that is performed by heating biomass material at relatively low temperatures of 225-300℃ under atmospheric pressure and in the absence of oxygen (Prins et al., 2006). In torrefaction, less thermal stability hemicellulose, as well as a proportion of cellulose and lignin, decomposes at the temperature range of 225-325°C (Prins et al., 2006). According to a research by Shang et al. (2012), during torrefaction, hemicellulose is totally consumed when treatement is performed at 300℃ for 2 hours. Since hemicellulose is the most reactive component, it contains a large amount of hydroxyl groups which in turn causes biomass to be hydrophilic. Additionally, through torrefaction, the carbon content of biomass increases due to the decrease of hydrogen and oxygen content. The increase in carbon content through torrefaction is important as the resulting biomass calorific value is closer to that of coal. Given these higher calorific values, torrefied biomass is often referred to as "bio-coal" with its coal-like properties (Kaliyan et al., 2014). Furthermore, the torrefied biomass could be used as a bio-fuel to be gasified or co-combusted with coal, or by itself to generate energy or heat with less greenhouse gases emission compared to fossil fuels. In Li et al. study (2012), the lower heating value of biomass was increased from 18.0 MJ/kg to 20.5 MJ/kg which is closer to coal (24.0 MJ/kg). Li also reported that torrefied biomass could be burned in pulverized coal boiler without coal or adding special conditions.

The components (e.g. hemicellulose, volatile extractives) of biomass are gasified in torrefaction and result in biomass weight loss which in turn increases the energy density of biomass. According to Bergman's (2005) report, 30% of the biomass weight containing only 10% of energy in biomass was lost in torrefaction. These results indicate that 90% of the energy in biomass was retained in 70% of the biomass weight, which signifies that the energy density was increased 1.3 times after torrefaction. Furthermore, the weight loss of torrefied biomass becomes greater as the torrefied temperature increases. Specifically, over a 2-hour period, the weight loss increases from 25% at the torrefied temperature of 250℃ to 53% at the torrefied temperature of 300℃. In addition, the calorific value and energy density of torrefied biomass also increased with higher torrefaction temperature or longer residence time, which are key parameters when controlling the severity of torrefaction. Because of the significant reduction of hemicellulose content, the lignin content percentage is larger than the growth of cellulose content due to its stable thermal property. However, the higher lignin content also causes the ash content increased with raising the torrefied temperature. Since hydroxyl groups are removed in torrefaction, the torrefied biomass would become less hydrophilic and maintain very low moisture content that commonly ranges from 1% to 6% (Li et al., 2012; Bergman, 2005). This reduction in the hydrophilic nature should improve the storage properties on biomass pellets.

Additionally, Chen et al. (2011) reported that biomass becomes more porous after torrefaction at varying levels. Specifically, at relatively low torrefaction temperature of 220°C and 250°C, the surface of biomass cell wall present cell structure under Scanning Electron Microscope (SEM). However, when the biomass was torrefied under higher temperature of 280°C, the resulting cell wall structure form was tubular. Due to the porous structure and low moisture content, the grindability is markedly improved by torrefaction to that matching coal, especially with increasing severity of torrefaction. Based on Phanphanich & Mani's (2010) report, the grinding energy consumption declined from 237 kWh/t for non-treated pine chips to 23-78 kWh/t for torrefied pine chips which was torrefied under 300°C for 30 minutes. Chen et al. (2011) suggests that the optimized condition for better biomass grindability is the torrefied temperature of 250℃ and residence time of 1 hour.

Biomass properties could also be improved by physical treatment such as densification, which is compacting biomass to be denser and have a stable shape for handling and transportation. Pelletizing, as an effective method of densification, is a process in which biomass is densified under high pressure and temperature into a solid cylindrical shape with the dimension of 4.8-19.0 mm in diameter and 12.7-25.4 mm length (Kaliyan & Morey, 2009a). Biomass as raw material for pellets is usually low-value biomass such as sawdust, small dismeter trees, and wood chips, which maximize the efficiency of green energy. Through pelletizing, the bulk density of biomass can be increased dramatically from 40-200 kg/m^3 to 600-800 kg/m^3 (Kaliyan & Morey, 2009a). The higher bulk and energy density lead to an increment in the calorific value and combustion efficiency of biomass. Biomass pellets can be burned with 78% to 85% of combustion efficiency in pellet stove ("Wood and Pellet Heating," 2012). According to Telmo & Lousada's study (2011), the calorific value of softwood pellets is 19.7-20.4 kJ/kg, while the calorific value of hardwood pellets is 17.6-20.8 kJ/kg. Burning 9 million tons of wood pellets could supply roughly 8.4 million families with electricity in a year ("Biomass Sustainability and Carbon Policy Study," 2010). Furthermore, biomass pellets are clean-burning with lower acid gas emissions compared to fossil fuels, which can largely relieve environmental problems such as greenhouse effect. In addition, biomass could keep lowering the moisture in pellets due to the high compacted form that contains less area for air contact, thus reducing the chances for the hydroxyl groups in hemicellulose and other extractives to absorb water. Because of the lower moisture content in pellets, the heat and cost losses due to water absorption could be sharply reduced. Also, by pelleting, the dust formation during transportation can be reduced because of the compacted small stable uniform volume. Additionally, biomass pellets not only facilitate handling and use, but also save transportation and storage costs. The importance of biomass pellets in the U.S. fuel sector is evident as the production of biomass pellets grew from 1.1 million tons in 2003 to 4.2 million tons in 2008 (Spelter & Toth, 2009). Furthermore, an estimation of the European market indicates the annual growth of biomass pellets will be 25-30% over next 10 years (Spelter & Toth, 2009). The expected expansion of the global pellet market may provide increased potential for the biomass pellets as fuel as the demand is expected to rise to more than 22 million oven-dry tons in 2014 ("Wood Pellet Markets/Trends," 2012).

In the pelletizing process, final biomass pellet quality can be affected by different parameters such as pretreatment to biomass, as well as moisture content and particle size of raw material. Furthermore, die temperature, die length to diameter ratio (L/D ratio), compaction pressure, and pelleting speed (rotations per minute, rpm) also can influence pellet quality. Considering pretreatment, adding a torrefaction step into pellet production is a value-added process that combines the advantages of both torrefaction and pelletizing at one location to further improve biomass properties. Even though torrefied biomass pellets are not widely applied in industrial production at present, torrefied wood pellets still have the potential to compete with non-treated biomass pellets. Bergman (2005) reported that torrefied biomass pellets have the bulk density of 750-850 kg/m^3, the calorific value of 19-22 MJ/kg, and energy density of 15-18.5 GJ/m^3. However, there does exist some limitations when pelleting torrefied biomass. Specifically, torrefied biomass particles are more difficult to compact due to the change of chemical components in torrefaction. Additionally, torrefaction itself also consumes energy. Moreover, the pelletizing process requires more energy to compress torrefied biomass with increasing the severity of torrefaction (Stelte et al., 2013). According to Stelte et al., (2013), at the pelletizing temperature of 125 °C and the compaction pressure of 300 MPa for 10 s, torrefied rice straw pellet strength and density decrease with increasing torrefaction temperature. This indicates higher die temperature or pressure would be helpful to improve the strength of torrefied pellet. However, adding die temperature also increases the energy consumption and costs in pelletizing process. In manufacturing, pelletizing die temperature cannot be held too high as the pellet surface would be burned when going through the pelleting die. However, pelleting temperature cannot be too low, as a certain temperature is needed to soften or melt the chemical components acting as binders (e.g., lignin). According to Shaw et al. (2009), biomass pellet density and strength are enhanced at die temperatures from 70°C to 100°C. Moreover, energy requirements of pelletizing could be decreased by increasing the die temperature, but some volatile organic compounds would gasified if the die temperature is over high (Arshadi et al., 2008). Based on Kaliyan & Morey (2009b) research, the optimum die temperature for grass-type biomass in densification is higher than 75 ℃ in laboratory scale. According to Larsson & Rudolfsson’s (2011) study, die temperature of 30 to 45 ℃ is suitable for grass-type pellet production in industrial scale pellet mill.

Compaction pressure is also a key parameter in biomass pelleting and should be maintained to optimize efficiency and economic return. Although higher compaction pressure can increase pellet bulk density, Rhen et al., (2005) suggests that the pressure need not be over 50 MPa when using single pellet die. According to Oporto’s study the strength of torrefied biomass pellets increases significantly when the die temperature is above 177 °C (350°F) and the highest strength pellets are formed at the die temperature of 193°C (380 °F). Nevertheless, unlike the large effect of die temperature, pellet strength only slightly improved when the die pressure was above 4.45 kN (1000 lbf). Thus, considering saving the energy of pelletizing pressure in the process as well as higher strength, the better compaction condition for torrefied biomass was found to be a compaction pressure of 4.45 kN and a die temperature of 193°C when using a single pellet die. Additionally, adding binders such as lignosulfonate and corn starch into torrefied material may be helpful for bonding torrefied particles together and reducing the energy consumption of pelletizing.

Other parameters such as moisture content and particle size of raw material may affect pellet quality. In most European standards, the moisture content is controlled below 10% for pellet production (García-Maraver et al., 2011). Moisture content could affect the pellet properties including bulk density, strength, energy requirement, or durability. According to Mani et al. (2006), pellet bulk density can be increased by decreasing moisture content (from 15% to 12%) and particle size (from 3.2 mm to 0.8 mm). Additionally, pellet strength can be improved by reducing moisture content (from 12.1% to 11.3%). However, reduced moisture content and decreased particle size appear to increase energy consumption during pelletizing because of increased friction between particles (Nielsen et al., 2009). In terms of grass type biomass, past research suggests that pellet durability is optimized in the moisture content range of 9-16% (Theerarattananoon et al., 2010). Relova et al. (2009) suggests that the best particle size range for biomass pellets is 1-2 mm. The single pellet die has been historically used to produce a small quantity of pellets for property testing on a laboratory scale. Parameters such as moisture content or particle size can be optimized in lab then applied on larger scale production.

Since the torrefied pellet production is relatively new in terms of commercialization, it is worthy to investigate the better conditions for the best quality torrefied pellets first based on the laboratory scale and then further apply the results to industrial production. In this research, the effect of torrefaction, as a pretreatment to biomass, as well as raw material moisture content and particle size were evaluated on the compaction behavior, calorific value, mechanical properties, and hydrophilic characteristic of pellets. Woody biomass red oak, and grass-type biomass swtichgrass and miscanthus were used in the experiment.

CHAPTER 2: Compaction Behavior, Mechanical Properties, and Moisture Resistance of Torrefied Red Oak Pellets

1. Introduction

Biomass as a renewable energy resource plays an important role in the 21st century. The Renewable Fuel Standard (RFS) (2010) is targeting an increase in biofuel use within the U.S. to 36 billion gallons by 2022 (Schnepf & Yacobucci, 2013). Nevertheless, most biomass is not convenient or economical for handling, transportation, storage, and usage due to disadvantages such as low density and hydrophilic properties. To deal with these problems, pretreatment (e.g. torrefaction, extraction) and densification (e.g. pelleting, briquetting) have been applied to improve biomass properties.

Pelletizing, as an effective method of densification, is a process in which biomass is densified under high pressure and temperature into a solid cylindrical shape with the dimension of 4.8-19.0 mm in diameter and 12.7-25.4 mm length (Kaliyan & Morey, 2009a). Biomass as raw material for pellets is usually small, waste particles such as sawdust, which maximize the efficiency of green energy. Through pelletizing, the bulk density of biomass can be increased dramatically from 40-200 kg/m^3 to 600-800 kg/m^3 (Kaliyan & Morey, 2009a). The higher bulk and energy density lead to an increase in the calorific value and combustion efficiency of biomass. Biomass pellets can be burned with 70% to 83% of combustion efficiency in pellet stove ("Wood and Pellet Heating," 2013). According to Telmo & Lousada (2011), the calorific value of softwood pellets is 19.7-20.4 kJ/kg, while the calorific value of hardwood pellets is 17.6-20.8 kJ/kg. Burning 9 million tons of wood pellets could supply roughly 8.4 million families with electricity in a year ("Biomass Sustainability and Carbon Policy Study," 2010). Furthermore, biomass pellets are clean-burning with lower emissions compared with fossil fuels and can serve as potential fuel replacement for fossil fuels with lower environmental impacts. Additionally, compacted small stable uniform volume of biomass pellets not only facilitate the handling and usage, but also save transportation and storage costs. The importance of biomass pellets in the fuel sector is evident as the production of biomass pellets in the U.S. grew from 1.1 million tons in 2003 to 4.2 million tons in 2008. Furthermore, an estimation of the European market indicates the annual growth of biomass pellets will be 25-30% over next 10 years (Spelter & Toth, 2009). The expected expansion of the global pellet market may provide increased potential for the biomass pellets as fuel as the demand is expected to rise to more than 22 million oven-dry tons in 2014 ("Wood Pellet Markets/Trends," 2012).

In pelletizing process, adding any pretreatment step into pellet production is a value-added process and takes advantages of both pretreatment and pelletizing to improve biomass properties. In recent years, torrefaction has become an attractive advanced pretreatment that heats biomass materials at relatively low temperature of 225-300℃ under atmospheric pressure and in the absence of oxygen (Prins et al., 2006). Biomass properties such as calorific value, grindability, and hydrophobicity can be improved to be more coal-like by torrefaction pretreatment. Torrefied biomass could be burned with coal or without coal, by adding special conditions in pulverized coal boilers to generate as high a heating value as coal (Li et al., 2012).

Various processing parameters influence how well a pellet material performs. Specifically, parameters such as pretreatment methods (e.g. torrefaction), moisture content and particle size of raw biomass material before pelletizing, die temperature and compaction pressure, as well as holing time, have effects on compaction behavior or pellet quality. In this study, torrefaction as pretreatment and moisture content and particle size of woody biomass raw material were investigated to evaluate whether or not they have an influence on final pellet quality. Through this study, compaction energy, calorific value, mechanical strength, and moisture resistance of red oak *(Quercus rubra*) torrefied and non-torrefied pellets were quantified. Additionally, the research allowed for determining the better processing parameters from producing pellets from torrefied and non-torrefied red oak particles.

2. Methods and Materials

2.1 Torrefied Material

Red oak (*Quercus rubra*) was obtained from West Virginia University Resources Forest in the form of seasoned (air-dried) lumber scrap material. The red oak lumber was then chipped using a typical in woods type chipper. The final chip size was variable, but averaged approximately 30 cm in length by 5 cm in width (Figure 2.1). The red oak chips were dried in a laboratory oven (Lindberg/Blue M Vacuum Ovens, Thermo Fisher Scientific Inc., US) at 103±2℃ for 24 hours to measure the moisture content (M.C.) in accordance with ASTM D4442 (method B) using Equation 1. The moisture content of the chips was determined to be 7.5%. The red oak chips were then torrefied at 300℃ for 30 minutes using a specially designed torrefaction unit housed at West Virginia University's Department of Chemical Engineering (Figure 2.2). The torrefied red oak chips (Figure 2.1) were sealed in a plastic bag at room temperature until further milling was performed.

$$\text{M.C.}\,(\%) = \frac{\text{Wet Weight (g)} - \text{Dry Weight (g)}}{\text{Dry Weight (g)}} \times 100\% \qquad \text{Equation 1}$$

Figure 2.1 *Quercus rubra* chips before torrefaction (left) and after torrefaction (right).

Figure 2.2 Sealed torrefaction chamber.

The torrefied chips were then ground using a Pulverisette 25 power cutting mill (Fritsch) fitted with a 1mm sieve. The milled particles were then further sieved by using a W.S. Tyler test sieve (Mentor, OH, USA) fitted with 1 mm, 0.7 mm, and 0.5 mm sieves. The sieving process was performed for 3 minutes and produced particles in two size ranges of 0.5-0.7 mm and 0.7-1 mm based on our previous study (Oporto et al.). The non-torrefied red oak material was milled and sieved using the same method as the torrefied material. The milled particles were sealed in different bags separately according to their particle size and pretreatment types and stored until needed for the pelleting/compaction studies.

2.2 Densification Procedure

A specially designed single pellet die apparatus was used for manufacturing pellets (Figure 2.3). The pellet die was placed onto MTS (MT Systems Corp. USA) universal test machine (UTM) (Figure 2.4) that applied compaction force. The diameter of cylindrical die hole was 6 mm. The pelletizing temperature was controlled by an Omega PID controller attached to a thermocouple that monitored die temperature. The metal die was wrapped with a heat tape that was covered by thermal insulation. Different types of non-torrefied and torrefied red oak materials with different moisture content and particle size were used for pellet production. Each specific sample type is shown in Table 2.1. Specifically, two levels of particle size were 0.5 – 0.7 mm and 0.7 - 1 mm, two levels of moisture content were 1.5% and 5%. The two moisture content levels of the particles were achieved using a CSZ Environmental Chamber set at varying levels of temperature and humidity based on the material. If should be noted that the torrefied particles and non-torrefied particles required different conditions to achieve the desired moisture content level. The pelletizing condition was set at the die temperature of 193℃ (380°F) and compaction pressure of 4.45 kN. These levels were based on our previous study (Oporto et al.), where we found the better temperature for pelletizing torrefied biomass was 193℃ (380°F). Additionally, our prior work indicated that pellet strength did not show any significant difference between the pelletizing pressure of 4.45 kN (1000 lbf) and 6.67 kN (1500 lbf); therefore, 4.45 kN was selected for the compression pressure in this research.

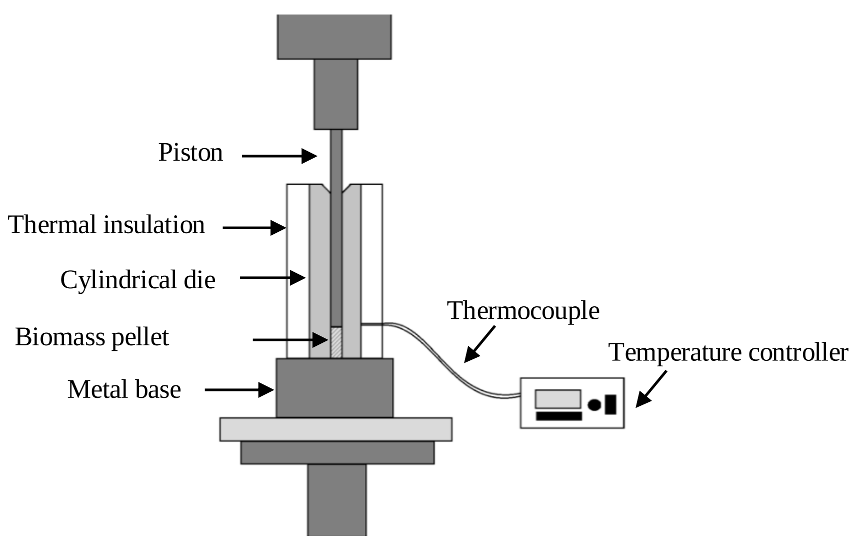

Figure 2.3 Schematic of the specially designed single pellet die.

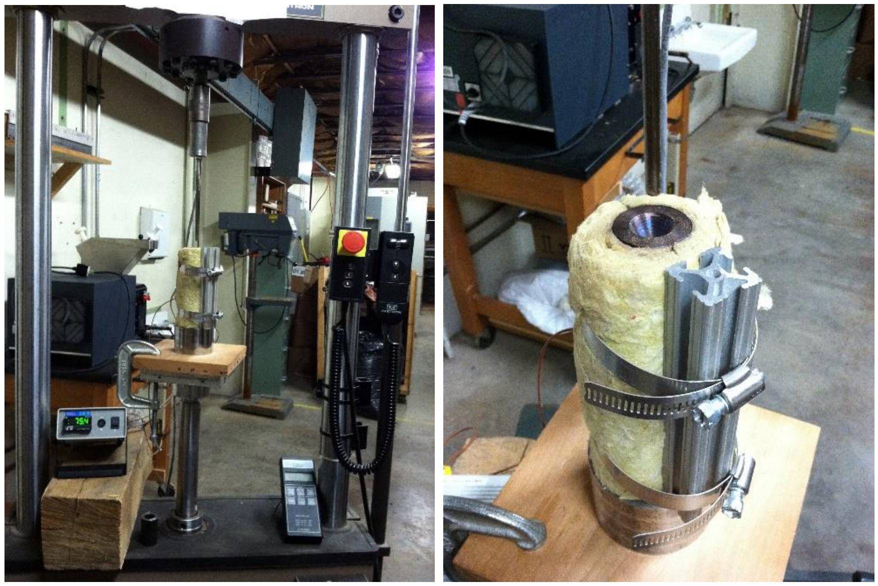

Figure 2.4 Single pellet die in place on the MTS UTM.

Table 2.1 Sample names for different red oak materials with different moisture content and particle size in the experiment.

Sample Name	Pretreatment	Particle Size (mm)	Moisture Content (wt%, d.b.)
RO_0.7-1_5%	Non-Torrefied red oak	0.7-1.0	5
RO_0.7-1_1.5%	Non-Torrefied red oak	0.7-1.0	1.5
RO_0.5-0.7_5%	Non-Torrefied red oak	0.5-0.7	5
RO_0.5-0.7_1.5%	Non-Torrefied red oak	0.5-0.7	1.5
TRO_0.7-1_5%	Torrefied red oak	0.7-1.0	5
TRO_0.7-1_1.5%	Torrefied red oak	0.7-1.0	1.5
TRO_0.5-0.7_5%	Torrefied red oak	0.5-0.7	5
TRO_0.5-0.7_1.5%	Torrefied red oak	0.5-0.7	1.5

For each pellet, 0.7 g of biomass particles was added into the die hole. The particles were then loaded using a piston attached to a moving crosshead of the MTS UTM. Loading occurred at pelletizing pressure rate of 12.7 mm/min. When the pressure reached the target pressure of 4.45 kN, the pellet was held in the die for 3 minutes. This level of holding time was determined during our previous study (Oporto et al.) where we found that 3 minutes was a sufficient time for making a pellet when using a constant pressure of 4.45 kN. Control of the pelleting cycle was performed through a BlueHill (Instron) software routine that controlled the UTM. The pelletizing force and displacement (e.g. compressed length) data was recorded by the BlueHill system computer and used to study compaction behavior. After the 3 minute holding time was completed, the pellet was pushed out of the die and cooled down to the room temperature. The target length of pellets was 2 mm. The pellets were sealed in plastic bags for storage. The compaction behavior of interest in this study was the compaction energy requirement during pelletizing. The compaction energy of a pellet was calculated from the area under the curve in the pelletizing force versus displacement graph (Figure 2.5).

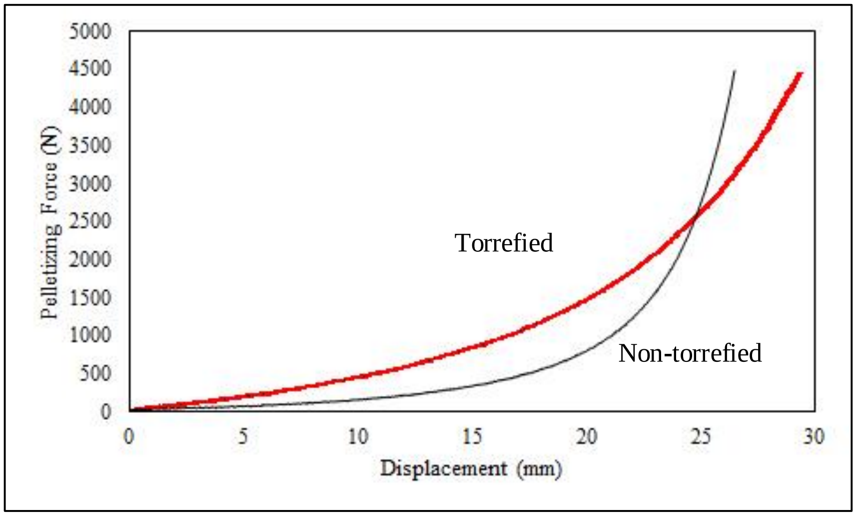

Figure 2.5. Example pelletizing force vs. displacement graph of pelleting non-torrefied red oak material and torrefied red oak material both with the particle size of 0.7-1 mm and moisture content of 5%.

2.3 Gross Heating Value

The gross heat value of the torrefied pellets was determined using a Parr Bomb Calorimeter (6400, Parr Instrument Company USA). Each pellet was weighed by analytical scale with 0.001g accuracy. Approximately 0.5 g sample pellet was placed in the sample cup and attached to a cotton thread which was used for igniting the sample. The gross heating value was measured by the pellet releasing heat during combustion in the bomb cylinder of the calorimeter. The average value of each set was calculated based on 3 pellets. Since the variance of gross heating value was found to be insignificant, 3 repetitions for each type of sample were deemed acceptable.

2.4 Mechanical Testing

The hardness (or diametric compressive resistance) of biomass pellets was evaluated by compression testing (Figure 2.6). Compression testing was performed using a MTS UTM (Figure 2.7). Before the compression test, the dimensions and weight of each pellet were respectively measured by digital caliper with 0.01 mm accuracy and by analytical scale with 0.001g accuracy. The density of the tested pellet could be calculated as Equation 2.

$$\text{Pellet Density}\left(\frac{\text{g}}{\text{m}m^3}\right) = \frac{\text{Pellet weight (g)}}{\text{Pellet volume (mm}^3\text{)}} = \frac{\text{Pellet weight (g)}}{\frac{\pi}{4}\times(\text{pellet diameter(mm)})^2\times\text{pellet length (mm)}} \qquad \text{Equation 2}$$

The pellet was then placed horizontally on a flat metal surface. Compression load was then applied to the pellet in a diametrical direction through the MTS hydraulic cylinder at a constant rate of 4 mm/min set and maintained by the BlueHill computer software. With the increasing load, the pellet was cracked diametrically. Testing was stopped when the compression load reached the maximum which the pellet could tolerate. The load vs. deformation data were recorded by computer. For each individual type (e.g., the pellet with 0.7 -1 mm particle size and 1.5% moisture content set), 20 repetitions were tested in hardness test and the data was analyzed. Overall, 160 pellets were tested.

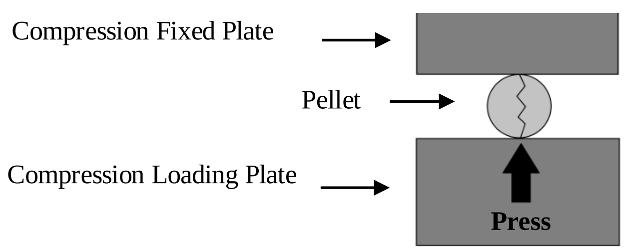

Figure 2.6. Schematic of diametric compression test for pellet hardness.

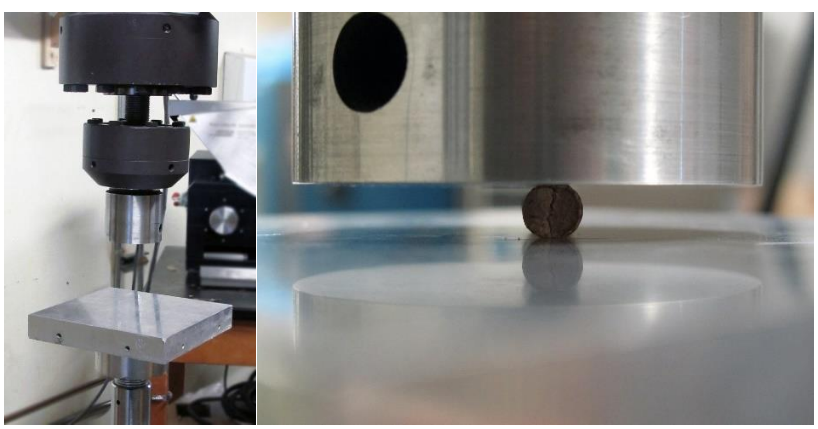

Figure 2.7. Compression test for pellet hardness.

During the study, it was determined that the length of the pellet would impact the overall diametric compression strength. Because the length of the produced pellets varied slightly, hardness was calculated by dividing the maximum compression load by pellet length (Equation 3).

$$\text{Hardness (N/mm)} = \frac{\text{Maximum load (N)}}{\text{pellet length (mm)}} \qquad \text{Equation 3}$$

2.5 Moisture Resistance of Biomass Chips and Pellets

Biomass chips and pellets were both tested for moisture resistance to compare the difference between chips and pellets and to evaluate the influence of pelletizing on the moisture uptake property. Both water immersion and moisture resistance under environmental conditions testing were conducted.

Water Immersion: Both torrefied and non-torrefied wood chips were evaluated for water absorption through immersion in a water bath. Torrefied and non-treated red oak chips (20 g) were first oven-dried at 103±2°C for 24 hours in accordance with ASTM D4442 (method B). After drying, the red oak chips were weighed using an analytical scale (0.001 g resolution) to calculate moisture content using equation 1. As shown in Figure 2.8, chips were fully immersed into a water bath at room temperature in a wood frame covered by mesh tacking on the top and bottom. During the first two hours, the chips were removed from the water bath and weighed every 10 minutes. Prior to weighing, the surface water on the chips was removed by sitting them briefly on a paper towel. Immediately after weighing, the samples were placed back in the water bath. After the first 2 hours, the samples were then left in water for another 22 more hours, then surface dried and weighed. The moisture content was calculated using Equation 1.

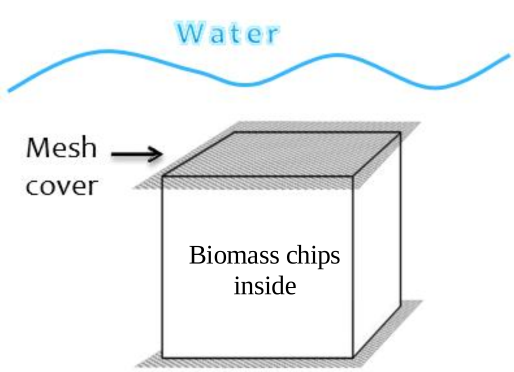

Figure 2.8. Water immersion test for biomass chips.

The water immersion test of torrefied and non-torrefied red oak pellets were tested as the similar method of testing time management as the chips (Figure 2.9). Torrefied and non-torrefied red oak pellets were first oven-dried at 103 ± 2°C for 24 hours then were weighed using an analytical scale (0.001 g resolution) to calculate moisture content using equation 1. The pellets were then placed in the beaker with 40 ml water. During the first two hours, the pellets were removed from the beaker and weighed every 10 minutes. Prior to weighing, the surface water on the pellets was removed by sitting them briefly on filter paper. Immediately after weighing, the pellets were placed back in the water bath. After the first 2 hours, the pellets were then left in water for another 22 more hours, then surface dried and weighed. The moisture content was calculated using Equation 1. Ten pellets for each individual type shown in Table 2.1 were tested and the average moisture content was calculated for each set. In total, 80 pellets were tested in the water immersion test.

Figure 2.9 Water immersion test for biomass pellets.

Moisture Resistance under Environmental Conditions: Both torrefied and non-torrefied wood chips and pellets were evaluated for moisture resistance through environmental conditions by changing target moisture contents. Torrefied and non-treated red oak chips (20 g for each) and pellets (ten pellets for each type shown in Table 2.1) were first oven-dried at 103 ± 2°C for 24 hours. The samples were then placed in a Cincinnati Sub-Zero (CSZ) environmental chamber set to for specific target moisture contents (Table 2.2). These target temperature and relative humidity settings were based off the *Wood Handbook* (2010) values for equilibrium moisture content of wood materials under various conditions. At each level of target moisture content, the samples were weighed every 24 hours. If the sample weight did not change significantly (at 0.05 significance level) from day to day, the environmental condition was changed to next set point. The moisture content for each sample at each condition was determined using equation 1 (with the weight at the condition being the wet weight)

Table 2.2 CSZ chamber settings for the target moisture content used in the experiment ("Wood Handbook," 2010).

Target Moisture Content (%)	Temperature ℃	Relative Humidity (%)
5	37.8	25
7.5	26.7	40
10	21.1	55
12.5	32.2	70
15	37.8	80
17.5	26.7	85
20	26.7	90

2.6 Statistical Analysis

All data were analyzed by OriginLab Data Analysis and Graphing Software (Guangzhou, China).

3. Results and Discussion

3.1 Compaction Energy

In general, it was observed that the area below the curve in Figure 2.5 of torrefied material is larger than the area of non-torrefied material with the same particle size and moisture content. The results of the compaction energy analysis indicated that compacting torrefied material into pellets required more energy than pelletizing non-torrefied material. Table 2.3 and Figure 2.10 show the average compaction energy of pelletizing at different particle sizes and moisture contents. All types of the torrefied red oak materials consumed more than twice of energy than any of the non-torrefied red oak materials in pelletizing. It is likely that the lubricants such as water, hemicellulose, and extractives that were removed by torrefaction caused more friction between die and material (Nielsen et al., 2009).

Table 2.3 Average compaction energy, gross heating value, density, and hardness of non-torrefied and torrefied red oak pellets with different particle sizes and moisture contents.

Sample Name	Compaction Energy (Joule)[a]	Gross Heat (MJ/kg) [a]	Density (kg/m^3) [a]	Hardness (N/mm) [a]
Red Oak		18.2±0.1		
RO_0.7-1_5%	17.7±1.9	19.0±0.1	1344.8±	71.9±19.8
RO_0.7-1_1.5%	13.6±1.8	19.0±0.1	1350.6±	69.7±19.2
RO_0.5-0.7_5%	17.2±2.2	19.4±0.3	1336.6	79.1±26.1
RO_0.5-0.7_1.5%	12.2±1.8	19.2±0.1	1384.0	91.7±21.9
Torrefied Red Oak		23.6±0.7		
TRO_0.7-1_5%	37.5±3.7	24.1±0.0	820.2	11.0±2.1
TRO_0.7-1_1.5%	39.1±4.8	24.1±0.1	836.4	12.6±2.8
TRO_0.5-0.7_5%	38.3±3.7	24.0±0.2	820.3	11.0±1.6
TRO_0.5-0.7_1.5%	38.8±4.5	23.7±0.2	802.6	9.9±1.5

a. Mean±standard deviation.

Based on the results of one-way analysis of variance testing (ANOVA), there was a statistically significant difference within non-torrefied red oak pellet groups (p-value < 0.0001, at α=0.05 significance level). Further analysis using the Tukey-Kramer-Comparison test indicated the non-torrefied materials with higher moisture content of 5% required statistically significant lower average compaction energy during pelletizing than the higher moisture content of 5% samples at both particles size ranges (p-value < 0.0001, at α=0.05 significance level). Figure B2 in Appendix B provides the details on the compaction energy of non-torrefied red oak materials. According to Nielsen et al. (2009) research, moisture content negatively affects pellet properties due to water covering the surface of wood particles, which hinders hydrogen bonding between particles. Thus, more energy was required for activating the coated hydroxyl sites to form bonds between polymer molecules in biomass particles. The non-torrefied materials with larger particle size of 0.7-1 mm required statistically significant higher average compaction energy in densification than the smaller particle size of 0.5-0.7 mm samples at the same moisture content level of 1.5% (p-value < 0.0001, at α=0.05 significance level). At the moisture content of 5% level, the particle size did not show significantly influence on the compaction energy (p-value = 0.7761). Thus the non-torrefied material with the moisture content of 1.5% and particle size of 0.5-0.7 mm consumed the least energy in pelletizing.

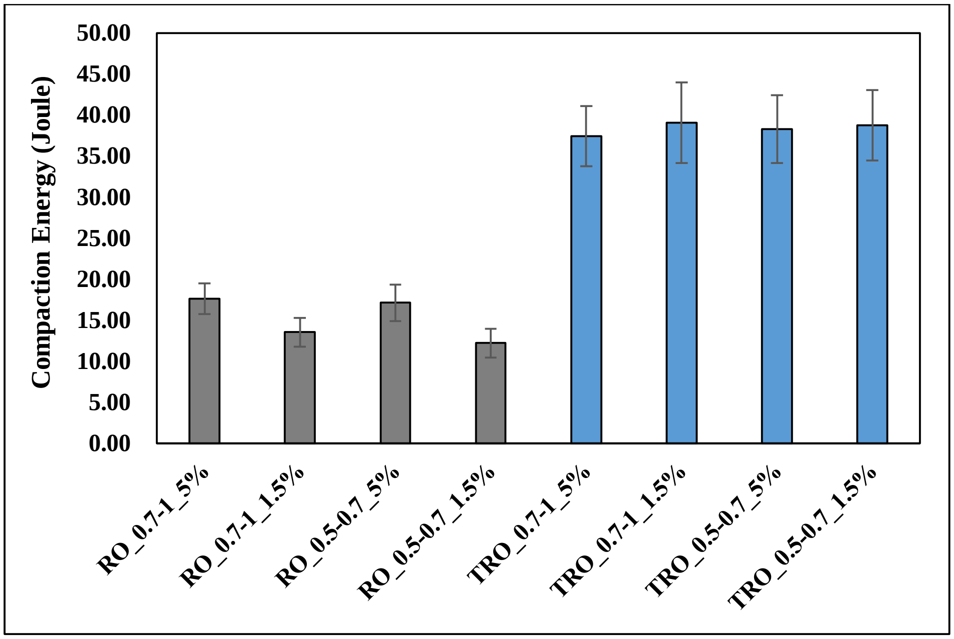

Figure 2.10 Compaction energy for pelleting of non-torrefied and torrefied red oak pellets with different particle size and moisture content.

However, in the torrefied types, there was no statistically significant difference within the four groups (p-value = 0.2509). Thus, the different particle size ranges of 0.7-1 mm and 0.5-0.7 mm and different moisture content of 1.5% and 5% did not have statistically significant influence on the compaction energy of pelletizing torrefied red oak material. This finding of no difference may be a result of the torrefaction removing a large amount of materials that impact the compaction energy of the particles. These results suggest that particle size and moisture content might not be of importance in the torrefied pellet production in terms of compaction energy.

3.2 Gross Heating Value

The weight loss of torrefied red oak material used in the experiment was 44.33%. Figure 2.11 shows the gross heating value of both non-torrefied and torrefied red oak raw particles and pellets. As can be seen from Table 2.3, the gross heating value of the red oak biomass increased from 18.19 MJ/kg to 23.56 MJ/kg after torrefaction, which is close to lignite (ASTM D 5865 – 04). During torrefaction, the carbon content increases due to the reduction of the both hydrogen and oxygen content, which was caused by removing water and decomposing or gasifying the reactive biopolymers (e.g., hemicellulose and volatile extractives) that are abundant with hydroxyl groups

(Stelte et al., 2011). Additionally, the torrefied pellets had higher gross heating value than the non-torrefied pellets.

One-way ANOVA analysis, indicated that there was a statistically significant difference between the gross heating value of non-torrefied particles and pellets (p-value < 0.0001, at $\alpha=0.05$ significance level). The results of the Tukey-Kramer-Comparison tests indicated a statistically significant higher average gross heating value for all four types of non-torrefied pellets when compared to the non-torrefied biomass particles (p-value < 0.0001, at $\alpha=0.05$ significance level). The non-torrefied pellet with smaller particle size of 0.5-0.7 mm had significantly higher gross heating value than the larger particle size of 0.7-1 mm pellets at the same moisture content level of 5% (p-value < 0.0001, at $\alpha=0.05$ significance level). Furthermore, non-torrefied red oak gross heating value was significantly increased by densification (p-value < 0.0001, at $\alpha=0.05$ significance level). This might since the non-torrefied particles were heated in the die hole resulting increasing in the gross heating value. There was, however, no statistically significant difference of gross heating value in the other non-torrefied pellet types (p-value < 0.0001, at $\alpha=0.05$ significance level). Furthermore, there was no statistically significantly difference between the gross heating values of torrefeid biomass particles and pellets (p-value = 0.1792). These findings indicate that the gross heating value of torrefied biomass particles was not improved by densification. This might due to the torrefied materials have already been heated in a higher torrefied temperature. However, there was a statistically significant difference in the gross heating value of torrefied pellet groups (p-value = 0.00966). Specifically, the torrefied pellet with the smaller particle size of 0.5-0.7 mm and lower moisture content of 1.5% had the lower gross heating value than other groups (p-value < 0.0001, at $\alpha=0.05$ significance level). Nevertheless, there was no obviously higher gross heating value in the torrefied red oak pellet types. Thus, for the torrefied biomass pellets, the gross heating value was mostly affected by the severity of torrefaction rather than densification processing.

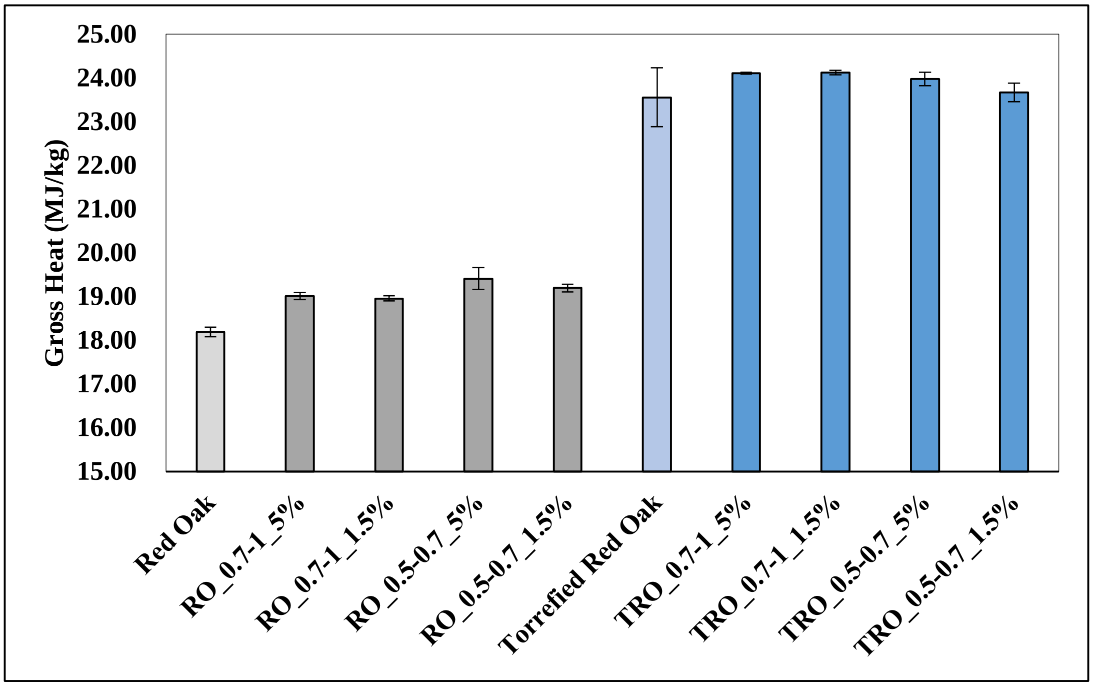

Figure 2.11 Gross heating value of non-torrefied and torrefied raw materials and pellets.

3.3 Hardness

Prior to the hardness test, the average density was calculated and shown in Figure B3 in Appendix B. Figure 2.12 shows the hardness of both non-torrefied and torrefied red oak pellets. As can be seen from Table 2.3, the average hardness of non-torrefied pellets is approximately 7 times larger than the torrefied pellets, which might be due to the density of non-torrefied pellets were sharply higher than the torrefied pellets. The particles in torrefied pellet were less compacted with large gaps between each other. These voids and lower densification resulted in cracking on the side surfaces at low force and resulted in total breakage at low force. In general, the fragility of torrefied pellets resulted in the dramatic reduction of hardness. These results indicate that the red oak as torrefied in this research may not be suitable for durable pellet production without the addition of binders or modification of the pelleting process. Stelte et al. (2013) and Li et al. (2012) also reported the same phenomenon that the torrefied biomass pellet strength and density both decreased as torrefied temperature increase. The non-torrefied particles have stronger linkage than the torrefied particles due to the softened lignin could fill the gaps and pores in the non-torrefied pellet, thus it is easier for torrefied particles to be deformed with pressure. In terms of the pretreatment types of the pellets, only the non-torrefied red oak pellet with the smaller particle size of 0.5-0.7 mm and lower moisture content of 1.5% showed a statistically significant higher average hardness than other pellets with larger particle size (p-value < 0.0001, at α=0.05 significance level). In addition, the average hardness of torrefied red oak pellets did not show any statistically significantly difference within four groups (p-value = 0.0742). This indicates that for torrefied pellets production, moisture content and particle size were not important parameters in terms of pellet hardness.

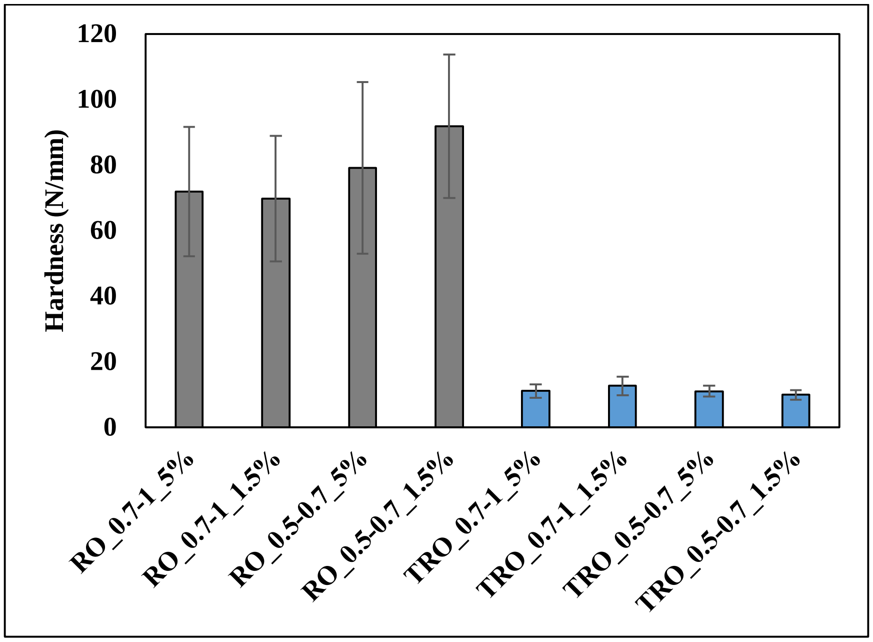

Figure 2.12 Hardness (N/mm) of non-torrefied and torrefied red oak pellets.

3.4 Moisture Resistance: Water Immersion

Figure 2.13 shows the moisture content of non-torrefied and torrefied biomass chips during the water immersion test. It was observed that the torrefied biomass chips absorbed far less water during the water immersion test (moisture content 33%) as compared to the non-torrefied chips (moisture content 76%) at the end of 24 hours. This was most likely due to the hydroxyl functional groups being removed in torrefaction, thus indicating that the torrefied biomass has a lower ability to bond with water molecules resulting better moisture resistance.

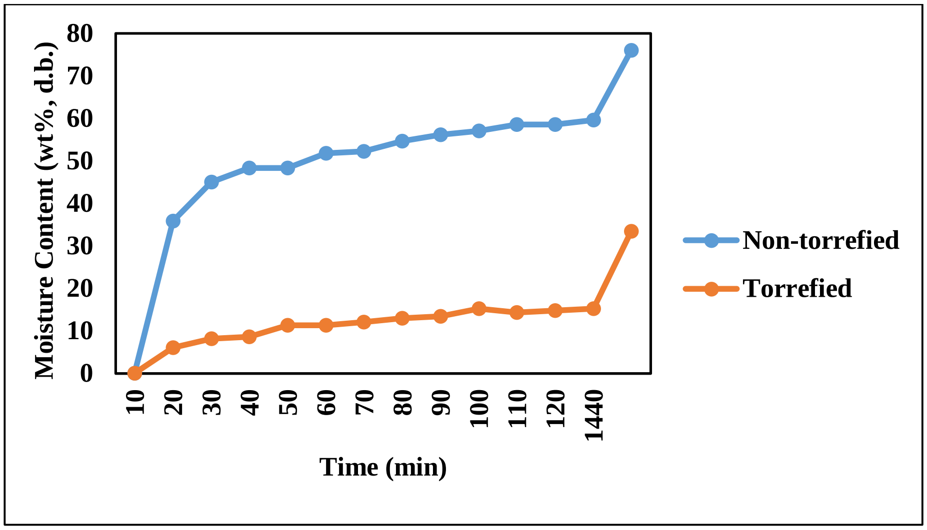

Figure 2.13. Moisture resistance of non-torrefied and torrefied biomass chips in water immersion test.

Figure 2.14 shows the moisture content of non-torrefied and torrefied biomass pellets in water immersion test. The torrefied biomass pellets were found to have a statistically significant lower average moisture content when compared to the non-torrefied pellets (p-value < 0.0001, at α=0.05 significance level). When comparing non-torrefied red oak pellets made at 5% MC, results from the Tukey-Kramer-Comparison test indicated a statistically significant lower average moisture content for the pellets with larger particle size of 0.7-1 mm as compared to the with smaller particle size of 0.5-0.7 mm (p-value < 0.0001, at α=0.05 significance level). When comparing non-torrefied red oak pellets made at 1.5% MC, results from the Tukey-Kramer-Comparison test also indicated a statistically significant lower average moisture content for the pellets with larger particle size of 0.7-1 mm as compared to the with smaller particle size of 0.5-0.7 mm (p-value < 0.0001, at α=0.05 significance level). This phenomenon is most likely due to the larger contact surface area of smaller particles which leads to higher water uptake than the larger particles. In terms of the 0.5-0.7 mm particle size level, the non-torrefied pellets produced at higher moisture content (5%) were found to have a statistically significant lower average moisture content than the non-torrefied pellets produced at the lower moisture content (1.5%) (p-value < 0.0001, at α=0.05 significance level). This might due to the lower material moisture content particles contained more unoccupied hydroxyl groups which could bond more water molecules on the surface than the higher material moisture content particles. However, at the same particle size level of 0.7-1 mm, the moisture content of the non-torrefied pellets did not show any statistically significantly difference between two levels of material moisture content of 5% and 1.5% (p-value=0.78035).

Similar to the non-torrefied pellets, at both levels of the pellet making moisture content of 5% and 1.5%, torrefied pellets with larger particle size of 0.7-1 mm showed a statistically significantly lower average moisture content than the torrefied pellets with smaller particle size of 0.5-0.7 mm (p-value < 0.0001, at α=0.05 significance level). At both particle size levels of 0.5-0.7 mm and 0.7-1 mm, the moisture content of the torrefied pellets with higher material moisture content of 5% was statistically significantly smaller than the moisture content of the torrefied pellets with lower material moisture content of 1.5% (p-value < 0.0001, at α=0.05 significance level).

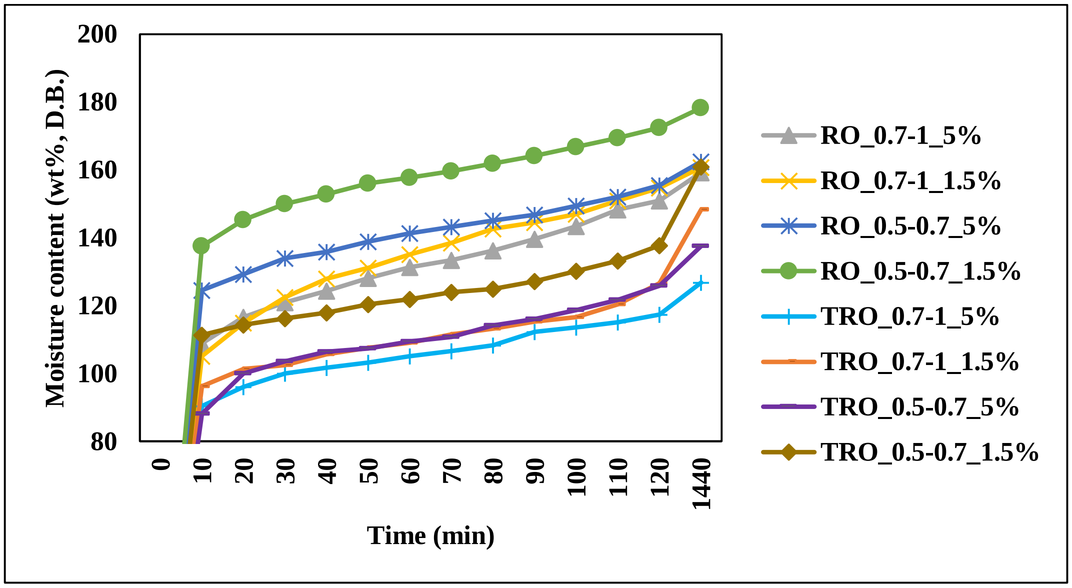

Figure 2.14 Moisture resistance of non-torrefied and torrefied red oak pellets in water immersion test.

Figure 2.15 shows the comparison of moisture content of non-torrefied and torrefied red oak chips and pellets (0.7-1 mm particle size and 5% moisture content) in water immersion test. From these results, it appears that the moisture content of both non-torrefied and torrefied pellets were far higher than the chips. This is again likely due to the much larger contact surface area for the pellets, as compared to the chips. Thus, it is necessary to protect pellets from water for both non-torrefied and torrefied biomass. More details on the individual values from the water absorption tests are shown in Table A1 in Appendix A.

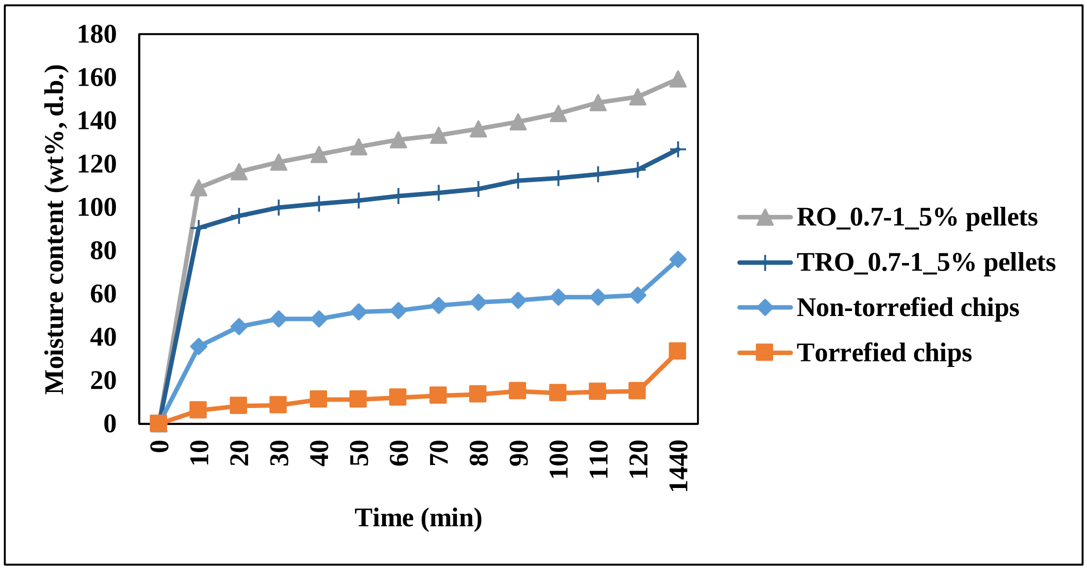

Figure 2.15 Moisture resistance of non-torrefied and torrefied red oak chips and pellets (0.7-1 mm particle size and 5% moisture content) in water immersion test.

3.5 Moisture Resistance: Environmental Conditioning

Figure 2.16 show the moisture content of non-torrefied and torrefied biomass chips in the environmental chamber conditioning test. Table A2 in Appendix A provides the details on specific values for each type of chips at each condition. In general, the results of the environmental tests indicated that there is a difference between the moisture contents of non-torrefied and torrefied red oak chips in after the 5% M.C. level. Non-torrefied biomass chips absorbed more moisture in the atmosphere than the torrefied material, especially under higher target moisture content conditions. Thus, the difference of moisture content between non-torrefied and torrefied biomass chips became larger as the target moisture content increased. Specifically, torrefied biomass chips moisture content never exceeded 10% even under as high as 20% target moisture content, which is far better than the untreated biomass chips that rose to a 25% moisture content. The reduction of moisture content of the torrefied chips is most likely to be explained by the decomposition of hemicellulose resulting the decreasing of hydroxyl groups in biomass which had the function of bonding water. Thus the torrefied biomass showed a lower tendency to absorb water than non-torrefied biomass.

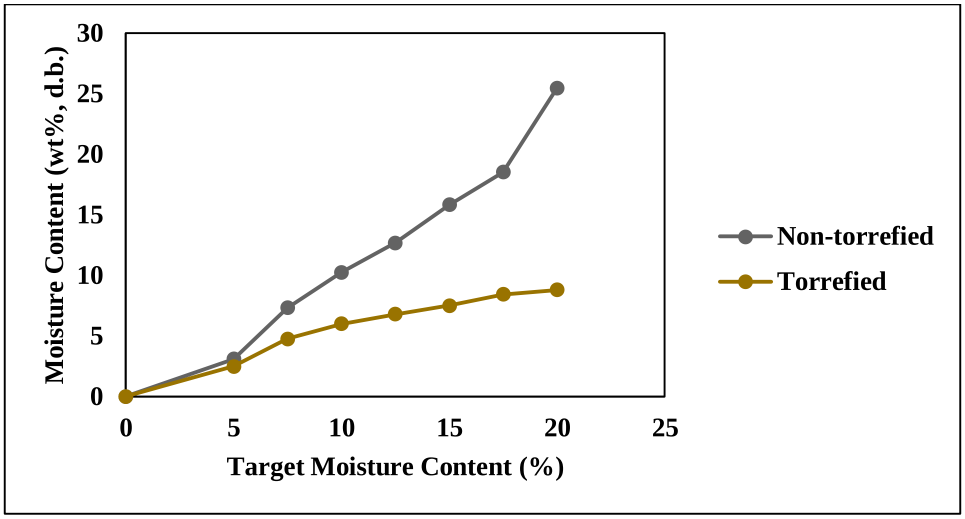

Figure 2.16 Moisture resistance of non-torrefied and torrefied biomass chips in chamber conditioning test.

Figure 2.17 shows the moisture content of non-torrefied and torrefied red oak pellets in the environmental chamber conditioning test. The moisture content of torrefied pellets were significantly lower than the non-torrefied pellets especially under higher target moisture content which was similar to the trend presented in Figure 2.16. The moisture content of torrefied pellets of four types were all 7% under the target moisture content of 20%, which were lower than the moisture content of 11% of non-torrefied pellets. Moreover, in the non-torrefied pellet groups, the pellets with larger particle size of 0.7-1 mm absorbed more water than the smaller particle pellets after the target moisture content over 15%. This might due to the smaller particle size were more compacted with less air flow within the pellet, thus moisture had a more difficult time moving into the pellets. The moisture content of the red oak particles at the time time of pelleting (1.5% and 5%) did not show any significant effect on the non-torrefied pellet moisture uptake. Furthermore, the moisture content of four torrefied biomass pellet groups were nearly the same, as they were not affected by either the moisture content at the time of pelleting or the particle size of pellet material.

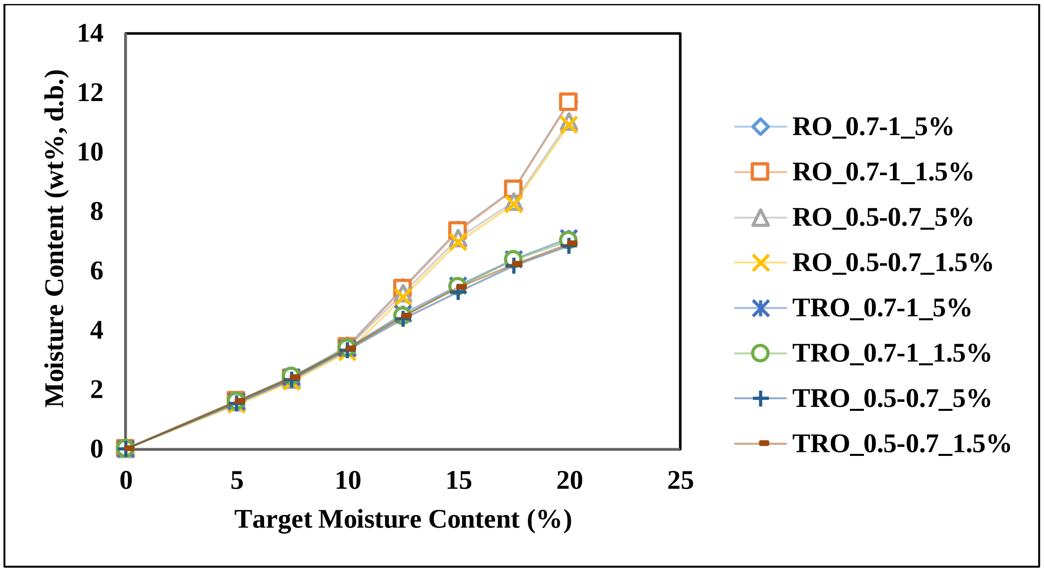

Figure 2.17 Moisture content of non-torrefied and torrefied red oak pellets in chamber conditioning test.

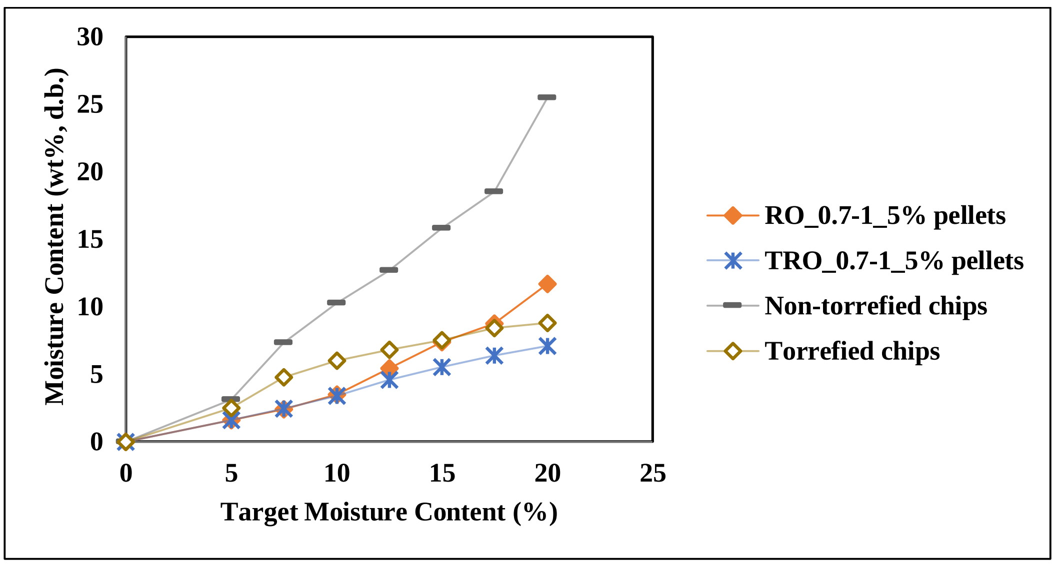

Figure 2.18 Moisture resistance of non-torrefied and torrefied red oak chips and pellets (0.7-1 mm particle size and 5% moisture content) in chamber conditioning test.

Figure 2.18 shows the comparison of the moisture content of chips and pellets of non-torrefied and torrefied biomass. These results show that both torrefied and non-torrefied pellets moisture resistance were improved by densification, as the moisture content of the pellets is much lower than the chips. At the target moisture content of 20%, the moisture content of non-torrefied pellets was 11%, which was dramatically lower than the moisture content of 25% of non-torrefied chips. Similarly, the moisture content of torrefied chips was higher at each condition, as compared to the torrefied pellets. Thus, pelletizing could improve the moisture resistance properties of the both non-torrefied and torrefied biomass.

3.6 Moisture Resistance: Visual Appearance

Figure 2.19, respectively shows the appearance of non-torrefied and torrefied pellets with the same particle size of 0.7-1 mm and moisture content of 5% before test, under the 20% target moisture content conditioning, and after water immersion for 24 hours. The non-torrefied pellets expanded considerably more in length and lost more particles than the torrefied pellets. The torrefied pellets shape did not change significantly under high target moisture content conditions. Furthermore, the torrefied pellets retained their shape better as compared to the non-torrefied pellets that appeared to break apart during the 24 hours of water immersion. Thus, the torrefied pellets presented better moisture resistance from an appearance standpoint.

Type	Before Moisture Testing	At 20% MC Target	After 24 Hour Water Immersion
Non Torrefied			
Torrefied			

Figure 2.19 Water absorption and environmental conditions test visual results on red oak pellets produced at 5% MC and a 0.7-1 mm particle size.

4. Conclusions

Biomass properties could be improved by torrefaction and pelletizing including higher calorific value, higher density, and less hydrophilic property. Thus the torrefied biomass pellets showed better moisture resistance than the non-torrefied pellets in both of the target moisture conditioning and water immersion tests. However, the torrefied biomass required more compaction energy in pelletizing. Additionally, torrefaction process also consumed energy as heating the biomass. Moreover, the hardness of the torrefied pellets were lower than the untreated pellets due to the decomposition of hemicellulose and part of cellulose, lignin and extractives in biomass. The particle size and moisture content of raw biomass material also had some effects on the pellet properties. Based on the results in the experiment, the material with smaller particle size of 0.5-0.7 mm and lower moisture content of 1.5% could be the better condition for non-torrefied biomass in pellet production since this type of pellet has the highest hardness, density, moisture resistance, relatively high gross heating value and the lowest compaction energy requirement. Since there was no statistically significant difference within the compaction energy, calorific value, hardness, as well as the moisture uptake in the chamber conditioning test of torrefied biomass pellet groups, the larger particle size of 0.7-1 mm and higher moisture content of 5% are the relatively better conditions for the torrefied biomass pellet according to the best moisture resistance in the water immersion test.

CHAPTER 3: Compaction Behavior, Mechanical Properties, and Moisture Resistance of Torrefied Miscanthus and Switchgrass Pellets

1. Introduction

Biomass as sustainable energy resource is used as feedstock to heat or generate power, as well as serving as a raw material for bio-diesel. Due to the limited fossil fuel resources and implications of using fossil fuels on greenhouse gas emissions, biomass is expected to play an increasing role in the energy sector, especially given biomass's renewable nature. Swtichgrass *(Panicum virgatum*), a native North America perennial warm season grass, could be an excellent biofuel energy crop as it has a high yield, is well adapted to various low-quality soils, and has a relatively low cost to grow (Mann et al., 2009). Furthermore, *Miscanthus*, a genus including 17 species of perennial tall grasses native to subtropical and tropical Asia, could also serve as another desirable energy crop. Specifically, miscanthus has high yield potential, a low maintenance cost, and a relatively high heating value attributed to its high cellulose content (Brosse et al., 2012). In 2012, the U.S. Department of Agriculture (USDA) and Navy and Department of Energy offered more than $30 million as investments for research of industrializing advanced drop-in biofuels. However, the low bulk density (e.g. 100-200 kg/m^3) of grass types of biomass has been found to be the main disadvantage, as its properties result in inconvenient handling and high transportation and storage costs.

Additionally, as biomass crops lose weight and energy content (through decomposition) during storage, they begin to release greenhouse gasses (Emery and Mosier, 2012). Therefore, to deal with these transportation and storage related problems, pretreatment (e.g. torrefaction, extraction) and densification (e.g. pelleting, briquetting) have been applied to improve biomass properties such as bulk density and calorific value. Torrefaction, could significantly improve biomass properties by decomposing hemicellulose and a proportion of cellulose, lignin and other extractives (Prins et al., 2006). Specifically, torrefaction preatreatment that heats biomass at temperatures between of 225-300℃ under atmospheric pressure and in the absence of oxygen could significantly improve biomass properties by decomposing hemicellulose and a proportion of cellulose, lignin and other extractives (Prins et al., 2006). Nearly 40% of hemicellulose is decomposed at the temperature of 260 and accounts for the majority of the biomass weight loss, as compared to 5% of cellulose and 3% of lignin (Chen et al., 2011).

Since hemicellulose is the most reactive component, it contains a large amount of hydroxyl groups which results in biomass's hydrophilic nature. By removing the most hydrophilic biomass material through torrefaction, there is potential to improve storage and handling properties. The carbon content also increases after torrefaction because of the decrease in hydrogen and oxygen content. Due to the higher carbon content torrefied biomass's calorific value can be increased to be closer to coal. In terms of energy content, during torrefaction, the removed chemical components are gasified causing weight loss to biomass. According to Bergman's (2005) report, 30% of the biomass mass with only 10% energy was lost in torrefaction, thus 90% energy was retained in the 70% of the mass, which indicates that the energy density was increased 1.3 times after torrefaction. Torrefied biomass also would then have more coal-like properties such as better grindability. Given the coal-like properties, torrefied biomass could then be used as biofuel to be gasified or co-combusted with coal or by itself to generate energy. However, to successfully transport torrefied biomass requires densification. Pelletizing has the potential to be an effective densification method to compact torrefied biomass and further increase both bulk and energy density. Specifically, through densification, the torrefied material could be compacted into small standard cylindrical shapes with higher strength and lower moisture content. However, pelletizing of torrefied biomass can be more energy intensive. Moreover, energy requirements of pelletizing could be decreased by increasing the die temperature, but some volatile organic compounds would gasified if the die temperature is over high (Arshadi et al., 2008). Based on Kaliyan & Morey (2009b) research, the optimum die temperature for grass-type biomass in densification is higher than 75 ℃ in laboratory scale. According to Larsson & Rudolfsson's (2011) study, die temperature of 30 to 45 °C is suitable for grass-type pellet production in industrial scale pellet mill.

In the previous study in Chapter 1, *Quercus rubra* (Red oak) as representative of woody biomass was studied as raw material for pellets. In this Chapter, the focus is on crop-based biomass. Specifically, *Miscanthus* and *Panicum virgatum* (Switchgrass) grass type biomass were investigated to determine the effects of torrefaction pretreatment, raw material moisture content and particle size on the properties of biomass pellets. During this study, compaction behavior, calorific value, mechanical properties, and moisture resistance were evaluated for both torrefied and non-torrefied miscanthus and switchgrass. The object is to determine the better moisture content and particle size for both torrefied and non-torrefied grass-type pellet production.

2. Methods

2.1 Torrefied Material

Panicum virgatum (Switchgrass) and *Miscanthus giganteus* (hybrid of *Miscanthus sinensis* and *Miscanthus sacchariflorus*) were obtained from material being studied at West Virginia University for use on marginal land. Since the miscanthus and switchgrass raw materials would be burned in higher torrefied temperature, they were respectively torrefied at 230 ℃ and 235 ℃ for 30 minutes using a specially designed torrefaction unit housed at West Virginia University's Department of Chemical Engineering (Figure 3.1). The torrefied miscanthus and switchgrass feedstock (Figure 3.2) were sealed in a plastic bag at room temperature until milling was performed.

Figure 3.1 Sealed torrefaction chamber.

Type	Before Torrefaction	After Torrefaction
Miscanthus chips		
Switchgrass chips		

Figure 3.2 Miscanthus and switchgrass chips before torrefaction and after torrefaction.

The non-torrefied miscanthus and switchgrass chips were then respectively ground using a Pulverisette 25 power cutting mill (Fritsch) fitted with a 1 mm sieve. The milled particles were then further sieved by using a W.S. Tyler test sieve (Mentor, OH, USA) fitted with 1 mm, 0.7 mm, and 0.5 mm sieves. The sieving process was performed for 3 minutes and produced particles in two size ranges of 0.5-0.7 mm and 0.7-1 mm. The milled particles were dried in a laboratory oven (Lindberg/Blue M Vacuum Ovens, Thermo Fisher Scientific Inc., US) at 103±2℃ for 24 hours to measure the moisture content (M.C.) in accordance with ASTM D4442 (method B) using Equation 1 (Chapter 1). The moisture content of the chips was determined to be 6.4% and 7.8% respectively. The dried chips were sealed in different bags separately according to their particle size and species. The torrefied miscanthus and switchgrass chips were respectively milled and sieved using the same method as the non-torrefied materials. The particles were then sealed in different bags separately according to their particle size, species, and pretreatment types and stored until needed for the pelleting/compaction studies.

2.2 Densification Procedure

A specially designed single pellet die apparatus was used for manufacturing pellets (Figure 2.3, Chapter 1). The pellet die was placed onto MTS (MT Systems Corp. USA) universal test machine (UTM) (Figure 2.4, Chapter 1) that applied compaction force. The diameter of cylindrical die hole was 6 mm. The pelletizing temperature was controlled by an Omega PID controller attached to a thermocouple that monitored die temperature. The metal die was wrapped with a heat tape that was covered by thermal insulation. Different types of non-torrefied and torrefied miscanthus and switchgrass materials with different moisture content and particle size were used for pellet production. Each specific sample type is shown in Table 3.1. Specifically, two levels of particle size were 0.5 – 0.7 mm and 0.7 - 1 mm, two levels of moisture content of non-torrefied materials were 5% and 12%, as well as two levels of moisture content of torrefied materials were 1.5% and 5%. The two moisture content levels of the particles were achieved using a CSZ Environmental Chamber set at varying levels of temperature and humidity based on the material. If should be noted that the torrefied particles and non-torrefied particles required different conditions to achieve the desired moisture content level.

The pelletizing condition was set at the die temperature of 121℃ (250°F) and compaction pressure of 4.45 kN. The force level was based on the values used in Chapter 1. Additionally, through preliminary testing it was determined that the miscanthus and switchgrass materials would burn in the die if the die temperature was set as high as red oak materials in Chapter 1. Furthermore, we also found that the torrefied miscanthus and switchgrass materials with weight loss of 20-30% could be compacted into good shape pellet under die temperature of 121℃.

Table 3.1 Sample names for different miscanthus and switchgrass materials with different moisture content and particle size.

Sample Name	Pretreatment	Particle Size (mm)	Moisture Content (%, d.b.)
M_0.7-1_5%	Non-Torrefied Miscanthus	0.7-1.0	5
M_0.7-1_12%	Non-Torrefied Miscanthus	0.7-1.0	12
M_0.5-0.7_5%	Non-Torrefied Miscanthus	0.5-0.7	5
M_0.5-0.7_12%	Non-Torrefied Miscanthus	0.5-0.7	12
TM_0.7-1_1.5%	Torrefied Miscanthus	0.7-1.0	1.5
TM_0.7-1_5%	Torrefied Miscanthus	0.7-1.0	5
TM_0.5-0.7_1.5%	Torrefied Miscanthus	0.5-0.7	1.5
TM_0.5-0.7_5%	Torrefied Miscanthus	0.5-0.7	5
SG_0.7-1_5%	Non-Torrefied Switchgrass	0.7-1.0	5
SG_0.7-1_12%	Non-Torrefied Switchgrass	0.7-1.0	12
SG_0.5-0.7_5%	Non-Torrefied Switchgrass	0.5-0.7	5
SG_0.5-0.7_12%	Non-Torrefied Switchgrass	0.5-0.7	12
TSG_0.7-1_1.5%	Torrefied Switchgrass	0.7-1.0	1.5
TSG_0.7-1_5%	Torrefied Switchgrass	0.7-1.0	5
TSG_0.5-0.7_1.5%	Torrefied Switchgrass	0.5-0.7	1.5
TSG_0.5-0.7_5%	Torrefied Switchgrass	0.5-0.7	5

For each pellet, 0.7 g of biomass particles was added into the die hole. The particles were then loaded using a piston attached to a moving crosshead of the MTS UTM. Loading occurred at pelletizing pressure rate of 12.7 mm/min. When the pressure reached the target pressure of 4.45 kN, the pellet was held in the die for 3 minutes. This level of holding time was kept the same as used in Chapter 1. Control of the pelleting cycle was performed through a BlueHill (Instron) software routine that controlled the UTM. The pelletizing force and displacement (e.g. compressed length) data was recorded by the BlueHill system computer and used to study compaction behavior. After the 3 minute holding time was completed, the pellet was pushed out of the die and cooled down to the room temperature. The target length of pellets was 2 mm. The pellets were sealed in plastic bags for storage. The compaction behavior of interest in this study was the compaction energy requirement during pelletizing as in the previous (Chapter 1). The compaction energy of a pellet was calculated from the area under the curve in the pelletizing force versus displacement graph (Figure 3.3 and Figure 3.4).

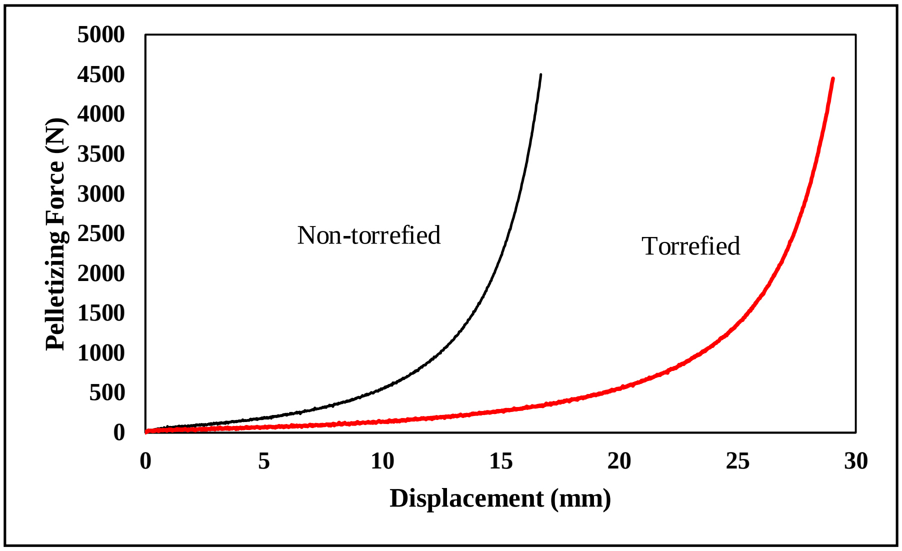

Figure 3.3 Example pelletizing force vs. displacement graph of pelleting non-torrefied and torrefied miscanthus material both with the particle size of 0.7-1 mm and moisture content of 5%.

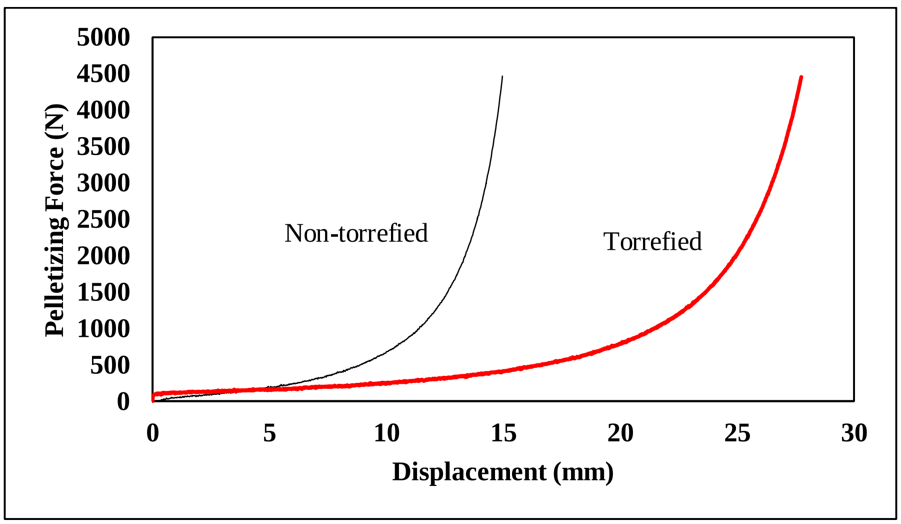

Figure 3.4 Example pelletizing force vs. displacement graph of pelleting non-torrefied and torrefied switchgrass material both with the particle size of 0.7-1 mm and moisture content of 5%.

2.3 Gross Heating Value

The gross heat value of the torrefied pellets was determined using a Parr Bomb Calorimeter (6400, Parr Instrument Company USA). Each pellet was weighed by analytical scale with 0.001g accuracy. Approximately 0.5 g sample pellet was placed in the sample cup and attached to a cotton thread which was used for igniting the sample. The gross heating value was measured by the pellet releasing heat during combustion in the bomb cylinder of the calorimeter. The average value of each set was calculated based on 3 pellets. Since the variance of gross heating value was found to be insignificant, 3 repetitions for each type of sample was deemed acceptable.

2.4 Mechanical Testing

The hardness (or diametric compressive resistance) of biomass pellets was evaluated by compression testing (Figure 2.6, Chapter 1). Compression testing was performed using a MTS UTM (Figure 2.7, Chapter 1). Before the compression test, the dimensions and weight of each pellet were respectively measured by digital caliper with 0.01 mm accuracy and by analytical scale with 0.001g accuracy. The density of the tested pellet could be calculated as Equation 2(Chapter 1). The pellet was then placed horizontally on a flat metal surface. Compression load was then applied to the pellet in a diametrical direction through the MTS hydraulic cylinder at a constant rate of 4 mm/min set and maintained by the BlueHill computer software. With the increasing load, the pellet was cracked diametrically. Testing was stopped when the compression load reached the maximum which the pellet could tolerate. The load vs. deformation data were recorded by computer. During the study, it was determined that the length of the pellet would impact the overall diametric compression strength. Because the length of the produced pellets varied slightly, hardness was calculated by dividing the maximum compression load by pellet length (Equation 3, Chapter 1). For each individual type (e.g., the pellet with 0.7 -1 mm particle size and 1.5% moisture content set), 20 repetitions were tested in hardness test and the data was analyzed. Overall, 320 pellets were tested.

2.5 Moisture Resistance of Biomass Particles and Pellets

Biomass particles and pellets were both tested the moisture resistance to compare the difference between uncompact particles and pellets and to evaluate the influence of pelletizing on the moisture uptake property. Environmental conditions testing were used to evaluate the water vapor uptake of biomass.

Moisture Resistance under Environmental Conditions: Both torrefied and non-torrefied miscanthus and switchgrass particles and pellets were evaluated for moisture resistance through environmental conditions by changing target moisture contents. Torrefied and non-treated miscanthus and switchgrass particles (3 g for each) and pellets (3 pellets for each type shown in Table 3.1) were first oven-dried at 103±2℃ for 24 hours. The samples were then placed in a Cincinnati Sub-Zero (CSZ) environmental chamber set to for specific target moisture contents (Table 3.2). These target temperature and relative humidity settings were based off the *Wood Handbook* (2010) values for equilibrium moisture content of wood materials under various conditions. At each level of target moisture content, the samples were weighed every 24 hours. If the sample weight did not change significantly (at 0.05 significance level) from day to day, the environmental condition was changed to next set point. The moisture content for each sample at each condition was determined using Equation 1 (with the weight at the condition being the wet weight)

Table 3.2 CSZ chamber settings for the target moisture content used in the experiment ("Wood Handbook," 2010).

Target Moisture Content (%)	Temperature ℃	Relative Humidity (%)
5	37.8	25
7.5	26.7	40
10	21.1	55
12.5	32.2	70
15	37.8	80
17.5	26.7	85
20	26.7	90

2.6 Statistical Analysis

All data were analyzed by OriginLab Data Analysis and Graphing Software (Guangzhou, China).

3. Results and Discussion

3.1 Compaction Energy

In the research in Chapter 1, the torrefied woody biomass particles required the die temperature as high as 193℃ (380°F) for stable pellet shape. However, the torrefied miscanthus and switchgrass could be compacted at lower die temperature of 121℃ (250°F), which would likely save energy consumption during pellet production. This might due to the degree of torrefaction of the grass materials were less severe than the woody biomass samples. The weight loss of torrefied grass biomass was 20-30% at the relative low torrefied temperature of 230-235 ℃ for 30 minutes compared to the 300 ℃ torrefied temperature of woody biomass in Chapter 1. At this temperature, the hemicellulos were not totally decomposed; thus, the biomass still had enough hydroxyl groups for bonding particles together after torrefaction.

Table 3.3 Average compaction energy, gross heating value, density, and hardness of non-torrefied and torrefied miscanthus and switchgrass pellets with different particle sizes and moisture contents.

Sample Name	Compaction Energy (Joule)[a]	Gross Heat (MJ/kg) [a]	Density (kg/m^3) [a]	Hardness (N/mm) [a]
Miscanthus		17.7±0.2		
M_0.7-1_5%	18.1±2.8	18.7±0.1	1048.5±78.8	22.4±10.9
M_0.7-1_12%	15.2±1.2	18.6±0.1	1099.3±32.3	29.0±6.3
M_0.5-0.7_5%	16.0±0.8	18.5±0.1	1109.8±70.8	30.6±12.7
M_0.5-0.7_12%	14.8±1.5	18.5±0.0	1080.7±39.2	28.8±6.8
Torrefied Miscanthus		19.6±0.4		
TM_0.7-1_1.5%	25.6±3.0	19.8±0.1	1067.1±31.7	25.8±11.3
TM_0.7-1_5%	23.4±3.1	19.9±0.0	1093.2±23.0	24.8±9.9
TM_0.5-0.7_1.5%	23.8±2.3	19.7±0.0	1093.5±41.8	25.8±10.6
TM_0.5-0.7_5%	21.5±2.0	19.8±0.1	1100.7±22.9	24.7±9.2
Switchgrass		17.4±0.2		
SG_0.7-1_5%	12.6±0.6	18.1±0.1	1109.8±29.6	22.6±6.5
SG_0.7-1_12%	12.6±1.0	18.0±0.1	1064.6±33.1	24.1±7.1
SG_0.5-0.7_5%	12.4±1.2	18.1±0.2	1080.2±44.4	18.0±5.8
SG_0.5-0.7_12%	13.3±1.9	17.9±0.2	1088.8±53.5	29.5±8.8
Torrefied Switchgrass		20.2±0.5		
TSG_0.7-1_1.5%	19.5±1.4	20.2±0.0	1055.8±38.9	20.9±4.5
TSG_0.7-1_5%	16.9±2.9	20.3±0.2	1085.8±39.7	21.3±5.8
TSG_0.5-0.7_1.5%	20.2±1.5	20.2±0.2	1067.9±50.9	20.1±5.0
TSG_0.5-0.7_5%	19.5±2.4	20.1±0.0	1080.2±16.2	18.1±2.4

a. Mean±standard deviation.

The compaction energy of pelletizing non-torrefied and torrefied miscanthus materials with different moisture content and particle size is shown in Figure 3.5 and Table 3.3. From these results it was apparent that the torrefied miscanthus materials required nearly 1.5 times more compaction energy than the non-torrefied materials. This increase in compaction energy was likely due to the plasticity of biomass being decreased through torrefation (Li et al., 2012). According to the one-way ANOVA analysis, there was a statistically significant difference in average compaction energy within the non-torrefied miscanthus pellet groups (p-value < 0.0001, at $\alpha = 0.05$ significance level). Results of the Tukey-Kramer-Comparison test indicated that only the pellet with the particle size of 0.7-1 mm and moisture content of 5% had statistically significantly higher required average compaction energy than other types of pellets (p-value < 0.0001, at $\alpha=0.05$ significance level). This finding is likely due to the lower moisture content of the particles generating more friction between die and particles or particles and particles during pelletizing. There was a statistically significant difference within the torrefied miscanthus pellet groups as well (p-value < 0.0001, at α=0.05 significance level). Based on the Tukey-Kramer-Comparison test, the compaction energy of the materials with lower moisture content of 1.5% showed a statistically significant higher average compaction energy than the higher moisture content of 5% at both particle size levels of 0.7-1 mm and 0.5-0.7 mm (p-value < 0.0001, at α=0.05 significance level). This finding may be related to the higher friction produced by the lower moisture content particles in densification. However, the particle size was not significant to torrefied miscanthus pellets in terms of compaction energy.

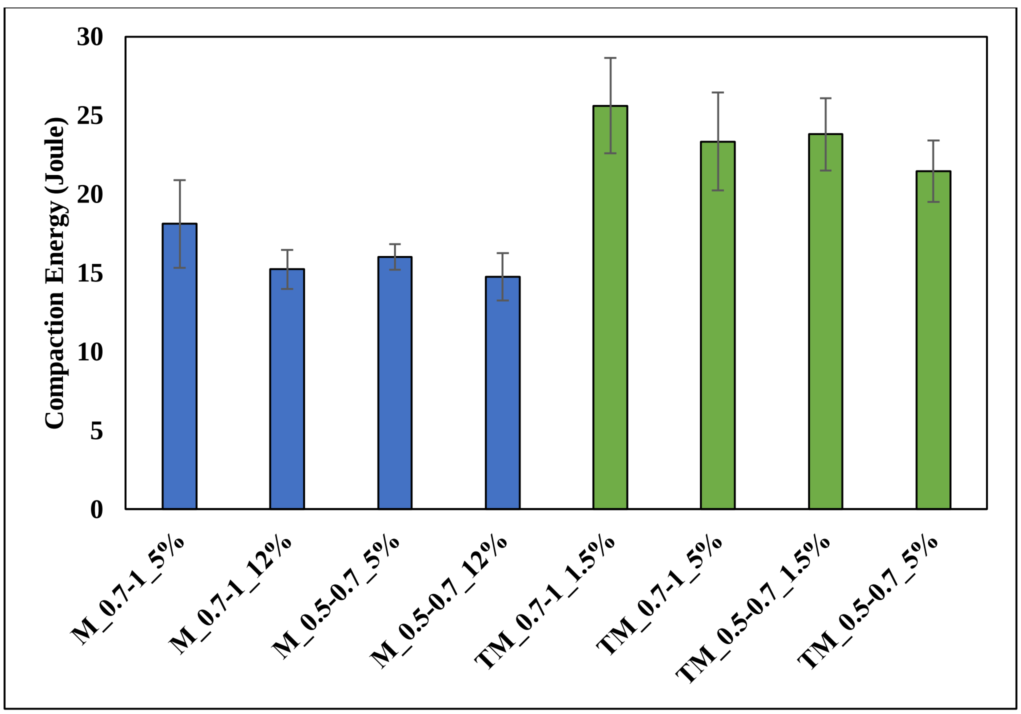

Figure 3.5. Compaction energy of pelleting non-torrefied and torrefied miscanthus pellets with different particle size and moisture content.

Figure 3.6 shows the compaction energy of pelletizing non-torrefied and torrefied switchgrass materials. Similarly to the miscanthus samples, the torrefied biomass materials required more than 1.5 times of compaction energy than the non-torrefied samples. However, unlike the miscanthus materials, there was no statistically significant difference in average compaction energy within non-torrefied switchgrass pellet groups (p-value = 0.1298) in one-way ANOVA analysis. Thus the moisture content and particle size did not have significant influence on the non-torrefied switchgrass pellet compaction energy. Within the torrefied switchgrass pellet groups, there was a statistically significant difference (p-value < 0.0001, at α = 0.05 significance level). According to the Tukey-Kramer-Comparison test results, only the switchgrass pellets with the larger particle size of 0.7-1 mm and higher moisture content of 5% showed a statistically significantly lower average compaction energy during pelleting (p-value < 0.0001, at α=0.05 significance level). In comparing miscanthus and switchgrass, as can be seen from Table 3.3, the required average compaction energy of miscanthus materials was higher than the switchgrass for both torrefied and non-torrefied types. Specific reasons for the differences require further investigation and are a potential area for future research.

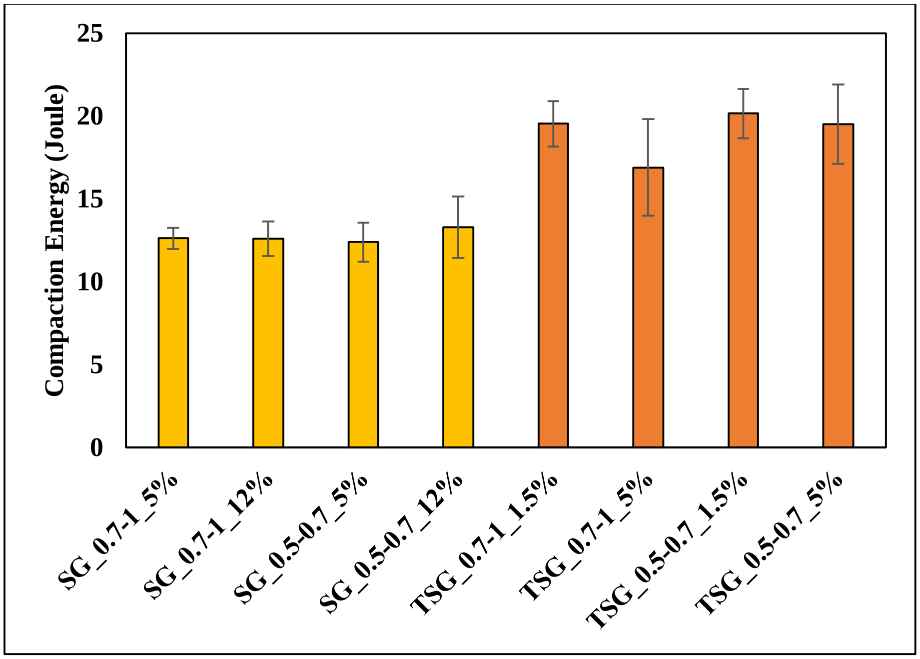

Figure 3.6 Compaction energy of non-torrefied and torrefied switchgrass pellets with different particle sizes and moisture contents.

3.2 Gross Heating Value

The gross heating value of miscanthus is shown in Figure 3.7. It is apparent that the torrefied samples gross heating values were higher than the non-torrefied samples for both the raw material and pellets. Again, this is expected as the torrefaction process results in improvement of the fixed carbon content in biomass. Figure 3.8 presents the gross heating value of non-torrefied and torrefied switchgrass raw materials and pellets. As similar to miscanthus, the gross heating value of torrefied switchgrass particles and pellets are higher than the non-torrefied samples.

In both non-torrefied miscanthus and switchgrass materials, the gross heating value was significantly increased by densification due to the increase in density (p-value < 0.0001, at α=0.05

significance level). According to one-way ANOVA analysis, there was a statistically significant difference in average gross heating value within non-torrefied miscanthus pellet groups (p-value < 0.0001, at α = 0.05 significance level). In further investigation the only statistically significant difference was found between miscanthus pellets made at 5% moisture content and 12% moisture at the particle size level of 0.7-1 mm (p-value < 0.0001, at α = 0.05 significance level). However, there was no statistically significant difference within non-torrefied switchgrass pellet groups (p-value = 0.4132).

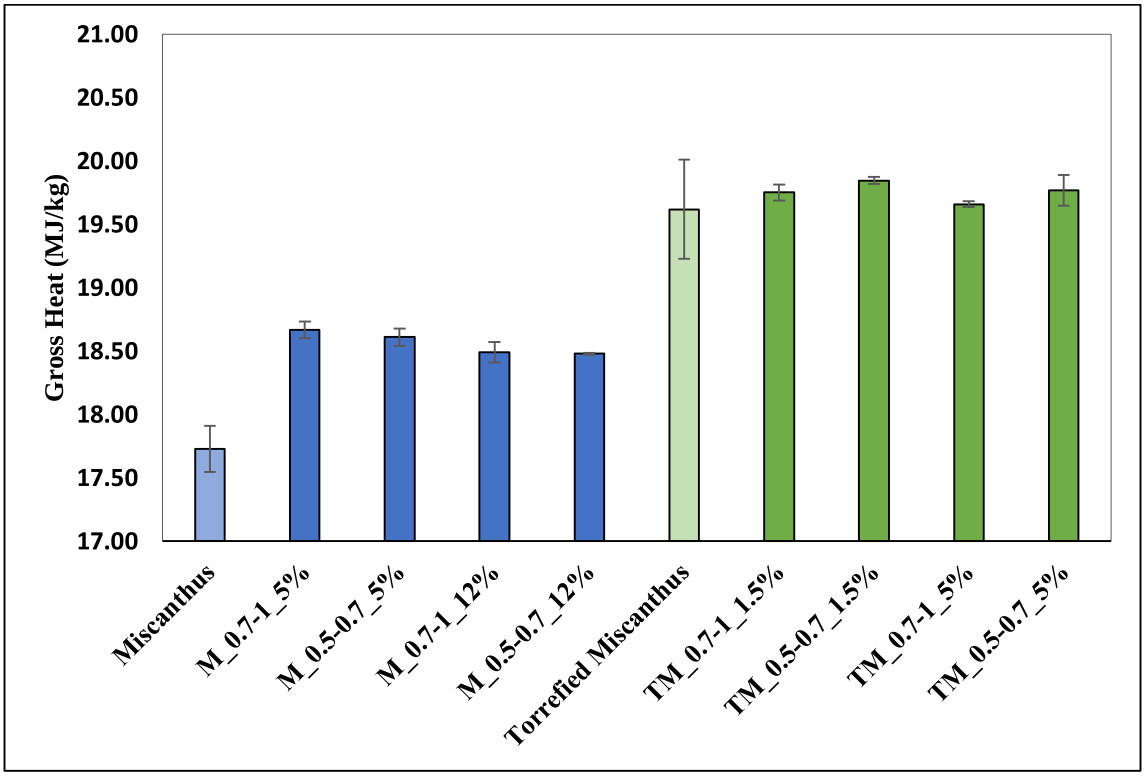

Figure 3.7 Gross heating value non-torrefied and torrefied miscanthus raw materials and pellets.

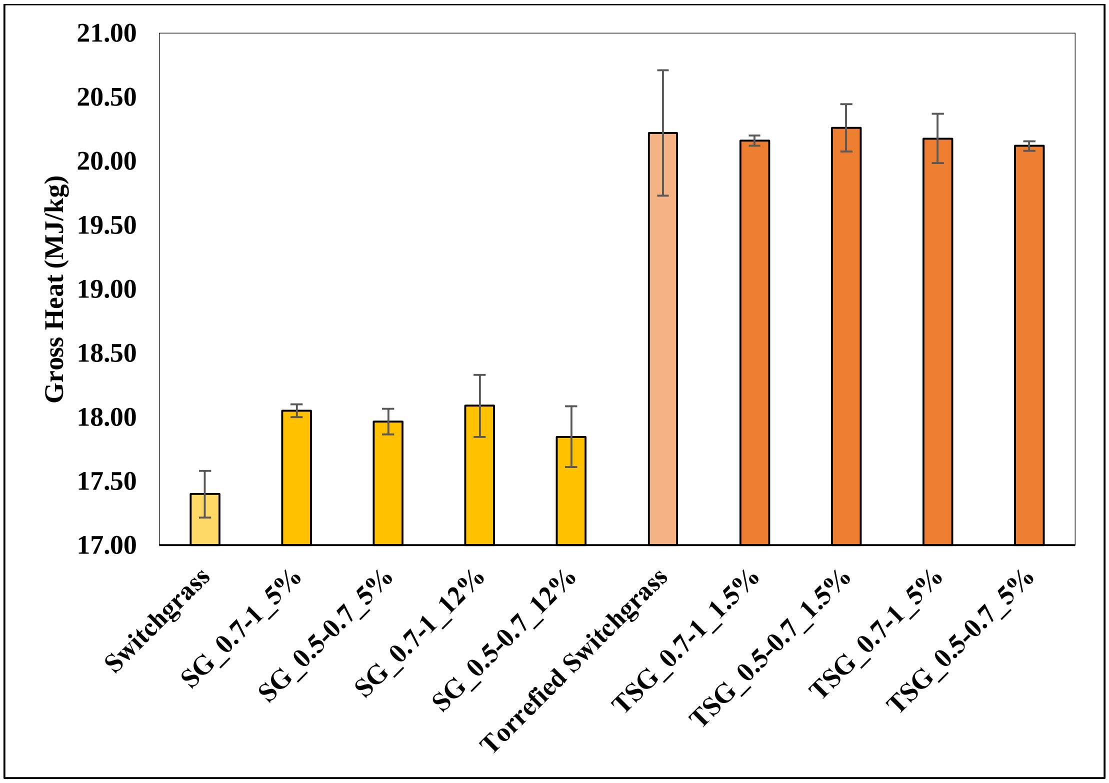

Figure 3.8 Gross heating value non-torrefied and torrefied switchgrass raw materials and pellets.

Nevertheless, the gross heating value did not show significant difference within the torrefied miscanthus groups (p-value=0.60479), as well as within the torrefied switchgrass groups (p-value=0.9628). Thus, pelletizing did not show any statistically significant influence on the gross heating value of torrefied miscanthus or switchgrass samples. Furthermore, there was no statistically significant difference of gross heating value within either miscanthus or switchgrass pellet groups. The particle size and moisture content were not significant parameters in torrefied miscanthus and switchgrass pellet production.

3.3 Hardness

Figure 3.9 shows the hardness (N/mm) of non-torrefied and torrefied miscanthus pellets. The hardness of torrefied miscanthus pellets was much improved, as compared to the torrefied red oak woody biomass pellet hardness in chapter 1. Specifically, with the grass-type biomass, there was not a large difference between pellet hardness when comparing non-torrefied and torredfied pellets. Furthermore, the density of non-torrefied and torrefied miscanthus pellets (Figure B8 in Appendix B) were also fairly similiar. The reason for these relative closeness between the non-torrefied and torrefied grasses might be due to the miscanthus material being torrefied at relatively mild condition with less weight loss compared to the woody biomass in Chapter 1. However, based on one-way ANOVA analysis, there was a statistically significant difference within non-torrefied miscanthus pellet groups (p-value=0.04093, at α=0.05 significance level). Specifically, only at the moisture content level of 5%, the average hardness of the pellets with smaller particle size of 0.5-0.7 mm was statistically significantly higher than the larger particle size of 0.7-1 mm (p-value < 0.0001, at α = 0.05 significance level). Furthermore, there was no statistically significant difference of hardness within torrefied miscanthus pellet groups (p-value = 0.9766).

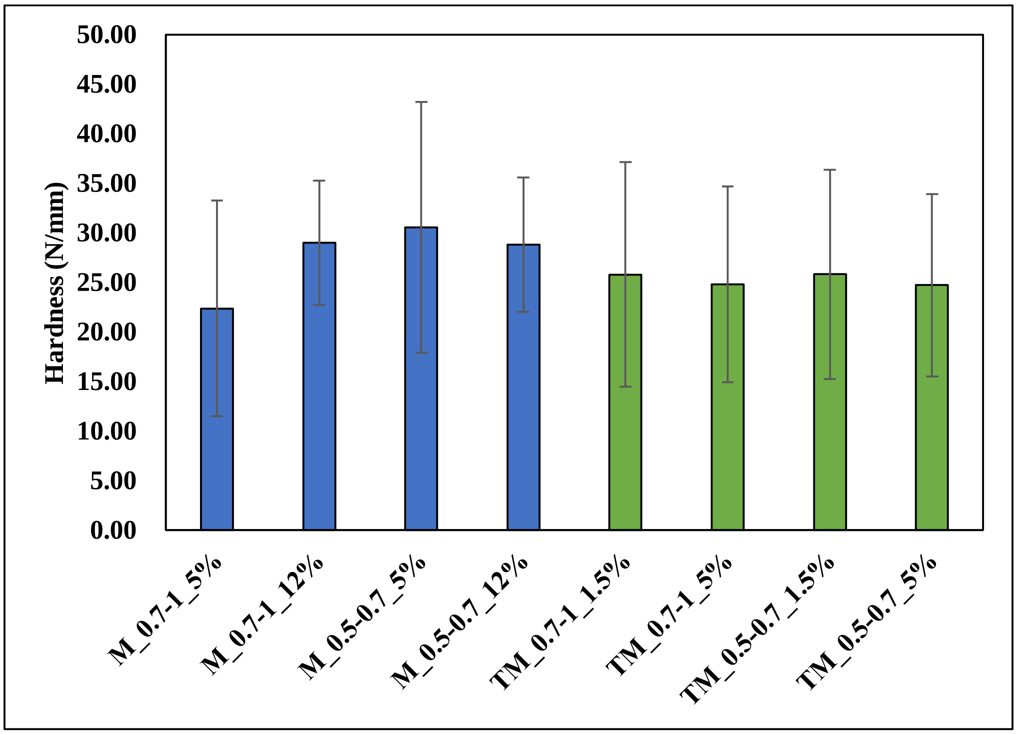

Figure 3.9 Hardness of non-torrefied and torrefied miscanthus pellets.

Figure 3.10 shows the hardness of non-torrefied and torrefied switchgrass pellets. From the results, it was apparent that the hardness of non-torrefied and torrefied switchgrass pellets was similar to the results found in the miscanthus pellets. Specifically, the hardness of non-torrefied and torrefied pellets was within the same range. The similar hardness results are likely in part due to the ability to produce densified material from the torrefied grass materials as both the non-torrefied and torrefied switchgrass pellets had a relatively similar density (Figure B9 in Appendix B). Within the non-torrefied switchgrass pellet group, there was a statistically significant difference in terms of average hardness (p-value < 0.0001, at α = 0.05 significance level). Upon further Tukey testing, it was found that the only statistically significant difference in average hardness in the non-torrefied switchgrass were those made at 5% and 12% when the particle size was 0.5-0.7 mm (p-value < 0.0001, at α = 0.05 significance level). In that instance, the non-torrefied switchgrass pellets made using raw materials at 12% moisture content produced harder pellets.

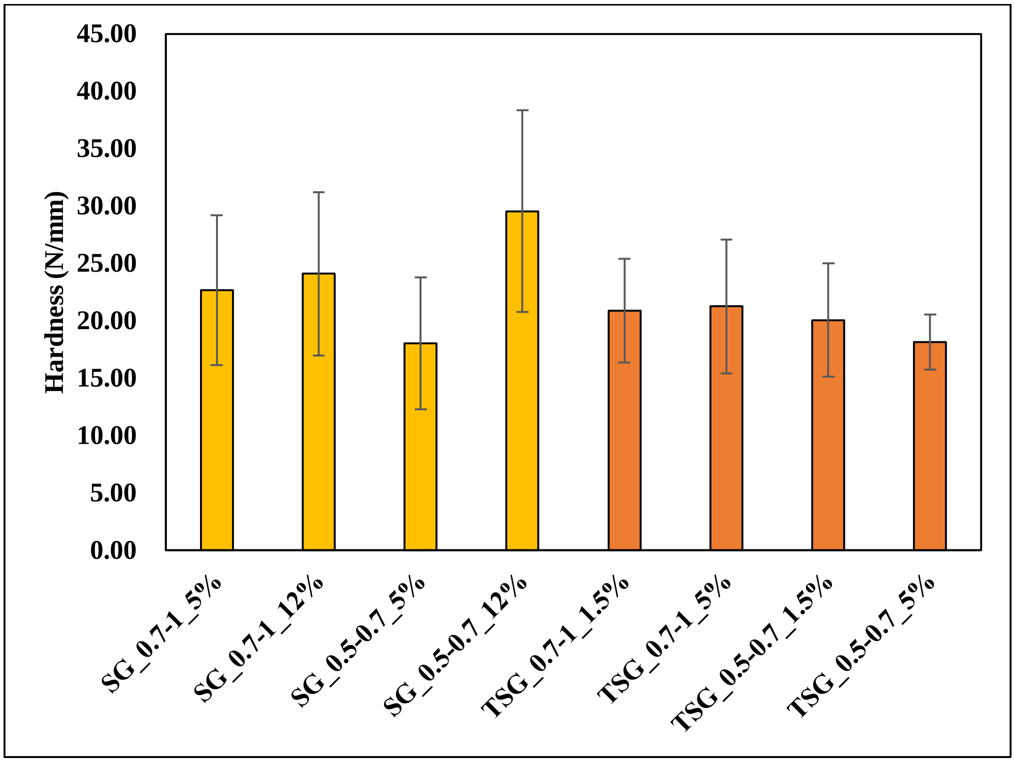

Figure 3.10 Hardness of non-torrefied and torrefied switchgrass pellets.

In terms of torrefied switchgrass pellets, results of the one-way ANOVA testing indicated that there was not any statistically significant difference within the torrefied switchgrass pellet groups (p-value=0.14869). These results suggest that the different moisture content and particle sizes tested had little impact on influencing the hardness in both torrefied miscanthus and torrefied switchgrass pellet production. More specifically, the results indicated that when producing torrefied pellet material, less attention may need to be paid to moisture content and particle size in terms of hardness, and that it may be more beneficial to select a moisture and particle size that reduces the compaction energy. For example in the torrefied switchgrass, the more ideal conditions would be the larger particle size of 0.7-1 mm and higher moisture content of 5% that had a significantly lower compaction energy requirement (p-value < 0.0001, at α = 0.05 significance level).

3.4 Moisture resistance: Visual Appearance

Type	Before Environmental Conditions Testing	At 15% MC Target	At 17.5% MC Target	At 20% MC Target
Non-Torrefied Miscanthus				
Torrefied Miscanthus				
Non-Torrefied Switchgrass				
Torrefied Switchgrass				

Figure 3.11 Environmental conditions testing visual results on miscanthus and switchgrass pellets.

Figure 3.11, respectively shows the appearance of non-torrefied and torrefied miscanthus and switchgrass pellets before test, as well as under the 20% target moisture content conditioning. With the exception of the torrefied miscanthus pellets, all the pellets expanded considerably in length and broke into particles after the test. The torrefied miscanthus pellets stayed, for the most part, rather intact under the high moisture conditions. The non-torrefied switchgrass pellets performed the worst in terms of moisture resistance and were degraded to particles at the 15% target moisture content. Compared with the non-torrefied switchgrass pellets, the torrefied switchgrass pellets performed slightly better in moisture resistance as they began to break into particles at the target moisture content of 17.5%. Similarly, the torrefied miscanthus did not degrade into particles nearly as much as the non-torrefied miscanthus pellets. From this qualitative analysis, the torrefied pellets presented better moisture resistance from an appearance standpoint.

3.5 Moisture Resistance: Environmental Conditioning

Figure 3.12 show the moisture content of non-torrefied and torrefied miscanthus particles in the environmental chamber conditioning test. Table A3 in Appendix A provides the details on specific values for each type at each condition. In general, the results of the environmental tests indicated that there is a difference between the moisture contents of non-torrefied and torrefied miscanthus materials. The torrefied materials showed better moisture resistance than the non-torrefied samples. Non-torrefied biomass particles absorbed more moisture in the atmosphere than the torrefied material, especially under higher target moisture content conditions. Thus, the difference of moisture content between non-torrefied and torrefied biomass particles became larger as the target moisture content increased. Specifically, torrefied biomass particles moisture content was 13% at 20% target moisture content, which is far less than the non-torrefied biomass particles that rose to a 21% moisture content. The reduction of moisture content of the torrefied material is most likely to be explained by the decomposition of hemicellulose resulting the decreasing of hydroxyl groups in biomass which had the function of bonding water. Thus the torrefied biomass showed a lower tendency to absorb water than non-torrefied biomass.

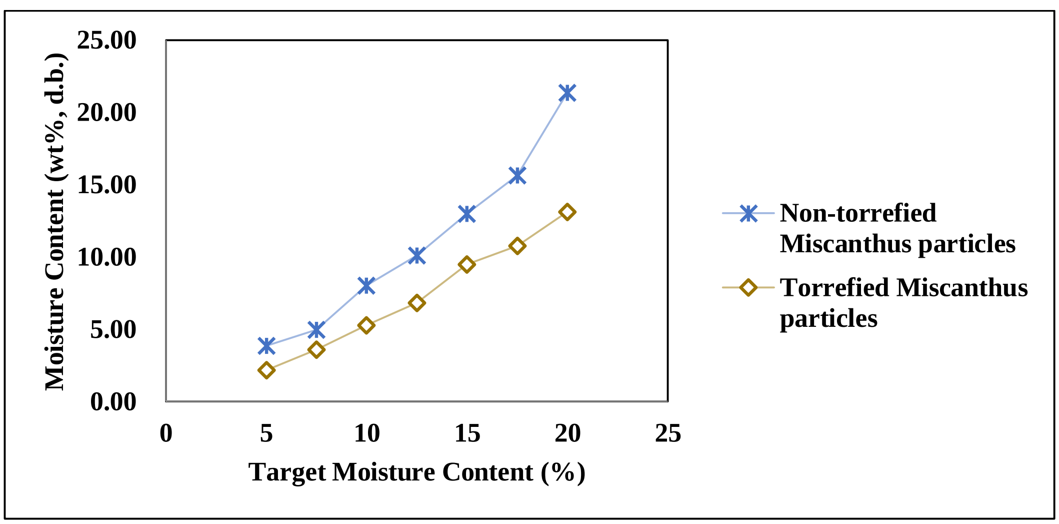

Figure 3.12. Moisture resistance of non-torrefied and torrefied miscanthus material particles.

Figure 3.13 shows the moisture content of non-torrefied and torrefied miscanthus pellets in the environmental chamber conditioning test. The moisture content of torrefied pellets were significantly lower than the non-torrefied pellets especially under higher target moisture content which was similar to the trend presented in Figure 3.12. The moisture content of torrefied pellets of four types were all about 13% under the target moisture content of 20%, which were lower than the moisture content of non-torrefied pellets. The non-torrefied pellets moisture content were 20% except the moisture content of the pellets with 0.5-0.7 mm particle size and 12% moisture content which just obtained 16% moisture content at 20% target moisture content. Moreover, in the torrefied pellet groups, the pellets with larger particle size of 0.7-1 mm absorbed less water than the smaller particle pellets with the same material moisture content level. This might due to the smaller size particles containing larger surface areas and resulting in the hydroxyl groups on the surface had more chances to contact the water molecules in the air. The moisture content of the miscanthus particles at the time of pelleting (1.5% and 5%) did not show any significant effect on the torrefied pellet moisture uptake.

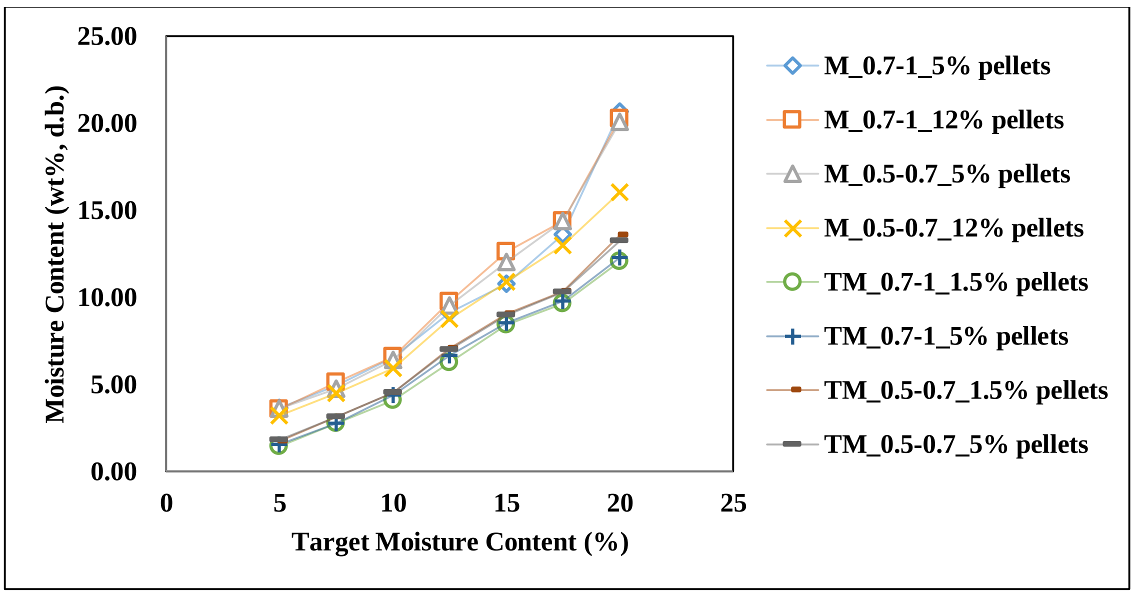

Figure 3.13 Moisture resistance of non-torrefied and torrefied miscanthus pellets.

Figure 3.14 shows the comparison of the moisture content of particles and pellets of non-torrefied and torrefied miscanthus. These results show that both torrefied and non-torrefied miscanthus pellets moisture resistance were improved by densification, as the moisture content of the pellets were lower than the uncompact particles. Specifically, under the target moisture content of 20%, both non-torrefied and torrefied miscanthus pellets absorbed 1% less water than the particles. Thus, pelletizing could improve the moisture resistance properties of the both non-torrefied and torrefied miscanthus.

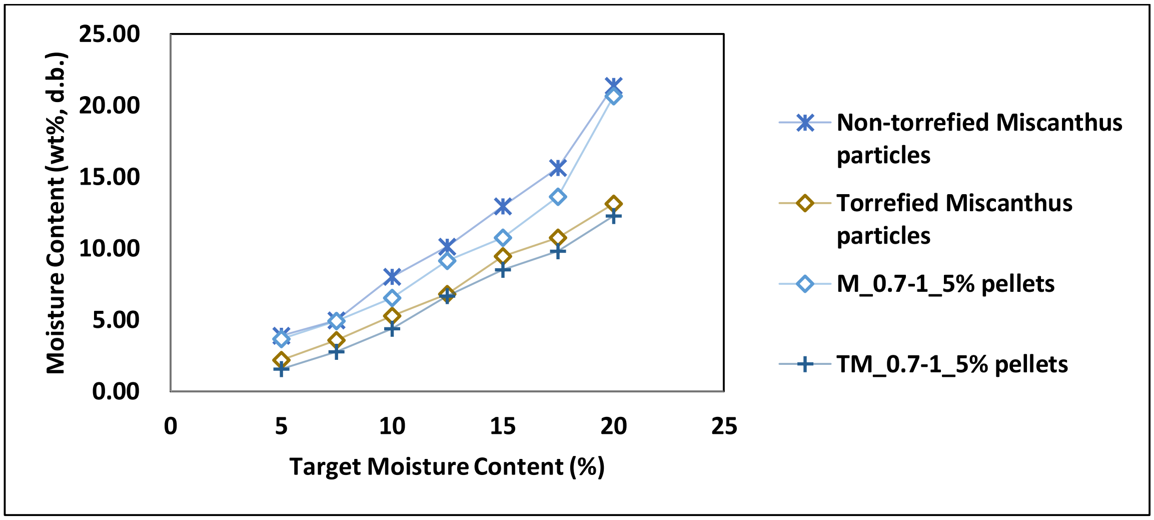

Figure 3.14 Moisture resistance of non-torrefied and torrefied miscanthus material particles and pellets with 0.7-1 mm particle size and 5% moisture content.

Figure 3.15 show the moisture content of non-torrefied and torrefied Swithgrass particles in the environmental chamber conditioning test. Table A4 in Appendix A provides the details on specific values for each type at each condition. Similar to miscanthus, the results of the environmental tests indicated that there is a difference between the moisture contents of non-torrefied and torrefied swithgrass materials. Non-torrefied biomass particles absorbed more moisture in the atmosphere than the torrefied material, especially under higher target moisture content conditions. Thus, the difference of moisture content between non-torrefied and torrefied biomass particles became larger as the target moisture content increased. Specifically, torrefied biomass particles moisture content was 16% at 20% target moisture content, which is far less than the non-torrefied biomass particles that rose to a 26% moisture content. The reduction of moisture content of the torrefied material is most likely to be explained by the decomposition of hemicellulose during torrefaction. Thus the torrefied switchgrass particles appear to absorb less water than non-torrefied switchgrass.

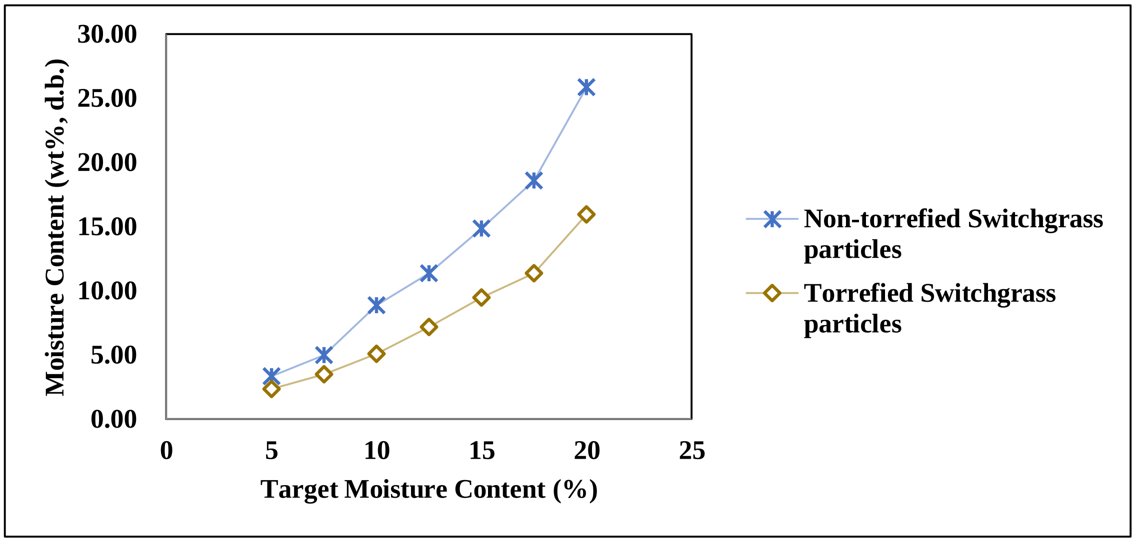

Figure 3.15 Moisture resistance of non-torrefied and torrefied swithgrass materials particles.

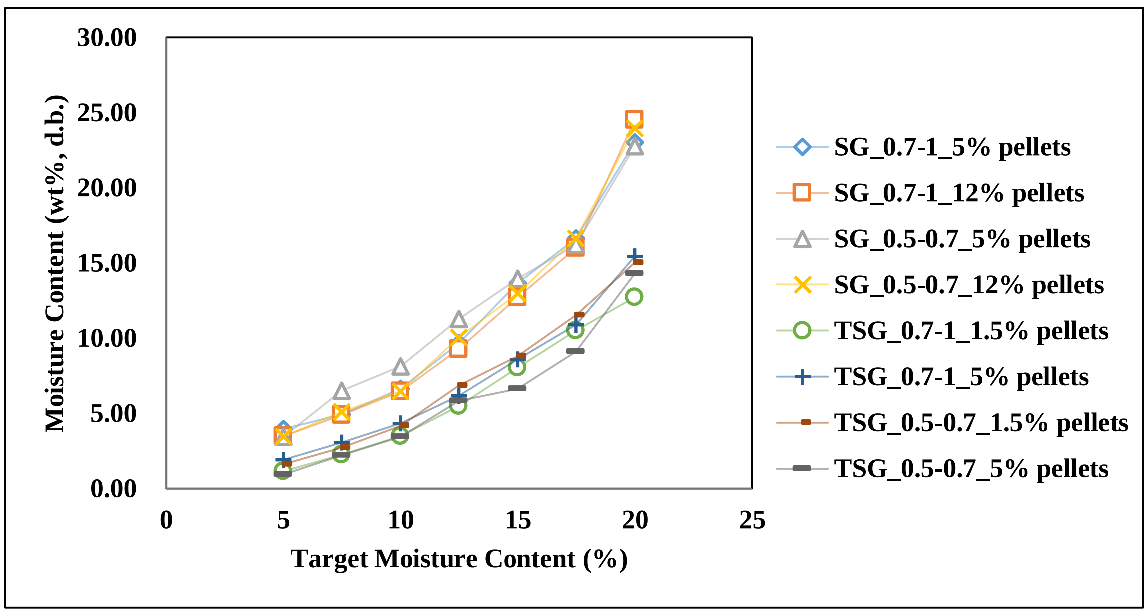

Figure 3.16 Moisture resistance of non-torrefied and torrefied Swithgrass pellets.

Figure 3.16 shows the moisture content of non-torrefied and torrefied swithgrass pellets in the environmental chamber conditioning test. The moisture content of torrefied pellets were significantly lower than the non-torrefied pellets especially under higher target moisture content which was similar to the trend presented in Figure 3.15. The moisture content of torrefied pellets of four types were all about 15% under the target moisture content of 20%, which were much lower than the moisture content of non-torrefied pellets around 23%. The lowest the moisture content in the non-torrefied pellets was the pellet with 0.5-0.7 mm particle size and 5% moisture content. In the torrefied pellet groups, the pellets with 0.7-1 mm particle size and 1.5% moisture content absorbed the least amount of water and obtained 13% moisture content under 20% target moisture content.

Figure 3.17 shows the comparison of the moisture content of particles and pellets of non-torrefied and torrefied switchgrass. These results show that both torrefied and non-torrefied pellets moisture resistance were improved by densification, as the moisture content of the pellets were lower than the uncompact particles. Specifically, at the target moisture content of 20%, the non-torrefied switchgrass pellet absorbed 3% less water than the particles; the torrefied switchgrass pellet also absorbed 1% less water than the particles. Therefore, pelletizing could improve the moisture resistance properties of the both non-torrefied and torrefied switchgrass.

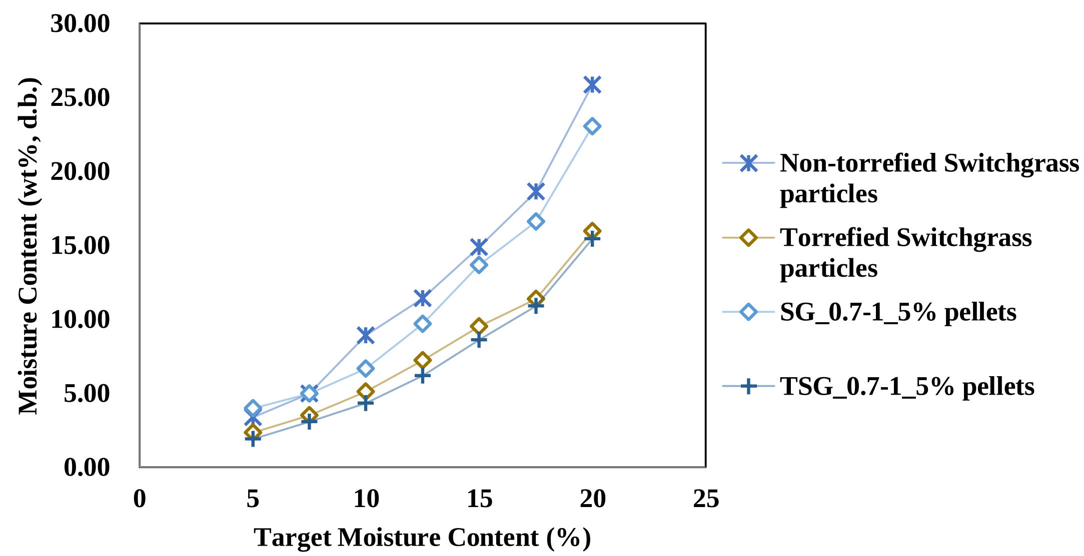

Figure 3.17 Moisture resistance of non-torrefied and torrefied Swithgrass materials particles and pellets with 0.7-1 mm particle size and 5% moisture content.

4. Conclusions

Torrefaction as pretreatment had significantly influence on the grass-type biomass properties including the improvement of calorific value and better resistance to moisture uptake. Furthermore, the mechanical strength of grass-type biomass pellets was not changed significantly after mild torrefaction. However, torrefied biomass also showed the disadvantages of higher compaction requirement during pelletizing process. Furthermore, since the moisture content and particle size also had some relative smaller influence on the pellet quality, the better condition might be considered for the pellet production based on biomass species and pretreatment, in order to produce the pellet with desirable final mechanical and physical properties.

For the non-torrefied miscanthus pellets, the moisture content of 5% and the particle size of 0.5-0.7 were found to be the most efficient processing variables as the pellets made under this condition performed a relatively high hardness. The better the conditions for the torrefied miscanthus was the moisture content of 5% and the particle size of 0.5-0.7 mm based on the lowest compaction energy in densification and equivalent hardness to the other types of pellets. In regards to the non-torrefied switchgrass, the processing condition of 0.5-0.7 mm particle size and 12% moisture content was the best in terms of pellet hardness. For torrefied switchgrass, the smaller particle size of 0.7-1 mm and higher moisture content of 5% was the better condition for due to the lowest energy consumption in pelletizing and equivalent hardness to the other types of pellets.

CHAPTER 4: Overall Conclusions

Many woody and grass-type biomass properties were improved by torrefaction and pelletizing based on the results in the Chapter 1 and 2. The calorific value of biomass was increased after torrefaction due to the removal of a proportion of hemicellulose and other components. The grass-type biomass could not be torrefied at as high as a temperature when compared to the woody biomass. Because of the lower torrefaction level, the gross heating value increase through torrefaction of grass-type biomass was relatively lower than that of the woody biomass. The gross heating values of both non-torrefied woody were slightly improved by densification. The increase was likely due to the high die temperature during compaction that heated the particles. Because of the milder torrefied conditions of grass-type biomass, it was likely that a higher percentage of hemicellulose and other extractives that act as binders in densification were left in the torrefied biomass, as compared to the torrefied woody biomass. Therefore, the torrefied grass-type biomass consumed relatively less energy in pelletizing as compared to the torrefied woody biomass. Additionally, the hardness of the torrefied grass-type pellets was higher than the torrefied woody biomass. Furthermore, the moisture resistance of torrefied biomass was better than the non-torrefied biomass in terms of both raw materials and pellets. The torrefied miscanthus pellets showed the best moisture resistance in all of the grass-type biomass pellets. Moreover, both non-torrefied and torrefied biomass (i.e., particles and chips) moisture resistance was improved by densification as evident from the results of the environmental conditioning tests.

When comparing the influence of torrefaction on the biomass pellet quality, the effects of moisture content and raw material particle size before pelletizing were relatively small. The results of the research on the influence of moisture content and particle size on the pellet quality allowed for determining the conditions for each material that would most likely influence the pellet production on laboratory scale. However, further research using a laboratory scale pellet machine is needed to confirm these results. The results indicated that the better moisture content and particles size for the non-torrefied red oak were smaller particles (size of 0.5-0.7 mm) and lower moisture content (1.5%). However, when scaling up to the larger mill, there could be issues with this lower moisture content in the event the pellet temperature is not able to reach a high enough level to cause the lignin to flow. On the contrary, the better conditions for the torrefied pellets were the larger particle size (0.7-1 mm) and higher moisture content (5%). For the non-torrefied miscanthus pellets, the moisture content of 5% and the particle size of 0.5-0.7 mm were found to be the most efficient processing variables as the pellets made under this condition has the highest hardness value. The better condition for producing the torrefied miscanthus was a moisture content of 5% and a particle size of 0.5-0.7 mm. This finding was based on these pellet requiring a lower compaction energy with equivalent hardness to the other types of torrefied miscanthus pellets. In regards to the non-torrefied switchgrass, a 05.-0.7 mm particle size and a 12% moisture content was found to be the highest in terms of pellet hardness. For torrefied switchgrass, the smaller particle size (0.7-1 mm) and the higher moisture content (5%) was found to be the better processing condition due to the lowest energy consumption in pelletizing and equivalent pellet hardness, as compared the other types of torrefied switchgrass pellets. Overall, the results of the research identified the influence of some key raw material variables (i.e., moisture content level, torrefaction pretreatment, particle size) on some important final pellet properties. These results will provide necessary fundamental information needed to scale-up to a larger lab type pellet machine for further investigation.

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A P P E N D I X   A

Table A1 Moisture content of non-torrefied and torrefied red oak chips and pellets in water immersion test.

Time (min)	Moisture Content (wt%, d.b.)									
	Chips		Pellets							
	Non-torrefied	Torrefied	RO_0.7-1_5%	RO_0.7-1_1.5%	RO_0.5-0.7_5%	RO_0.5-0.7_1.5%	TRO_0.7-1_5%	TRO_0.7-1_1.5%	TRO_0.5-0.7_5%	TRO_0.5-0.7_1.5%
0	0	0	0	0	0	0	0	0	0	0
10	35.80	6.05	108.98	105.13	124.54	137.59	90.48	96.39	88.17	111.34
20	45.02	8.22	116.64	114.94	129.26	145.34	96.02	101.33	100.03	114.41
30	48.41	8.65	121.03	122.42	134.07	150.02	100.03	102.61	103.69	116.28
40	48.41	11.26	124.39	127.99	135.92	152.85	101.77	105.82	106.38	117.96
50	51.81	11.26	128.12	131.18	138.92	156.08	103.34	107.56	107.43	120.33
60	52.29	12.13	131.36	135.20	141.30	157.81	105.23	109.18	109.52	121.93
70	54.72	13.00	133.42	138.57	143.17	159.55	106.69	111.55	110.75	123.91
80	56.17	13.43	136.22	142.64	145.03	161.84	108.38	113.20	114.22	124.89
90	57.14	15.17	139.71	144.63	146.90	164.08	112.29	115.38	116.03	127.22
100	58.60	14.30	143.39	146.92	149.51	166.87	113.55	116.64	118.74	130.18
110	58.60	14.74	148.39	150.92	152.12	169.39	115.24	120.42	121.67	133.32
120	59.57	15.17	151.00	154.72	155.52	172.39	117.33	126.60	125.82	137.83
1440	76.06	33.43	159.25	160.71	162.42	178.27	126.76	148.32	137.58	160.99

Table A2 Moisture content of non-torrefied and torrefied red oak chips and pellets in chamber conditioning test.

Target Moisture Content (%)	Moisture Content (wt%, d.b.)									
	Chips		Pellets							
	Non-torrefied	Torrefied	RO_0.7-1_5%	RO_0.7-1_1.5%	RO_0.5-0.7_5%	RO_0.5-0.7_1.5%	TRO_0.7-1_5%	TRO_0.7-1_1.5%	TRO_0.5-0.7_5%	TRO_0.5-0.7_1.5%
0	0	0	0	0	0	0	0	0	0	0
5	3.12	2.49	1.61	1.63	1.58	1.50	1.61	1.60	1.53	1.60
7.5	7.33	4.78	2.41	2.38	2.33	2.27	2.43	2.44	2.34	2.41
10	10.26	6.00	3.48	3.44	3.41	3.26	3.40	3.39	3.32	3.37
12.5	12.70	6.81	5.45	5.41	5.26	5.12	4.57	4.48	4.39	4.48
15	15.83	7.51	7.39	7.35	7.09	6.97	5.51	5.46	5.30	5.43
17.5	18.53	8.44	8.77	8.74	8.33	8.25	6.40	6.37	6.18	6.23
20	25.46	8.81	11.70	11.68	11.03	10.93	7.11	7.01	6.85	6.91

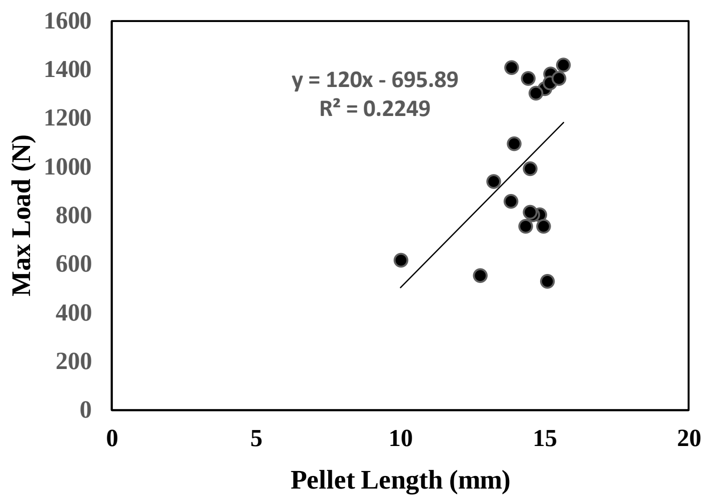
Table A3 Moisture content of non-torrefied and torrefied miscanthus raw materials and pellets in environmental conditioning test.

Target Moisture Content (%)	Moisture Content (wt%, d.b.)									
	Particles		Pellets							
	Non-torrefied Miscanthus particles	Torrefied Miscanthus particles	M_0.7-1_5%	M_0.7-1_12%	M_0.5-0.7_5%	M_0.5-0.7_12%	TM_0.7-1_1.5%	TM_0.7-1_5%	TM_0.5-0.7_1.5%	TM_0.5-0.7_5%
5	3.87	2.19	3.64	3.58	3.62	3.21	1.44	1.57	1.73	1.82
7.5	4.98	3.59	4.93	5.14	4.76	4.48	2.79	2.76	3.15	3.12
10	8.02	5.27	6.55	6.61	6.41	5.92	4.10	4.38	4.53	4.53
12.5	10.11	6.82	9.12	9.76	9.53	8.78	6.26	6.66	7.06	7.01
15	12.96	9.47	10.77	12.63	12.05	10.90	8.43	8.52	9.06	8.99
17.5	15.65	10.77	13.61	14.39	14.38	12.97	9.65	9.79	10.36	10.30
20	21.35	13.11	20.67	20.27	20.06	16.02	12.08	12.27	13.57	13.26

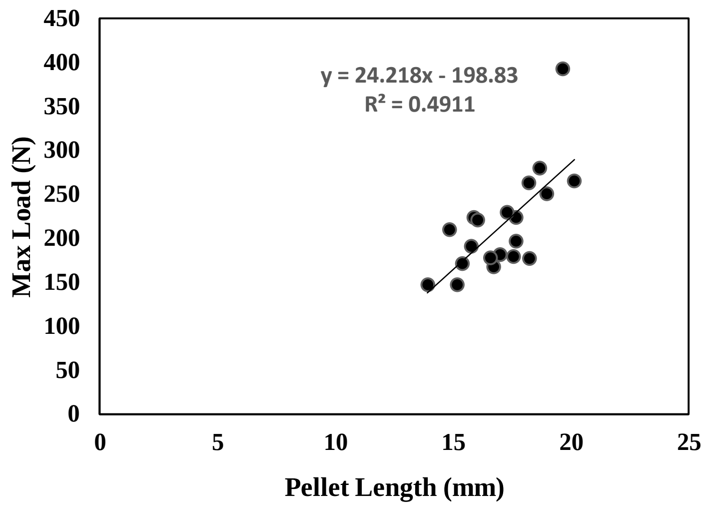
Table A4 Moisture content of non-torrefied and torrefied switchgrass raw materials and pellets in environmental conditioning test.

Target Moisture Content (%)	Moisture Content (wt%, d.b.)									
	Particles		Pellets							
	Non-torrefied Switchgrass particles	Torrefied Switchgrass particles	SG_0.7-1_5%	SG_0.7-1_12%	SG_0.5-0.7_5%	SG_0.5-0.7_12%	TSG_0.7-1_1.5%	TSG_0.7-1_5%	TSG_0.5-0.7_1.5%	TSG_0.5-0.7_5%
5	3.36	2.34	3.97	3.48	3.45	3.44	1.15	1.89	1.60	0.92
7.5	4.98	3.52	4.95	4.90	6.49	5.09	2.26	3.05	2.73	2.20
10	8.91	5.08	6.64	6.48	8.12	6.48	3.47	4.33	4.15	3.45
12.5	11.40	7.20	9.66	9.28	11.27	10.05	5.47	6.17	6.83	5.82
15	14.88	9.49	13.68	12.72	13.94	12.97	8.05	8.61	8.82	6.64
17.5	18.62	11.38	16.62	16.00	16.21	16.63	10.53	10.89	11.52	9.09
20	25.87	15.96	23.04	24.50	22.77	23.96	12.74	15.44	15.03	14.32

APPENDIX B: Statistical Test Results



(a)



(b)

Figure B1. Correlation between the length (mm) and the maximum load (N) of (a) non-torrefied and (b) torrefied red oak pellets with the same moisture content of 1.5% and particle size of 0.7-1 mm.

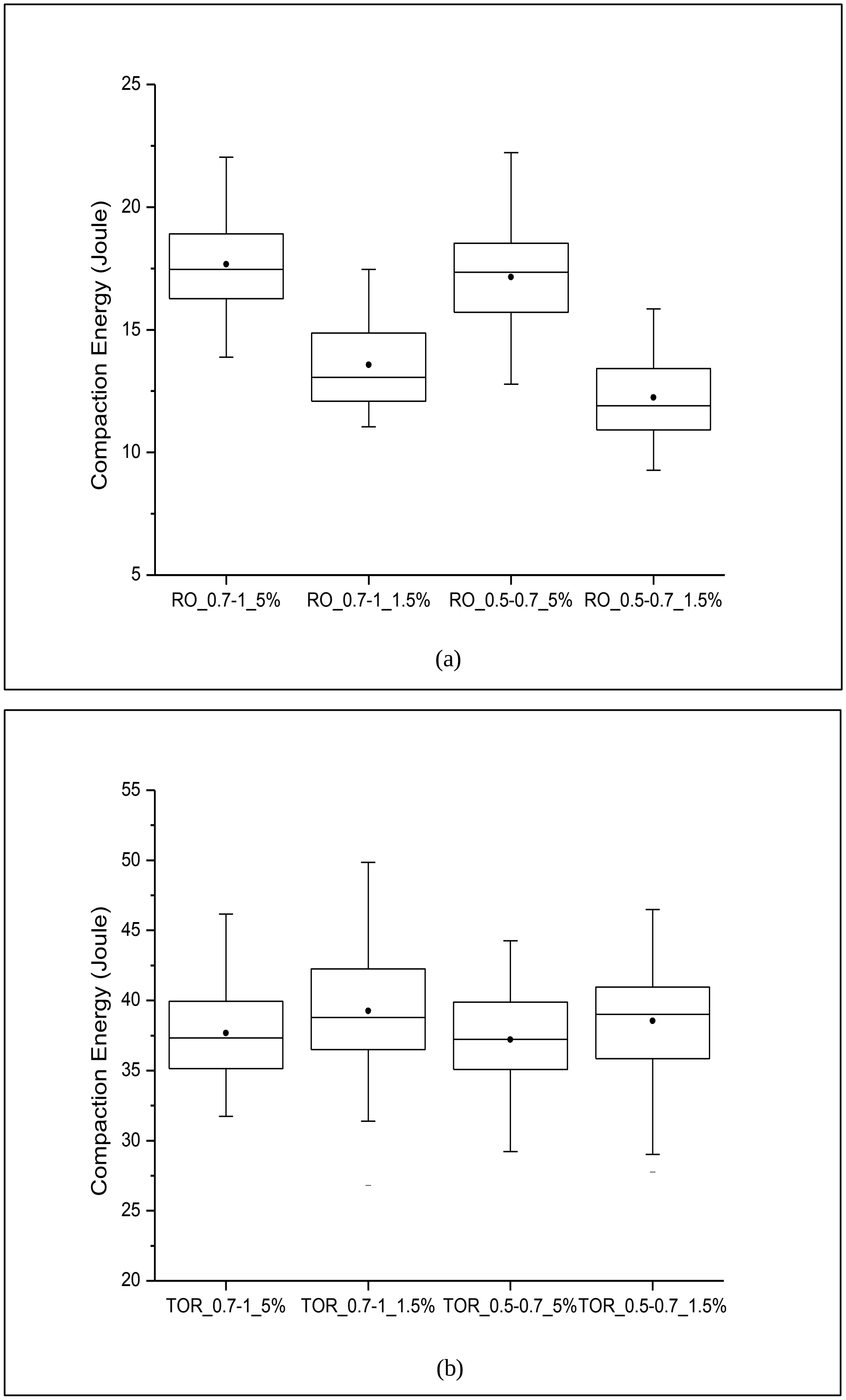


Figure B2. Compaction energy for pelleting of (a) non-torrefied and (b) torrefied red oak pellets.

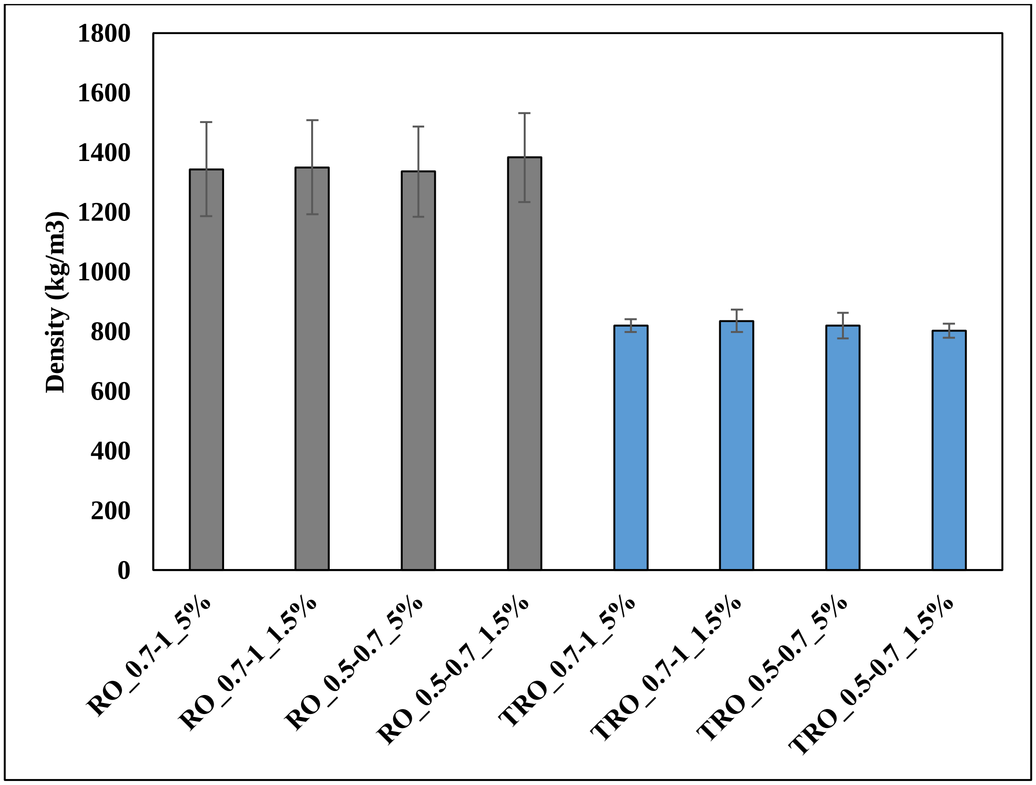
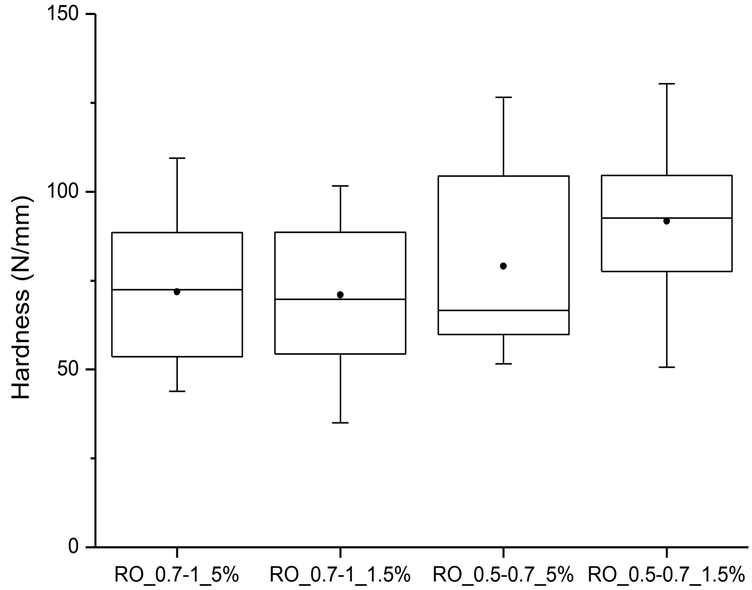
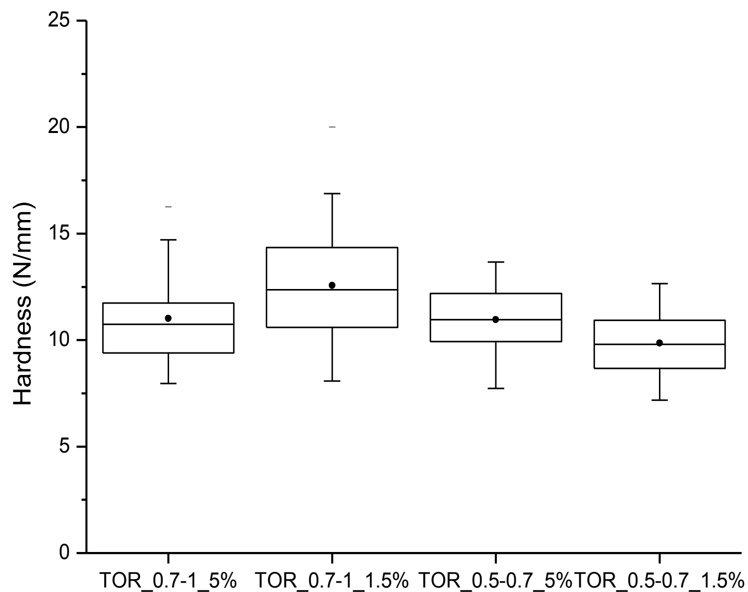


Figure B3. Density of non-torrefied and torrefied red oak pellets.



(a)



(b)

Figure B4. Hardness of (a) non-torrefied and (b) torrefied red oak pellets.

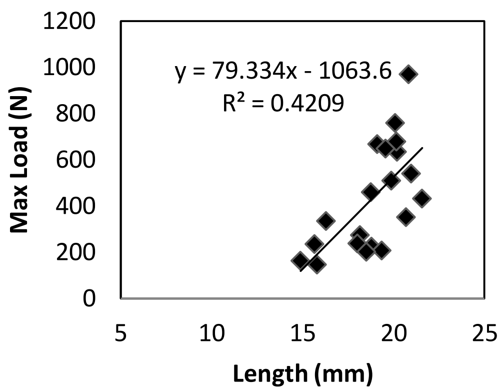
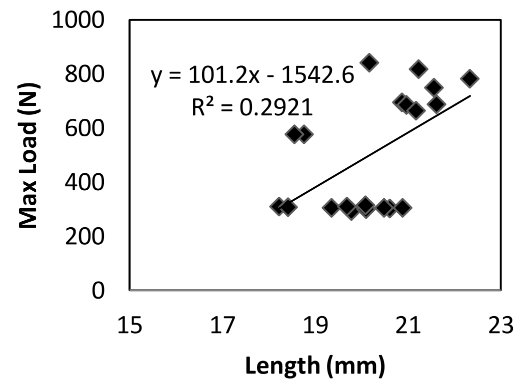
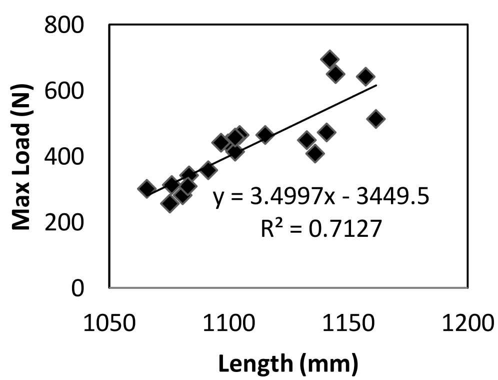
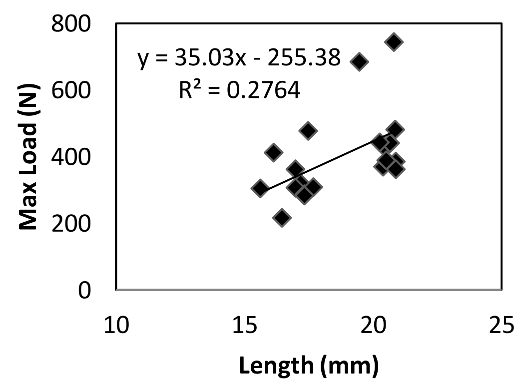
Type	Non-torrefied	Torrefied
Miscanthus		
Switchgrass		

Figure B5. Correlation between the length (mm) and the maximum load (N) of non-torrefied and torrefied miscanthus and switchgrass pellets with the same moisture content of 5% and particle size of 0.7-1 mm.

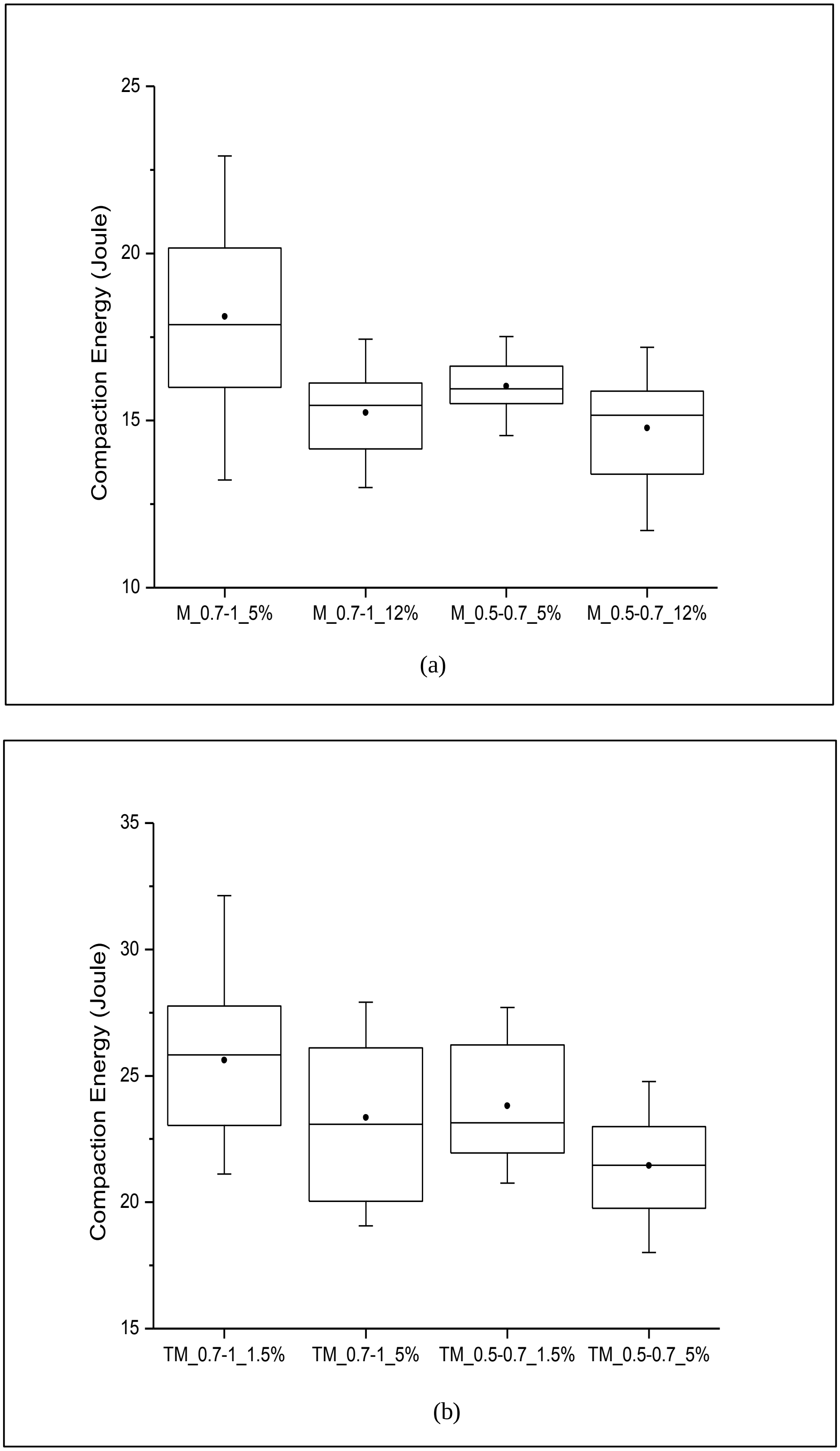
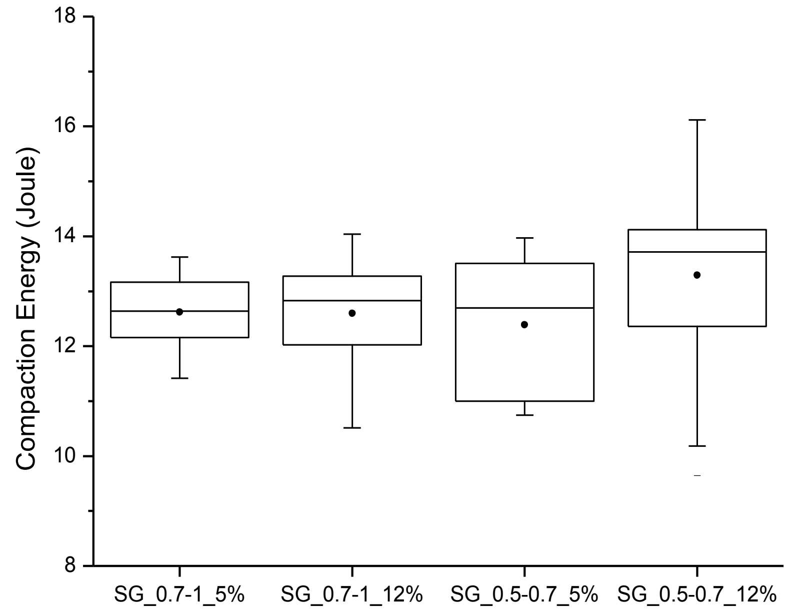
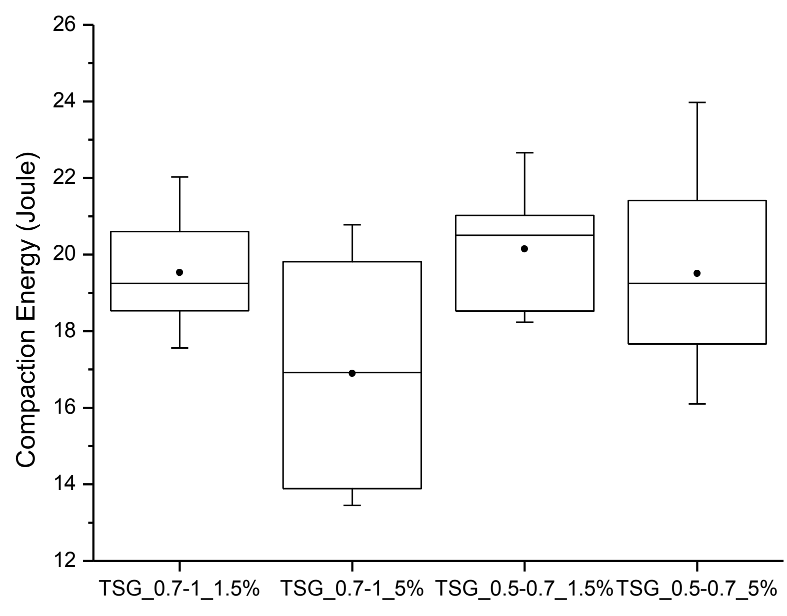


Figure B6. Compaction energy of pelleting (a) non-torrefied and (b) torrefied miscanthus pellets.



(a)



(b)

Figure B7. Compaction energy of pelleting (a) non-torrefied and (b) torrefied switchgrass pellets with different particle size and moisture content.

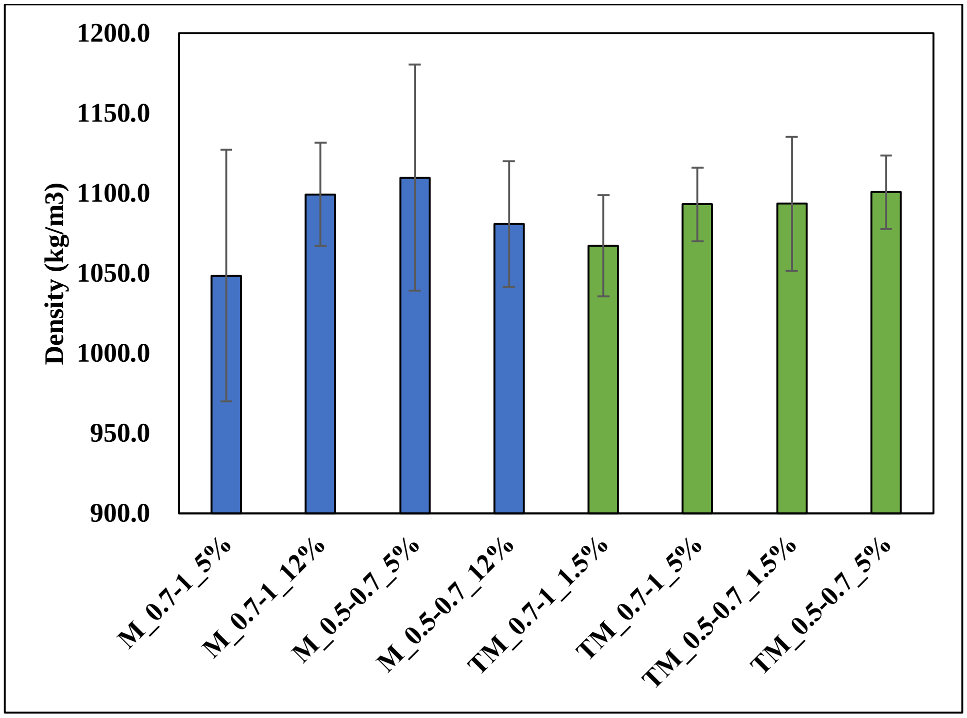


Figure B8. Density of non-torrefied and torrefied miscanthus pellets.

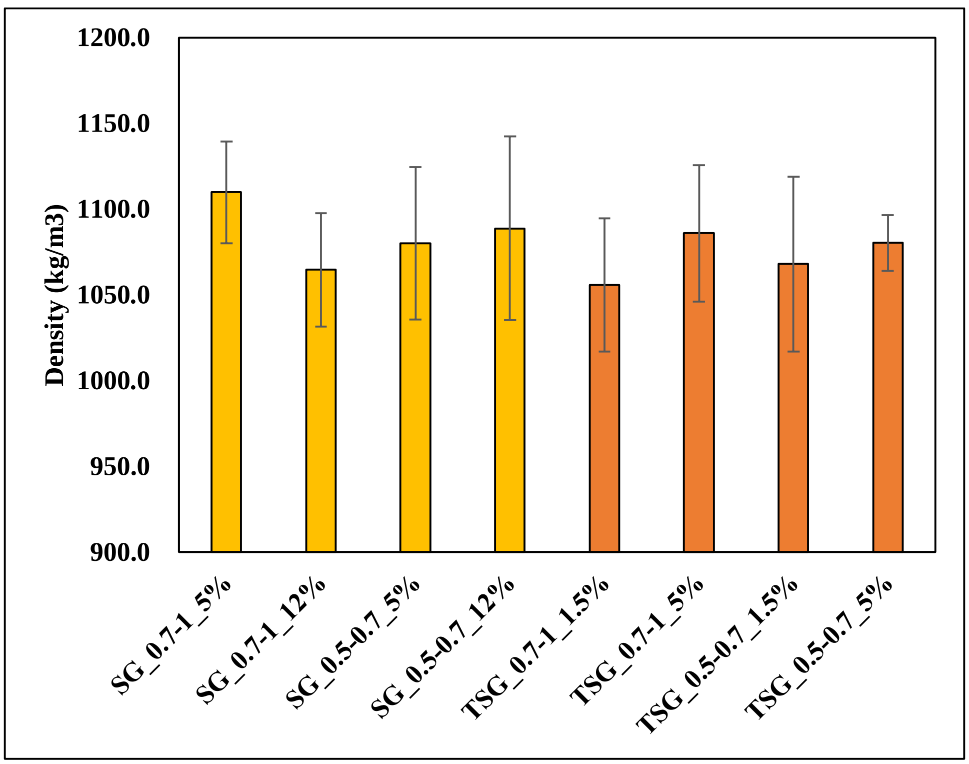
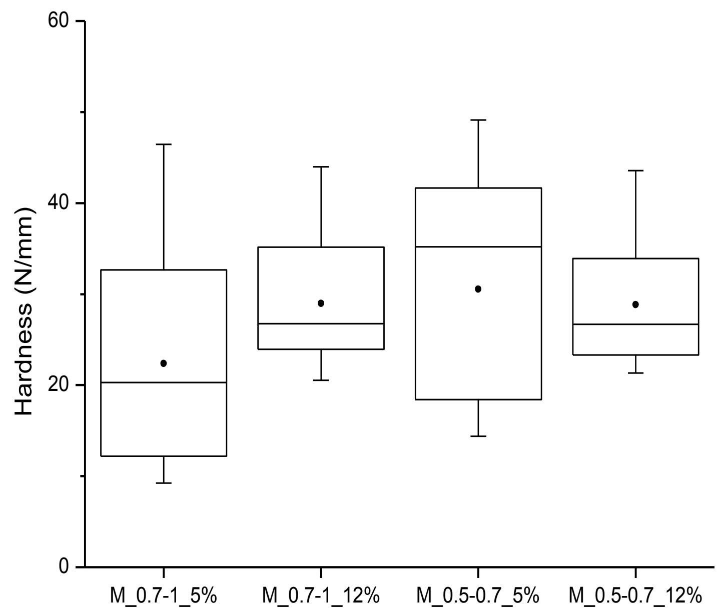
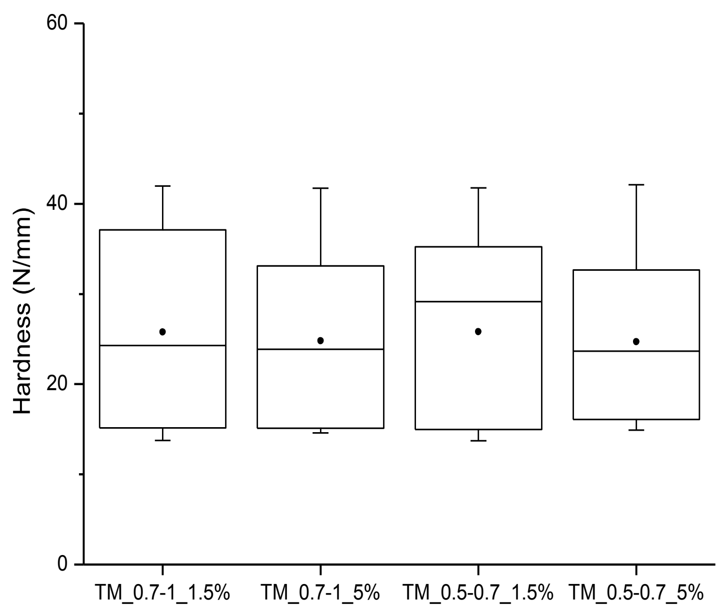


Figure B9. Density of non-torrefied and torrefied switchgrass pellets.

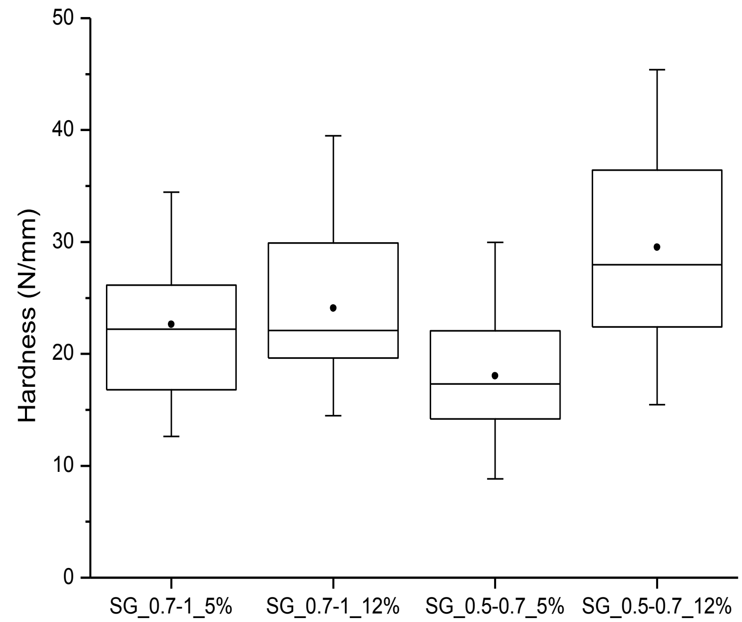


(a)

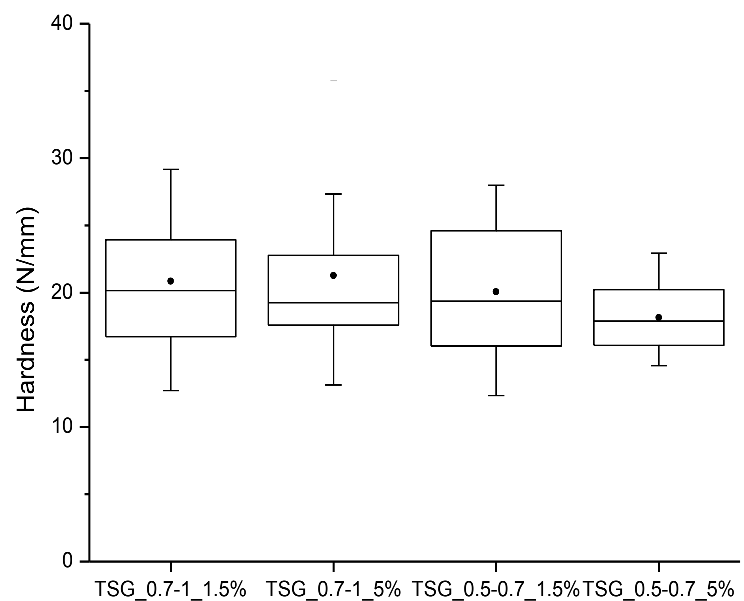


(b)

Figure B10. Hardness of (a) non-torrefied and (b) torrefied miscanthus pellets.



(a)



(b)

Figure B11. Hardness of (a) non-torrefied and (b) torrefied switchgrass pellets.