LIGHTWEIGHT COMPOSITES FROM BIOLOGICAL MATERIALS AND POLYPROPYLENE

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LIGHTWEIGHT COMPOSITES FROM BIOLOGICAL MATERIALS AND
POLYPOLYPROPYLENE

by

Yi Zou

A THESIS

Presented to the Faculty of
The Graduate College at the University of Nebraska
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Biological materials (wheat straws (WS), switchgrass stems (SG) and hop bines (HB)), were used as reinforcing materials to make lightweight composites with polypropylene (PP) webs. The long WS, SG and HB (length up to 10 cm) with simple cut or split and without chemical treatment, were used directly in the composites. Utilizing biological materials for composites not only increases the values of wheat, switchgrass and hop crops, but also provides green, sustainable and biodegradable materials for the composites industry. Lightweight composites are preferred, especially for automotive applications due to the potential saving in energy. In this research, the effects of manufacturing parameters, such as concentrations, lengths, widths and split configurations of reinforcing materials, on the properties of composites have been studied. Compared with the jute-PP composites of the same density (0.47 g/cm³), composites reinforced by biological materials showed generally better properties. The composites reinforced by biological materials with optimized properties have the potential to be used for industrial applications such as the support layers in automotive interiors, office panels and ceiling tiles.
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# Table of Contents

CHAPTER 1: INTRODUCTION ............................................................................... 1

CHAPTER 2: LITERATURE REVIEW ..................................................................... 5

CHAPTER 3: OBJECTIVES ...................................................................................... 8

CHAPTER 4: EXPERIMENTAL ............................................................................... 9
  4.1 Materials ........................................................................................................ 9
  4.2 Composite Fabrication Procedure ................................................................... 10
  4.3 Parameters of Composites Fabrication .......................................................... 13
    4.3.1 Wheat Straw Reinforced Composites ................................................ 13
    4.3.2 Switchgrass Stems Reinforced Composites ....................................... 14
    4.3.3 Hop Bines Reinforced Composites .................................................... 16
  4.4 Material Characterizations .......................................................................... 18
    4.4.1 SEM Analysis ..................................................................................... 18
    4.4.2 Mechanical and Acoustic Testing ....................................................... 18
    4.4.3 Void Content Calculation ................................................................... 19
  4.5 Statistical Method ........................................................................................ 20

CHAPTER 5: RESULTS AND DISCUSSION ........................................................ 21
  5.1 Wheat Straw Reinforced Composites ......................................................... 21
    5.1.1 Effect of WS concentration on the mechanical properties of WS-PP composites ................................................................. 21
    5.1.2 Effect of straw length on the mechanical properties of WS-PP composites ........................................................................... 28
    5.1.3 Effect of splitting configuration on the mechanical properties of WS-PP composites ................................................................. 30
    5.1.4 Effect of WS concentration on the mechanical properties of half split WS-PP composites ................................................................. 32
    5.1.5 Effect of whole and splitting on sound absorption properties of WS-PP composites ........................................................................ 34
    5.1.6 Comparison of WS-PP and jute-PP composites ...................................... 36
  5.2 Switchgrass Stems Reinforced Composites ................................................ 38
    5.2.1 Effect of SG stem concentration on the mechanical properties of SG-PP composites ............................................................. 38
    5.2.2 The effect of stem lengths on the mechanical properties of SG-PP composites ................................................................. 45
    5.2.3 The effect of stem diameters on the mechanical properties of SG-PP composites ................................................................. 47
    5.2.4 The effect of the split configuration on the mechanical properties of SG-PP composites ................................................................. 50
    5.2.5 Comparison of the mechanical properties between SG-PP and jute-PP composites ................................................................. 50
5.2.6 Effect of composites densities on mechanical properties of SG-PP and jute-PP composites .................................................................................................. 53
5.2.7 Effect of the whole and split configurations of SG stems on sound absorption properties of SG-PP composites ......................................................... 55

5.3 Hop Bines Reinforced Composites ......................................................................................... 57
  5.3.1 Effect of regular HB and thin branches on the mechanical properties of composites .......................................................... 57
  5.3.2 Effect of widths of OB on the mechanical properties of composites .......................... 59
  5.3.3 Effect of length of OB on the mechanical properties of composites .......................... 61
  5.3.4 Effect of length of HF on the mechanical properties of composites ................... 63
  5.3.5 Comparison of the mechanical properties among HB-PP, OB-PP and HF-PP composites .............................................................. 66
  5.3.6 Comparison of the mechanical properties between hop-PP and jute-PP composites ......................................................................................... 69
  5.3.7 Comparison of the sound absorption properties between hop-PP and jute-PP composites ......................................................................................... 71

CHAPTER 6: CONCLUSIONS ......................................................................................... 74
  6.1 Wheat Straw Reinforced Composites ............................................................................. 74
  6.2 Switchgrass Stem Reinforced Composites ...................................................................... 75
  6.3 Hop Bines Reinforced Composites ................................................................................. 75

REFERENCE ............................................................................................................ 77
List of Tables

Table 1. Comparison of the effect of WS concentration on mechanical properties of composites from whole and half-split WS................................. 33

Table 2. Comparison of mechanical properties of composites from mechanically split wheat straws and jute, both with PP, at 1500 g/m², and 3.2 mm thickness (0.47 g/cm³).................................................................. 37

Table 3. Comparison of mechanical properties of composites from mechanically split SG stem and jute. .......................................................... 52

Table 4. Comparison of mechanical properties of composites among different densities of composites. ............................................................ 54

Table 5. Comparison of mechanical properties of composites from regular HB (5 cm long), OB (7 cm long and 2 mm wide). HF (7.9 cm long) and jute fibers........................................................................................................... 67

Table 6. Properties of chemically extracted natural cellulose fibers from hop barks compared with cotton and jute. ......................................................... 70
List of Figures

Figure 1. Effect of WS concentrations (weight %) on flexural, impact resistant and tensile properties of WS-PP composites.............................................. 22

Figure 2. SEM images of the cross sections of WS-PP composites. .............. 27

Figure 3. Effect of WS lengths on flexural, impact resistant and tensile properties of WS-PP composites................................................................. 29

Figure 4. Effect of split configurations on flexural, impact resistant and tensile properties of WS-PP composites................................................................. 31

Figure 5. Effect of whole and split configurations on sound absorption properties of WS-PP composites. The sound absorption property of jute-PP composites was compared................................................................. 35

Figure 6. The effects of SG stem concentrations on flexural strength, modulus of elasticity, impact resistance, tensile strength and Young’s modulus of SG-PP composites................................................................. 39

Figure 7. SEM images of the cross sections of SG-PP composites. .............. 44

Figure 8. The effects of lengths of regular SG stem on flexural strength, modulus of elasticity, impact resistance, tensile strength and Young’s modulus of SG-PP composites................................................................. 46

Figure 9. The effects of the split configuration and diameter of SG stem on flexural, impact resistance properties and tensile properties of SG-PP composites................................................................. 49

Figure 10. The effects of the regular SG stem, small SG stem and split SG stem on sound absorption properties of SG-PP composites................................. 56

Figure 11. The effect of the diameters of the HB on flexural, impact resistant, and tensile properties. ................................................................. 58

Figure 12. The effect of the widths of OB on flexural, impact resistant and tensile properties................................................................. 60

Figure 13. The effect of the lengths of OB on flexural, impact resistant, and tensile properties................................................................. 62
Figure 14. The effect of lengths of HF on flexural, impact resistant, and tensile properties. 65

Figure 15. The comparison of the sound absorption properties among lightweight composites reinforced by HB, OB, HF and jute fibers, respectively. 72
CHAPTER 1: INTRODUCTION

In this thesis, three biological materials (wheat straws (WS), switchgrass stems (SG) and hop bines (HB)) were chosen as reinforcement materials to make composites with polypropylene as a matrix material. The reasons to choose these biological materials as reinforcement materials in composites were because of their advantages. For example, they are abundant at low cost, annually renewable for sustainable economy, and have no waste disposal problems (Micusik et al., 2006).

For wheat straw, the annual worldwide production was estimated to be approximately 540 million tons in 2007 (Reddy and Yang, 2007 a). After harvest, much of this agricultural waste is left on the ground to decompose (Schirp et al., 2006). In some parts of the world, WS is burnt in open fields, causing air pollution (Li et al., 2007; Alemdar et al., 2008). Although a small portion of WS has been used as animal feed-stock and bedding (Panthapulakkal et al., 2006), industrial applications of WS are still under investigations.

SG is both a perennial and self-seeding crop. In addition, SG can grow on marginal lands and requires relatively moderate levels of chemical fertilizers. Generally, SG is considered a resource-efficient, low-input energy crop from farmland (Goel et al., 2000; Law et al., 2001; Reddy and Yang, 2007 b).

The HB which form a major portion of the plant are discarded after the farmers harvest the hop flowers that are primarily used as a flavoring and stability agent in beer production. The annual production of hop bines in the USA was estimated to be
approximately 652.5 million pounds in 2006 (based on the information given by the
Washington Hop Commission that there usually are 29000 acres of hop bines grown
annually in US, 1500 bines per acre and 15 pounds per bine).

If these agricultural byproducts such as WS and HB and natural cellulose
resource such as SG can be used properly for industrial applications, the potential
value of the crops can be increased, the income of the farmers may also be increased
and even new jobs may be created in the United States.

In my research, a novel method was applied to make composites reinforced with
long WS, SW stems and HB (up to 10 cm) instead of particles from other sources. A
preformed spunbonded nonwoven polypropylene (PP) web was used as a matrix.
Straws, stems or bines were spread randomly on the web, and then PP webs
containing the reinforcements on the top were stacked layer by layer and extra layers
of web were placed on both surfaces. Finally, the stacked layers were compression
molded to make composites. This method not only makes the long reinforcing
materials to possibly be used in the composites, but also is suited for large scale and
continuous manufacture in the industry since PP web can be used. To the best of our
knowledge, no literature exists related to lightweight composites from long size WS,
SG and HB with the matrix in web form.

There were 51.52 million automobiles sold in the world in 2006; North America
dominated with 36.2% of the total consumption (Scotiabank Group 2007). Therefore,
utilization of composites reinforced by WS, SG stems and HB in the automotive
composites industry alone may potentially lead to utilization of a substantial quantity
of WS, SG, and HB. Natural fibers such as jute, flax, and hemp are currently being
used to reinforce composites in the automotive industry. However, WS, SG and HB
are abundant and at a low cost, for example WS is available for around $0.04/Kg, the
cost to collect WS from the field (Reddy and Yang, 2007 a). The price for natural
cellulose fibers is much higher, for example jute price was about $1.5/kg in May,
2008 (Worldjute.com, 2008). Therefore, a substantial cost advantage can be achieved
from composites reinforced with WS. Besides the automotive industry, composites
from WS, SG and HB can also be used as ceiling tiles and office panels in the
construction and furniture industries.

The composites used as structural parts in automotive interior are required to be
light in weight. Lightweight composites are those composites with the densities lower
than the summation of the density contributions from all the materials used to build
the composites (Huda and Yang, 2009a, 2009b, 2008a and 2008b). For example,
composites made from 50% polypropylene (density of 0.9 g/cm$^3$) and 50% jute fibers
(density of 1.5 g/cm$^3$) with a density of 0.5 g/cm$^3$ are considered lightweight
composites because $0.5 < (0.5\times0.9 + 0.5\times1.5) = 1.2$. The composites are the
lightweight composites.

Obviously, the only way to make the lightweight composites using relatively
heavier materials is to create voids inside the composites. These voids create defects
and decrease the mechanical properties of the composites. Thus, the density of the
composites, which is highly related to the amount of voids in the composites,
becomes an important parameter for the mechanical properties of composites. In this
research the properties from WS-PP, SG-PP and HB-PP composites were compared with the properties from jute-PP composites with the same density instead of the properties from some compact composites with much higher density reported elsewhere.
CHAPTER 2: LITERATURE REVIEW

To the best of our knowledge, there is no published literature about lightweight composites reinforced by long biological materials (1 to 10 cm) with matrix materials in web form. There is also no literature found about composites from HB. However, there are several literatures found about conventional composites reinforced by biological materials such as WS, rice straws, corn stalks and SG. With regard to those conventional composites reinforced by biological materials, a major approach is to chop and mill biological materials directly into particles and then use the particles to make composites. (Buzarovska et al., 2008; Han, 2001; Hassan et al., 2003; Hervillard et al., 2007; Hornsby et al., 1997a; Le Digabel et al., 2004; Micsik et al., 2006; Mo et al., 2005; Panthapulakkal and Sain, 2006; Schirp et al., 2006a, 2006b; Shakeri and Hashemi, 2004; Yao et al., 2008). Other approaches include acid hydrolysis before milling (Digabel et al., 2004), steam explosion before chopping (Avella et al., 1995), chemical pulping before shearing (Panthapulakkal et al., 2006), and a chemi-mechanical technique to produce nanofibers (Alemdar and Sain, 2008). The most commonly used matrix materials for the biological materials composites are PP and high-density polyethylene (Digabel et al., 2004; Frounchi et al., 2007; Hornsby et al., 1997b; Johnson et al., 1999; Mengeloglu and Karakus, 2008; Panthapulakkal et al., 2006; Panthapulakkal and Sain, 2006; Schirp et al., 2006a, 2006b; Shakeri and Hashemi, 2004). Other matrix materials used for the biological materials reinforced
composites include urea formaldehyde (UF) resin (Han, 2001; Zhang et al. 2003) or melamine-UF resin (Hervillard et al., 2007), novolac resin (Mishra and Patil, 2004), starch-based thermoplastic polymer (Alemdar and Sain, 2008), polymeric methylene di-phenyl diisocyanate resin (Frounchi et al., 2007) and phenol-formaldehyde resin (Hervillard et al., 2007). Various coupling agents (Frounchi et al., 2007; Han, 2001; Hornsby et al., 1997a; Mishra and Patil, 2004; Schirp et al., 2006a; Shakeri and Hashemi, 2004) or compatibilizers (Digabel et al., 2004; Frounchi et al. 2007; Panthapulakkal and Sain, 2006; Panthapulakkal et al. 2006) were also used to increase the adhesion between biological materials and resin for the improvement of the mechanical properties of the composites. In some literature, WS was treated with fungus (Panthapulakkal and Sain, 2006, Schirp et al., 2006a, 2006b) or enzymes (Zhang et al., 2003) to improve its interactions with the matrix materials. Extrusion (Digabel et al., 2004, Hornsby et al., 1997a; Johnson et al., 1997; Panthapulakkal et al., 2005; Schirp et al., 2006a, 2006 b; Shakeri and Hashemi, 2004) and injection molding (Frounchi et al., 2007; Hornsby et al., 1997a; Johnson et al., 1999; Panthapulakkal et al., 2006; Panthapulakkal and Sain, 2006) were the most frequently used methods for making the composites, while compression molding (Avella et al., 1995; Frounchi et al., 2007; Hervillard et al., 2007) was also reported.

Most of the composites reported in the literature utilized straws in particle form at lengths ranging from 0.5 to 5 mm. The mechanical properties could potentially be increased by using longer straws. Chemical treatment to extract useable reinforcing materials from WS or other biological materials to improve the adhesion between
straws and the matrix polymer not only increases manufacturing cost but also poses an environmental challenge. Thus, in my research, the newly developed method not only utilizes the long biological materials but also avoids the chemical treatment during the composites making process.
CHAPTER 3: OBJECTIVES

The objective of this research was to utilize long and non-chemically treated WS, SG stems and HB to make lightweight and thermoplastic composites through a simple and cost effective method. And try to understand the relationship between the mechanical and sound absorption properties, and the different composites making parameters.

The lightweight composites were developed for industrial applications. The composites were intended for use as headliner composites in automobile interiors. They can also be used in the construction industry for office and furniture panels.
CHAPTER 4: EXPERIMENTAL

4.1 Materials

WS were obtained from fully mature wheat crops. The major constituents of WS are 71.24% cellulose and hemicellulose, 23.22% lignin and 5.54% ash (Zhang et al., 2003). The wheat straw was cut into specific lengths (1, 5, and 10 cm) in order to investigate effect of length on composites properties. To study the effect of the surface area of WS on composite, straws were split in half along longitudinally using a knife to double the surface area. Straws were further split into quarter to investigate the effect of aspect ratio (length/width) on mechanical properties. In order to investigate the feasibility of utilizing a mechanical device to split WS for mass production, a two-roller laboratory milling machine (KICE Industries incorporation, Wichita, Kansas) was used to split WS.

Switchgrass was obtained from an experimental plot at the University of Nebraska-Lincoln. Stems were manually separated from the plants. The diameters of the SG stems ranges from 4.0 mm to 1.2 mm. SG stems were cut into certain length (1, 5, and 10 cm) in order to investigate the effect of length on composites properties. The effect of the stem diameter was also studied; two different diameters of stems (3.31 mm and 1.61 mm) were used to make composites and mechanical properties were compared. In order to study the effect of surface area of SG on composite, stems were split along the longitudinal direction using a two-roller laboratory milling machine.
HB was from research fields of Washington State University. Different parts of HB were used in the composites. Large mature bines (7 to 16 mm in diameter) were too thick to be used in the composites, thus the outer bark of bines (OB) was peeled from these bines and used as reinforcement. The effect of various widths and lengths of the OB on the composite properties was investigated. The peeled bark from the mature bines was used to extract fibers, which are referred to as hop fiber (HF), to make composites with PP fiber. In addition, bines with an average diameter of 2.6 mm (standard deviation 0.3) and branches with an average diameter of 1.3 mm and standard deviation of 0.3 were collected to make composites. The 2.6 mm diameter bines were from the middle and top of the plant and are referred to as regular bines in this paper. The 1.3 mm branches were more common in the top of hop plants and are referred to as thin branches.

Spunbonded PP web was provided by Spunfab, Ltd. (Cuyahoga Falls, OH). The weight/area of the web is 23.7 g/m², melting temperature is 162 °C, Melt Flow Index (MFI) is 38 g/10 min measured at 230°C, and density is 0.90 g/m³.

PP fiber was bought from Drake Extrusion (Martinsville, VA). The density of the PP fiber is 0.90 g/cm³, melting temperature is 162 °C, and Melt Flow Index (MFI) is 20 g/10 min measured at 230°C.

4.2 Composite Fabrication Procedure

The weight/area of all composites was set for 1500 g/m² at an area of 25.4 cm ×
Metal spacers were used to set a thickness of 3.2 mm during the compression molding, thus the density of the composites was 0.47 g/cm$^3$. The nonwoven PP webs were laid on a smooth table from a let-off shaft. Based on the concentration of reinforcement materials (WS, SG stems, HB, OB and HF), composite weight, and web weight/area, the total area of required web was calculated and translated to the number of pieces of 25.4 cm × 30.5 cm PP web, which were left on the table. Weighed reinforcement materials were laid randomly on the web through sprinkling in order to achieve random and homogeneous distribution. Some reinforcement materials protruded from the edges of the web and were cut and put back on the web. The web with reinforcement materials on top was cut carefully into 25.4 cm × 30.5 cm pieces and stacked one by one. Equal numbers of layers of PP webs were placed at the top and bottom of the composites with 98.6 g/m$^2$ weight/area to achieve smooth surfaces and to balance the composites. This also provided smoothness and kept the reinforcement materials at the top and bottom from being exposed, reduced moisture absorption, and created an I-beam structure, which led to increased mechanical properties. The stacked layers were placed in between two aluminum sheets coated with Teflon and pressed in a laboratory-scale press (Carver, Inc., Wabash, IN, USA) preheated to the desired temperature. The thickness was controlled by metal spacers.

This method is different from the ones used for typical compact composites reported in other literature because in this method, composites are made with built-in voids leading to lighter composites compared with the consolidated ones. After a desired holding temperature and time to make PP melt and bond to the reinforcement
materials, the press was turned off and the cooling system was turned on. When the temperature decreased to about 35°C, the composite was removed from the press.

For composites reinforced by HF and jute fiber, the composites making method was different. The HF, jute fiber and PP fiber were first individually opened in a “Louet” electric carder, mixed in specific weight ratio and carded together in the same carder until fibers are mixed homogenously. Well-mixed fibers were then separated from the mat in tiny bundles and placed in a 30.5 cm long, 25.4 cm wide, and 18.4 cm high rectangular wooden container. Tap water was sprayed on the fiber mix at high velocity using a multi-purpose nozzle and further mixed by hand. After a visibly homogeneous mat was formed, water was filtered out from the top and the container was tilted for the rest of the water to drain out due to gravity. The wet mat was transferred to an oven and dried for 24 hours at 85 °C to drive off the remaining water from the fiber mat. The dry mass was placed in between two aluminum sheets coated with Teflon and pressed in a laboratory-scale “Carver” press preheated to the desired temperature. The thickness was controlled by metal spacers. After a desired holding temperature and time to make PP melt and bond to reinforcing materials, the press was turned off and the cooling system was turned on. When the temperature decreased to about 35°C, the composite was removed from the press.

Each data point in the research was the average from five tests, which was from at least three composites made from different time using the same conditions. Samples were placed in the conditioning room set at 21 °C and 65% relative humidity for at least 48 hours before testing.
4.3 Parameters of Composites Fabrication

4.3.1 Wheat Straw Reinforced Composites

The effect of WS concentration (weight %) on flexural, impact resistance, and tensile properties was studied first. The concentrations were chosen at 40, 50, 60, 70, and 80 weight %. An attempt to use 30 weight % straw led to substantial non-uniform mechanical properties because the amount of straw was not enough to homogeneously cover the area of the composites at low concentrations. On the other hand, making composites with 80 weight % WS was extremely difficult, because there were not enough webs to hold the large quantity of straws and to prevent straws from sliding off the edges of the composites. The length of straw was first set at 10 cm because longer straws were not easy to spread evenly in the lab size composites.

The effect of straw length on flexural, impact resistance and tensile properties was studied at 60 weight % concentration, which was determined to be the optimum from the first study. The chosen WS lengths were 1, 5, and 10 cm.

The effect of split straw on flexural, impact resistance, and tensile properties was studied at 60 weight % straws and 5 cm straw length which were determined to be the optimum conditions. There were three types of split: half split along the longitudinal direction (double the surface area compared with the whole straw), quarter split straw (has the same surface area with the half split WS, but twice the aspect ratio) and mechanically split straw (using two-roller milling machine, similar aspect ratio and surface area compared with quarter split but compressed nearly 100% by the rollers).
The mechanical properties of composites prepared with various splitting configuration were compared with each other. In order to study the effect of concentration with split straws, composites with 50, 60, and 70 weight% of half-split straw were also prepared. The samples from the study of 60 weight % with 5 cm whole WS composites and split straw composites were used to study the effects of these variables on the sound absorption properties. Finally, the mechanical properties of WS-PP composites were compared with jute-PP composites, as reported by Huda and Yang (2009b) for the same weight and thickness.

### 4.3.2 Switchgrass Stems Reinforced Composites

The effect of the concentrations of the SG stem (weight %) on flexural, impact resistance, and tensile properties was studied. The concentrations were chosen at 40, 50, 60 and 70 weight %. An attempt to use 30 weight % straw led to non-uniform mechanical properties because the stems with low weight were difficult to homogeneously cover the area of the PP webs. Thus, the lack of weight of stems and the uneven distribution of stems resulted in poor mechanical properties. On the other hand, making composites with 80 weight % SG stems was extremely difficult, because there were not enough webs to hold the large quantity of the stems and to prevent the stems from sliding off the edges of the composites. The length of SG stems was set at 5 cm.

The effect of the lengths of SG stem on flexural, impact resistance and tensile properties was studied at 60 weight % concentration, which was determined to be the
optimum condition from the first study. The lengths of SG stem were chosen at 1, 5, and 10 cm.

The effect of the diameters of SG stem on flexural, impact resistance, and tensile properties was studied at 60 weight % stem and 5 cm stem length, which were determined to be the optimum conditions. The small SG stems (an average diameter of 1.60 mm with standard deviation of 0.33) grown on the top of SG plants were used to make composites, which were compared with the previous composites reinforced by the regular SG stems (an average diameter of 3.30 mm with standard deviation of 0.57).

For the study of the effect of surface area of stems on composite, the effect of the split configuration of SG stem on flexural, impact resistance, and tensile properties was studied at 60 weight % of stem and 5 cm stem length. Each stem was split along the longitudinal direction of stem into two to four parts using a two-roller milling machine (KICE Industries incorporation, Wichita, Kansas). The mechanical properties of composites reinforced by split SG stems were compared with the composites reinforced by the whole stems. The samples from the three types of composites reinforced by the regular SG stems, small SG stems and split SG stems respectively were used to study the sound absorption properties. And then, the mechanical properties of SG stem-PP composites were compared with jute-PP composites of the same density and thickness as reported by Huda and Yang (2008).

The relationship between the densities of composites and mechanical properties of the SG-PP and jute-PP composites was studied. The density was chosen at 750
g/m² (0.24 g/cm³), 1500 g/m² (0.47 g/cm³) and 3000 g/m² (0.94 g/cm³) with the same thickness of 3.2 mm. The SG-PP composites were made at 60 weight %, 5 cm long and split SG stems, and jute-PP composites were made at 60 weight % of jute concentration according to the previous studies.

4.3.3 Hop Bines Reinforced Composites

The effect of various widths and lengths of the OB on the composite properties was investigated. The bark peeled from the mature bines was cut into a length of 5 cm and widths of 2, 4 and 6mm. It was too difficult to peel uniform length of the barks with less than 2mm widths and therefore the smallest width of the barks used was 2mm. OB with widths greater than 6mm would result in inferior mechanical properties of the composites because of large aspect ratio, therefore the largest width of OB used was 6mm. Then, the width of the OB was fixed at 2 mm which were determined to be the optimum condition and the lengths of the OB were chosen at 5 cm, 7 cm and 9 cm.

In addition, bines with an average diameter of 2.6 mm (standard deviation 0.3) and branches with an average diameter of 1.3 mm and standard deviation of 0.3 were collected. The 2.6 mm diameter bines were from the middle and top of the plant and are referred to as regular bines in this paper. The 1.3 mm branches were more common in the top of hop plants and are referred to as thin branches. The length of both the regular bines and thin branches was cut to 5cm and used for the study. Our previous researches on wheat straw and switchgrass which have similar shape and
bulk density as the HB have shown that the 5 cm length of the reinforcement materials is the optimum size to achieve the best mechanical properties. Therefore, the length of HB and thin branches was also fixed at 5 cm.

The peeled bark from the mature bines was used to extract fibers according to the method previously reported (Reddy and Yang, 2005 and 2009). Briefly, the peeled barks were boiled with 1N sodium hydroxide for 30 minutes with about 20% (w/w) material in the alkali solution. Fibers obtained after the alkali treatment were washed several times in warm water and finally in dilute acetic acid (10% w/w) solution and dried under ambient conditions.

The effect of lengths of HF on flexural, impact resistance, tensile properties and sound absorption properties was studied. The mature bines were cut into 5, 10 and 20 cm to make fibers. After the chemical treatment and mechanical carding, the averages fiber lengths obtained were 4.3, 7.9 and 14.6 cm (the standard deviations were 0.8, 2.5 and 4.3 respectively).

The samples from the composites reinforced by the regular HB, OB (7.0 cm long and 2.0 mm wide) and HF (7.9 cm long) were used to study the sound absorption properties and compared with jute-PP lightweight composites of the same density and thickness as reported by Huda and Yang (Huda and Yang, 2009 b). The mechanical properties of the composites from HB, OB and HF were also compared with jute-PP composites.
4.4 Material Characterizations

4.4.1 SEM Analysis

A Hitachi model S-3000N scanning electron microscope was used for SEM (scanning electron microscope) imaging. Cross section of samples was sputter coated with gold palladium, mounted on conductive adhesive tapes, and observed under the microscope at a voltage of 25 kV.

4.4.2 Mechanical and Acoustic Testing

An MTS QTest\10 tester was used for flexural testing according to procedure A of ASTM D790-03. The size of samples was 20.3 cm × 7.6 cm with 15.2 cm support length; load cell was 500N with the crosshead speed of 10 mm/min for the three-point-bend tests. Tensile tests were carried out in an MTS tester (QTest/10) according to procedure ASTM D638-03. The machine was equipped with an extensometer (model MTS 634.11F-24) and a 500 N load cell. The samples were cut into dog-bone shape using a Type I sample template. The sample length was 165 mm, the wide section of the sample had a width of 19 mm, and the width of the narrow section was 13 mm, the gage length was 115 mm. Crosshead speed was 5 mm/min. Tensile properties of reinforcement materials were also measured by the MTS QTest\10 tester. Gage length of the straws was 50 mm. Impact resistance test was performed in a Plastic Impact Tester by Tinius Olsen (model 66) using procedure ASTM D256-03. Sample size was 63.5 mm × 10.2 mm. The notch was cut
perpendicular to the cross section. Sound absorption test was carried out in a medium size Brüel & Kjær (type 4206 A) impedance tube at 0-3 kHz frequency based on the two-microphone transfer-function method followed by procedure ASTM E1050–98. The diameter of samples was 63 mm.

4.4.3 Void Content Calculation

The volume percentage of voids which was located between the matrix and reinforcement materials was calculated based on the densities of composites, PP, the bulk densities of reinforcements and the weight fraction of materials. There are three types of voids in the composites: the voids between PP and reinforcements, the inner voids of PP and the inner voids of the reinforcement. The volume of inner voids in the molten PP is negligible (Huda and Yang, 2009b, 2008a) and the micro voids inside the reinforcements have much less influence on the mechanical properties compared to those large voids between reinforcement and matrix material. The densities of lightweight composites and PP are 0.47 and 0.90 g/cm$^3$. The bulk densities of reinforcements are tested according to glass bead displacement method (Bhatnagar and Hanna, 1995). However, fine sands with an average diameter of 0.34 ± 0.11 mm were used as the displacement medium instead of glass beads. The calculation of void content percentage of lightweight composites is as follow:

$$V_{\text{CF}} = 1 - \frac{V_{\text{PP}}}{V_{\text{compo}}} - \frac{V_{\text{reinf}}}{V_{\text{compo}}} = 1 - \frac{W_{\text{PP}} \times \rho_{\text{compo}}}{\rho_{\text{PP}}} - \frac{W_{\text{reinf}} \times \rho_{\text{compo}}}{\rho_{\text{reinf}}}$$

(1)

where $V_{\text{CF}}$ = void content fraction of the composites, $V_{\text{PP}}$ = the total volume of PP, $V_{\text{reinf}}$ = the total volume of reinforcements, $V_{\text{compo}}$ = the total volume of composites,
\[ \rho_{\text{compo}} = \text{the density of composites}, \ \rho_{\text{PP}} = \text{the density of PP}, \ \rho_{\text{reinf}} = \text{the bulk density of reinforcements}, \ W_{\text{pp}} = \text{the weight fraction of PP in the composites}, \ W_{\text{reinf}} = \text{the weight fraction of reinforcements in the composites}. \]

4.5 Statistical Method

Fisher’s Least Significant difference (LSD) was used to test the effect of various conditions on the composites properties using SAS (SAS Institute Inc., NC). The P-value was set at 0.05.
CHAPTER 5: RESULTS AND DISCUSSION

5.1 Wheat Straw Reinforced Composites

5.1.1 Effect of WS concentration on the mechanical properties of WS-PP composites

As seen in Figure 1, the flexural strength first increased significantly (P value < 0.05) from 40% to 60% of WS concentration, and then decreased significantly when the concentration increased from 60% to 70% and 80%. The modulus of elasticity increased significantly from 40% to 60% of WS concentration, and did not show statistical differences (P value > 0.05) among 60% and higher concentrations. The impact resistance did not show significant differences among the concentrations of 50%, 60% and 70%, however, there was a decrease from 70% to 80%. The tensile strength increased from 40% to 60%, and decreased from 60% to 70% and 80% of WS concentrations. Young’s modulus increased form 40% to 50%, then stayed non-significantly different among the concentrations of 50%, 60% and 70%, and then decreased from 70% to 80%
Figure 1. Effect of WS concentrations (weight %) on flexural, impact resistant and tensile properties of WS-PP composites. Composites were fixed at weight/area of 1500 g/m² and thickness of 3.2 mm (0.47 g/cm³), and pressed at 185 °C for 80s. WS were 10 cm long with whole configuration. If any two data points of the same mechanical property had totally different letters, the two data points were statistically different.
The bulk densities of straws were tested to be $0.34 \pm 0.02 \text{ g/cm}^3$ at 40% of WS concentration, $0.38 \pm 0.03 \text{ g/cm}^3$ at 50% of WS concentration, $0.41 \pm 0.02 \text{ g/cm}^3$ at 60% of WS concentration, $0.46 \pm 0.03 \text{ g/cm}^3$ at 70% of WS concentration and $0.51 \pm 0.03 \text{ g/cm}^3$ at 80% of WS concentration. The increased bulk densities of straws are because straws were compressed more tightly at higher concentrations than lower concentrations of WS. According to equation 1, the void content percentages of composites are 13.4% at 40% of WS concentration, and 12.0% at 50% of WS concentration, 10.3% at 60% of WS concentration, 12.8% at 70% of WS concentration and 15.8% at 80% of WS concentration.

When the concentrations of WS are low, at about 40%, there are not enough straws to reinforce the polymer matrix. In addition, the small amount of WS can only cover a limited area on the PP webs; thus the visible voids are formed between WS and the matrix material as demonstrated by the SEM image in Figure 2(a). The higher void percentage of composites at 40% WS than that of composites at 50% and 60% of WS leads to poor mechanical properties. With the increase of WS concentration, there is more WS to reinforce the composites. Meanwhile, the WS can cover large areas on the webs, and results in a decrease of the amount of voids in the composites. As shown in Figure 2(b) and (c), the amount of voids decreased and the straws were compressed tightly due to the high concentrations of WS. Both the increased concentration of WS and decreased voids lead to the improved properties. However, as the WS concentration increases, as high as 80%, the flexural strength, impact resistance tensile strength and Young’s modulus decrease. This is because the density
of the composites is fixed. The increase of WS concentration must decrease the weight percentage of the PP webs. These changes result in a decreased interaction or adhesion between WS stems and PP webs at higher concentrations of WS. The poor adhesion, because of the lack of matrix materials, and the high void content percentage result in the decrease of mechanical properties.
Figure 2 (a)
Figure 2. SEM images of the cross sections of WS-PP composites. Composites were fixed at weight/area of 1500 g/m² and thickness of 3.2 mm (0.47 g/cm³), and pressed at 185 °C for 80s. WS were 10 cm long with whole configuration. (a) 40 weight %, (b) 60 weight %, (c) 80 weight %, bar = 1 mm
5.1.2 Effect of straw length on the mechanical properties of WS-PP composites

As demonstrated in Figure 3, the flexural strength, modulus of elasticity, impact resistance, tensile strength and Young’s modulus initially increased significantly with the increase the lengths of WS from 1 to 5 cm. These properties were not significantly different between the composites reinforced by 5 and 10 cm long WS. However, the standard deviations of the properties from the composites reinforced by 10 cm WS were larger than that from the composites reinforced by 5 cm long WS.
Figure 3. Effect of WS lengths on flexural, impact resistant and tensile properties of WS-PP composites. Composites were fixed at weight/area of 1500 g/m² and thickness of 3.2 mm (0.47 g/cm³), and pressed at 185 °C for 80s. The concentration of whole WS was fixed at 60%. a, b, If any two data points of the same mechanical property had totally different letters, the two data points were statistically different.
The increased length with unchanged width of the WS is directly related to the increase of the aspect ratio of WS. The aspect ratio of the reinforcing materials is a critical factor in determining mechanical properties of composites (Huda and Yang, 2009b and 2008a). The larger aspect ratio leads to a better adhesion property between reinforcements and matrix materials, and finally results in the increase in mechanical properties. However, when the lengths of WS further increased from 5 to 10 cm, the distribution of the WS on PP webs became less homogeneous due to the larger size of the straws. The lessened homogeneity leads to the uneven distribution of voids and results in enlarged standard deviations of the properties.

5.1.3 Effect of splitting configuration on the mechanical properties of WS-PP composites

As shown in Figure 4, the flexural strength, modulus of elasticity and impact strength increased significantly when the whole WS were half split. Compared with the composites reinforced by half split WS, the flexural strength, modulus of elasticity, tensile strength and Young’s modulus further increased when WS were quarter split. No statistically significant differences were observed between the mechanical properties from composites reinforced by quarter split and mechanically split WS.
Figure 4. Effect of split configurations on flexural, impact resistant and tensile properties of WS-PP composites. Composites were fixed at weight/area of 1500 g/m² and thickness of 3.2 mm (0.47 g/cm³), and pressed at 185 °C for 80s. The concentration and length of WS were fixed at 60% and 5 cm respectively. If any two data points of the same mechanical property had totally different letters, the two data points were statistically different.
Splitting the straws into half essentially doubles the surface area of WS and opens up the rough hollow center rendering it accessible to PP matrix. The enlarged surface area of WS improves the adhesive property between WS and PP matrix. In addition, the half split WS have an increased aspect ratio because of the reduction of the width of WS. As discussed previously, the increased aspect ratio leads to increased mechanical properties. Some mechanical properties further increased as the half split straws were split again into quarter split. This is because the aspect ratio of WS was further increased. The composites reinforced by mechanically split WS showed similar mechanical properties to the composites reinforced by quarter split WS. These results prove that it is ideally desirable to be able to split straws in a milling machine for commercial operations.

5.1.4 Effect of WS concentration on the mechanical properties of half split WS-PP composites

As shown in Table 1, composites from half split WS had overall higher mechanical properties compared with composites from whole WS, except for impact resistance. Moreover, the effect of concentration on mechanical properties was similar for both the whole and half split straws. Based on the results, it is concluded that with whole or split configuration, the effect of concentration would most probably be the same with 60 weight % leading to the optimized mechanical properties.
Table 1. Comparison of the effect of WS concentration on mechanical properties of composites from whole and half-split WS.

<table>
<thead>
<tr>
<th>Whole/Split</th>
<th>Wt. (%)</th>
<th>FS (MPa)</th>
<th>MOE (GPa)</th>
<th>IR (J/m)</th>
<th>TS (MPa)</th>
<th>YM (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Whole</td>
<td>50</td>
<td>$11.0 \pm 1.0$</td>
<td>$1.2 \pm 0.1$</td>
<td>$52.9 \pm 15.1$</td>
<td>$5.5 \pm 0.7$</td>
<td>$739.1 \pm 78.1$</td>
</tr>
<tr>
<td>Whole</td>
<td>60</td>
<td>$12.3 \pm 0.7$</td>
<td>$1.5 \pm 0.1$</td>
<td>$46.3 \pm 10.7$</td>
<td>$5.5 \pm 0.8$</td>
<td>$840.8 \pm 87.5$</td>
</tr>
<tr>
<td>Whole</td>
<td>70</td>
<td>$10.5 \pm 0.9$</td>
<td>$1.4 \pm 0.2$</td>
<td>$45.6 \pm 13.0$</td>
<td>$4.1 \pm 0.9$</td>
<td>$763.5 \pm 91.3$</td>
</tr>
<tr>
<td>Half</td>
<td>50</td>
<td>$15.2 \pm 1.8$</td>
<td>$1.7 \pm 0.2$</td>
<td>$50.5 \pm 11.3$</td>
<td>$6.0 \pm 1.0$</td>
<td>$898.6 \pm 90.1$</td>
</tr>
<tr>
<td>Half</td>
<td>60</td>
<td>$15.7 \pm 1.0$</td>
<td>$2.0 \pm 0.1$</td>
<td>$55.8 \pm 9.7$</td>
<td>$6.5 \pm 0.9$</td>
<td>$924.3 \pm 85.8$</td>
</tr>
<tr>
<td>Half</td>
<td>70</td>
<td>$14.1 \pm 1.0$</td>
<td>$2.0 \pm 0.1$</td>
<td>$43.7 \pm 11.6$</td>
<td>$5.9 \pm 1.2$</td>
<td>$850.5 \pm 88.6$</td>
</tr>
</tbody>
</table>

Wt. = weight, FS = Flexural Strength, MOE = Modulus of Elasticity, IR = Impact Resistance, TS = Tensile Strength, YM = Young’s modulus, $a, b, c, d, e$ If any two data points in the column have totally different letters, the two data points are statistically different.
5.1.5 Effect of whole and splitting on sound absorption properties of WS-PP composites

As seen in Figure 5, the composites reinforced by WS had higher sound absorption coefficients within the range of 0.3 to 2.2 KHz than that of composites reinforced by jute fiber. This is mainly because WS has much lower bulk density than that of jute fiber. The bulk density of WS (at 60% WS concentration of composites) is approximately 0.41 g/cm$^3$ which is much lower than that of jute fiber (1.02 g/cm$^3$). The void content percentage of WS-PP composites at 60% of WS concentration is 10.3 %, which is much lower than the 51.5 % of void content of jute-PP composites. Since the sound waves are more likely to be weakened by going through different phases of materials than directly going through large voids, the lower void content can result in a better sound absorption property. Among the WS-PP composites, split WS-PP composites have generally stronger sound absorption capability than whole WS-PP composites. Although similar amount of void content in all split and whole WS-PP composites, the split WS with enlarged surface can cover more area on the web and distribute more homogeneously in the composites than the whole WS in the composites. Thus, the split WS-PP composites with smaller and more homogeneously distributed voids than those of whole WS-PP composites can result in better sound absorption properties. Sound absorption at low frequency (below 1.5 kHz) is desired for automotive composites because this frequency zone corresponds with noise from wind, tires, road, conversation, and the running engine. Thus, WS-PP composites have better sound absorption properties than jute-PP composites.
Figure 5. Effect of whole and split configurations on sound absorption properties of WS-PP composites. The sound absorption property of jute-PP composites was compared. Composites were fixed at weight/area of 1500 g/m² and thickness of 3.2 mm, and pressed at 185 °C for 80s. The concentration of reinforcements was 60%. The length of WS and jute fiber was 5 cm.
5.1.6 Comparison of WS-PP and jute-PP composites

Composites from mechanically split WS have nearly 114% higher flexural strength, 38% higher modulus of elasticity, 10% higher tensile strength, 140% higher Young’s modulus, and 50% lower impact resistance properties compared with jute composites, as shown in Table 2. Wheat straw has 62 MPa tensile strength, 0.7% breaking elongation, and 8.35 GPa modulus compared with 295 MPa tensile strength, 1.1% elongation, and 16.9 GPa modulus for jute respectively (Reddy and Yang, 2005). Although significantly lower in tensile properties, WS composites demonstrate better mechanical properties compared with jute composites mainly due to the fact that WS has a substantially lower bulk density (0.41 g/cm$^3$ at 60% of WS concentration) compared with the bulk density of jute (1.02 ± 0.04 g/cm$^3$). This very low bulk density allows WS to be packed very tightly and leaves a few voids. The inner voids in composites were reduced at high concentrations of WS, as shown in Figures 2(b) and 2(c). According to the calculation, the void content percentage of WS-PP composites at 60% of WS concentration is 10.3 %, which is much lower than the void content percentage of jute-PP composites (51.5 % of void content). The higher void percentage of jute-PP composites leads to the poor mechanical properties.
Table 2. Comparison of mechanical properties of composites from mechanically split wheat straws and jute, both with PP, at 1500 g/m², and 3.2 mm thickness (0.47 g/cm³).

<table>
<thead>
<tr>
<th>Composites</th>
<th>FS</th>
<th>MOE</th>
<th>IR</th>
<th>TS</th>
<th>YM</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mechanically</td>
<td>19.5 ± 1.2</td>
<td>2.2 ± 0.1</td>
<td>56.4 ± 10.0</td>
<td>9.8 ± 1.0</td>
<td>1126.8 ± 88.4</td>
</tr>
<tr>
<td>Split Wheat</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Straw-PP</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Natural</td>
<td>9.1 ± 0.4</td>
<td>1.6 ± 0.1</td>
<td>112.3 ± 8.6</td>
<td>8.9 ± 0.8</td>
<td>469.3 ± 30.1</td>
</tr>
<tr>
<td>Jute-PP*</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>


If any two data points in the column have totally different letters, the two data points are statistically different.
5.2 Switchgrass Stems Reinforced Composites

5.2.1 Effect of SG stem concentration on the mechanical properties of SG-PP composites

As demonstrated in Figure 6, flexural strength, modulus of elasticity, impact resistance properties and Young’s modulus increased significantly from 50 to 60% of SG stem concentration (with the P value of 0.0409 for flexural strength, <0.0001 for modulus of elasticity, 0.0081 for impact resistance and 0.0001 for Young’s modulus), and then decreased significantly from 60% to 70% of the stem concentration (with the P value of 0.0481 for flexural strength, 0.0071 for modulus of elasticity, <0.0001 for impact resistance and 0.0005 for Young’s modulus). However, the tensile strength did not show significant differences among the concentrations of 50%, 60% and 70%.
Figure 6. The effects of SG stem concentrations (40%, 50%, 60% and 70%) on flexural strength, modulus of elasticity, impact resistance, tensile strength and Young’s modulus of SG-PP composites. Composites were fixed at weight/area of 1500 g/m$^2$ and thickness of 3.2 mm, and pressed at 185 °C for 80s. SG stems were 5 cm long with regular-sized and whole configuration.
As shown in Figure 6, the void content percentages of composites showed a decrease from 23.9% to 19.8% as the concentration of SG increased from 40% to 70%. The bulk densities of SG were tested to be $0.42 \pm 0.01 \text{ g/cm}^3$ at 40% of SG concentration, $0.46 \pm 0.02 \text{ g/cm}^3$ at 50% of SG concentration, $0.49 \pm 0.01 \text{ g/cm}^3$ at 60% of SG concentration and $0.51 \pm 0.02 \text{ g/cm}^3$ at 70% of SG concentration. The increased bulk densities of SG are because SG was compressed more tightly at higher concentrations than lower concentration of SG.

When the concentrations of SG stems are low, at about 40%, there are not enough stems to reinforce the polymer matrix. In addition, the small amount of SG stems can only cover limited area on the PP webs, thus the visible voids are formed between SG stems and the matrix materials as demonstrated by the SEM image in Figure 7(a). The void content percentage of composites at 40% of SG also shows higher value than the composites with higher concentrations of SG. The voids in the composites lead to poor mechanical properties. With the increase of SG concentration, there are more SG stems to reinforce the composites. Meanwhile, the SG stems can cover large areas on the webs and occupy more volume in the composites because of the lower density of SG than PP, and results in a decrease of the amount of voids in the composites. As shown in Figure 7(b) and (c), the amount of voids decreased and the SG stems were compressed tightly due to the high concentrations of SG stems. Both the increased concentration of SG stems and decreased voids lead to the improved properties. However, as the stem concentration increases, as high as 70%,
the flexural properties, impact resistance and Young’s modulus decrease. This is because the density of the composites is fixed. The increase of SG concentration must decrease the weight percentage of the PP webs. These changes result in a decreased interaction or adhesion between SG stems and PP webs at higher concentrations of SG stems. The poor adhesion, because of the lack of matrix materials, results in the decrease of mechanical properties. However, it is difficult to explain the unchanged tensile strength among different concentrations.
Figure 7 (b)
Figure 7 (c)

Figure 7. SEM images of the cross sections of SG-PP composites. Composites were fixed at weight/area of 1500 g/m² and thickness of 3.2 mm, and pressed at 185 °C for 80s. SG stems were 5 cm long with regular-sized and whole configuration. (a) 40 Weight %, (b) 60 weight %, (c) 70 weight %, bar = 1 mm.
5.2.2 The effect of stem lengths on the mechanical properties of SG-PP composites

As shown in Figure 8, the flexural strength, modulus of elasticity, impact resistance and Young’s modulus properties first increased significantly with the increase of stem lengths from 1 to 5 cm (the P values are <0.0001, <0.0001, <0.0001 and 0.0074 for flexural strength, modulus of elasticity, impact resistance and Young’s modulus respectively) and then decreased significantly with the increase of the length from 5 to 10 cm (the P values are 0.0019, <0.0001, <0.0001 and 0.0006 for flexural strength, modulus of elasticity, impact resistance and Young’s modulus respectively). The tensile strength did not show significant differences among the lengths of 1, 5 and 10 cm.
Figure 8. The effects of lengths of regular SG stem (1cm, 5cm and 10cm) on flexural strength, modulus of elasticity, impact resistance, tensile strength and Young’s modulus of SG-PP composites. Composites were fixed at weight/area of 1500 g/m² and thickness of 3.2 mm, and pressed at 185 °C for 80s. The concentration of SG stems was 60%.
The increased length with unchanged width of the stem is directly related to the increase of the aspect ratio of the stem. Although the aspect ratio is usually applicable for much finer things such as fibers, the explanation using the theory of aspect ratio is also applicable for the discussion of the configurations of stems. The aspect ratio of the reinforcing materials is a critical factor in determining mechanical properties of composites (Huda and Yang, 2008a). The larger aspect ratio leads to a better adhesion property between reinforcements and matrix materials, and finally results in the increase in mechanical properties. However, when the lengths of SG stem further increased from 5 to 10 cm, the distribution of the stems on PP webs became less homogeneous due to the larger size of the stems. The lessened homogeneity leads to an increase in the amounts of defects, and results in the reduction of the mechanical properties.

5.2.3 The effect of stem diameters on the mechanical properties of SG-PP composites

Compared with the composites reinforced by the regular SG stems, the composites reinforced by the small SG stems have significantly higher modulus of elasticity and tensile strength (P values are 0.0026 and 0.0032 for modulus of elasticity and tensile strength respectively), as shown in Figure 9. Although the tensile properties of the small SG stem are significantly worse than the regular SG stem (the datum for the stem properties were presented at the experimental section), the aspect
ratio and bulk density play more important roles in determining the properties of the composites. The decreased diameter with unchanged length of the stem leads to an increased aspect ratio which helps to improve the adhesion between the stems and the PP to achieve higher properties of composites. The lower bulk density of the small SG stem \((0.42 \pm 0.03 \text{ g/cm}^3 \text{ at 60\% of SG concentration})\) than that of the regular stem \((0.49 \pm 0.01 \text{ g/cm}^3 \text{ at 60\% of SG concentration})\) allows the stems to be used in larger volume and cover more area on the webs to decrease voids and improve the properties. However, other mechanical properties of the composites reinforced by the small SG stems do not show significant differences from the composites using regular SG stems.
Figure 9. The effects of the split configuration and diameter of SG stem on flexural, impact resistance properties and tensile properties of SG-PP composites. Composites were fixed at weight/area of 1500 g/m² and thickness of 3.2 mm, and pressed at 185 °C for 80s. The concentration of SG stem was 60%. The length of the SG stem was 5 cm.
5.2.4 The effect of the split configuration on the mechanical properties of SG-PP composites

As shown in Figure 9, the flexural strength, modulus of elasticity, tensile strength and Young’s modulus of the composites reinforced by the split SG stems were significantly higher than those of the composites reinforced by the regular SG stems (the P values were 0.0003, <0.0001, 0.0004 and <0.0001 for flexural strength, modulus of elasticity, tensile strength and Young’s modulus respectively). The regular SG stems (round with a hollow center) were split into two to four parts in order to increase the aspect ratio and enlarge the surface area. The opened inner and rough surface can improve the adhesive property between the stems and the matrix materials. The increased aspect ratio also leads to the improved mechanical properties.

5.2.5 Comparison of the mechanical properties between SG-PP and jute-PP composites

As shown in Table 3, composites from mechanically split SG stem had nearly 56% higher flexural strength, 19% higher modulus of elasticity, 15% higher impact resistance, 63% higher Young’s modulus, and 52% lower tensile strength compared with jute composites based on the data of jute-PP composites reported by Huda and Yang (2009 b). The P values were <0.0001, 0.0164, 0.0317, <0.0001 and <0.0001 for flexural strength, modulus of elasticity, impact resistance, tensile strength and
Young’s modulus respectively. However, the tensile properties of SG stems were much worse than that of jute fiber. The regular SG stem had 113.4 MPa tenacity, 0.9% breaking elongation, and 15.1 GPa modulus compared with 295 MPa tenacity, 1.1% breaking elongation, and 16.9 GPa modulus for jute (Huda and Yang, 2009 a). The reasons for the better mechanical properties, except the tensile strength, of composites reinforced by SG stems are mainly because of the low bulk density of SG stem. The bulk density of the stem is $0.49 \pm 0.01 \text{ g/cm}^3$ at 60% of SG concentration. That is lower than the bulk density of jute fiber ($1.02 \pm 0.04 \text{ g/cm}^3$). The void content percentage of SG-PP composites at 60% of SG concentration is 21.6%, which is much lower than the void content percentage of jute-PP composites (51.5% of void content). With the same weight of the reinforcing material in the composites, the lower bulk density of the stem allows the SG stems to be used in a higher volume that leads the stems to cover a larger area on the webs and to be packed very tightly in composites. During compression molding, the voids between stems and PP are reduced, due to the highly packed SG stems, to improve the mechanical properties.
Table 3. Comparison of mechanical properties of composites from mechanically split SG stem and jute. Both composites were fixed at weight/area of 1500 g/m² and thickness of 3.2 mm (0.47 g/cm³), and pressed at 185 °C for 80s. The concentration of SG stem and jute was 60%. The length of the split SG stem and jute fiber was 5 cm.

<table>
<thead>
<tr>
<th>Composites</th>
<th>FS (MPa)</th>
<th>MOE (GPa)</th>
<th>IR (J/m)</th>
<th>TS (MPa)</th>
<th>YM (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mechanically Split SG Stem-PP</td>
<td>14.2 ± 1.0</td>
<td>1.9 ± 1.0</td>
<td>129.4 ± 11.6</td>
<td>4.3 ± 0.6</td>
<td>763.6 ± 58.4</td>
</tr>
<tr>
<td>Natural Jute-PP*</td>
<td>9.1 ± 0.4</td>
<td>1.6 ± 1.0</td>
<td>112.3 ± 8.6</td>
<td>8.9 ± 0.8</td>
<td>469.3 ± 30.1</td>
</tr>
</tbody>
</table>

5.2.6 Effect of composites densities on mechanical properties of SG-PP and jute-PP composites

For lightweight composites, the density of composites plays a critical role in determining the mechanical properties of the composites. When the density of composites is low, there are many voids in the composites. These voids create defects and decrease the mechanical properties of composites. Increasing of the density of composites leads to increase weight of both reinforcing materials and matrix materials, as well as a decrease of the amount in voids. These changes lead to better mechanical properties than the composites at low density. As shown in Table 4, both mechanical properties of SG stem-PP and jute-PP composites increase greatly as the densities increase. The relationship between densities (750, 1500 and 3000 g/m$^2$ with fixed thickness of 3.2 mm) and mechanical properties was found to be linear with $R^2$ higher than 0.92.
Table 4. Comparison of mechanical properties of composites among different densities of composites. Both composites were fixed at a thickness of 3.2 mm, and pressed at 185 °C for 80s. The concentration of SG stem and jute was 60%. The length of the split SG stem and jute fiber was 5 cm. Different composites densities were chosen at 750 g/m² (0.24 g/cm³), 1500 g/m² (0.47 g/cm³) and 3000 g/m² (0.94 g/cm³) with the same thickness of 3.2 mm.

<table>
<thead>
<tr>
<th>Composites Density (g/m³)</th>
<th>0.24</th>
<th>0.47*</th>
<th>0.94</th>
<th>R²</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flexural Strength (Mpa)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SG</td>
<td>2.8 ±0.3</td>
<td>14.2 ±1.0</td>
<td>37.4 ±1.8</td>
<td>0.99</td>
</tr>
<tr>
<td>Jute</td>
<td>1.5 ±0.3</td>
<td>9.1 ±0.4</td>
<td>25.3 ±2.1</td>
<td>0.99</td>
</tr>
<tr>
<td>Modulus of Elasticity (Mpa)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SG</td>
<td>442.3 ±57.1</td>
<td>1917.6 ±106.8</td>
<td>5151.5 ±223.7</td>
<td>0.99</td>
</tr>
<tr>
<td>Jute</td>
<td>227.2 ±95.6</td>
<td>1635.0 ±99.0</td>
<td>4100.8 ±178.1</td>
<td>0.99</td>
</tr>
<tr>
<td>Impact Resistance (J/m)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SG</td>
<td>0</td>
<td>129.4 ±11.6</td>
<td>259.5 ±29.0</td>
<td>0.97</td>
</tr>
<tr>
<td>Jute</td>
<td>24.3 ±4.5</td>
<td>112.3 ±8.6</td>
<td>464.2 ±21.4</td>
<td>0.96</td>
</tr>
<tr>
<td>Tensile Strength (MPa)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SG</td>
<td>0</td>
<td>4.3 ±0.6</td>
<td>12.2 ±0.9</td>
<td>0.99</td>
</tr>
<tr>
<td>Jute</td>
<td>1.2 ±0.2</td>
<td>8.9 ±0.8</td>
<td>16.2 ±0.6</td>
<td>0.94</td>
</tr>
<tr>
<td>Young’s Modulus (MPa)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SG</td>
<td>0</td>
<td>764.2 ±58.4</td>
<td>1435 ±61.5</td>
<td>0.95</td>
</tr>
<tr>
<td>Jute</td>
<td>81.1 ±15.2</td>
<td>469.3 ±30.1</td>
<td>1004.8 ±67.3</td>
<td>0.99</td>
</tr>
</tbody>
</table>

* Jute composites (0.47 g/m³) data is from Huda and Yang 2009.
5.2.7 Effect of the whole and split configurations of SG stems on sound absorption properties of SG-PP composites

As seen in Figure 10, composites from the regular SG stem have slightly higher sound absorption coefficients than composites from the small SG stem and split SG stem. The composites reinforced by the split SG stems have the lowest sound absorption coefficients in the comparison. This is because the split SG stems with larger surface area can cover more area in the composites, and are highly packed in the composites and leave a small amount of micro voids in the composites. The micro voids can help absorb the energy of sound waves when sound waves travel through different phases. Thus, the lower micro-voids content of composites leads to lower sound absorption abilities compared to the composites reinforced by the regular and small SG stems. Compared to jute-PP composites, the composites reinforced by the whole SG stems (the regular and small SG stems) have similar sound absorption properties, while the composites reinforced by the split SG stems have slightly worse property than jute-PP composites. However, good sound absorption property at low frequency (below 1.5 kHz) is desired for automotive composites because this frequency zone corresponds with noise from tires, the running engine, road, conversations, and wind. Thus SG-PP composites have similar sound absorption behavior as the jute-PP composites.
Figure 10. The effects of the regular SG stem (3.31 mm average in diameter), small SG stem (1.61 mm average in diameter) and split SG stem on sound absorption properties of SG-PP composites. The sound absorption of jute-PP composites was compared. Composites were fixed at weight/area of 1500 g/m² and thickness of 3.2 mm, and pressed at 185 °C for 80s. The concentration of reinforcements was 60%. The length of SG stem or jute fiber was 5 cm.
5.3 Hop Bines Reinforced Composites

5.3.1 Effect of regular HB and thin branches on the mechanical properties of composites

As illustrated in Figure 11, the composites reinforced by regular HB had significantly higher flexural strength, modulus of elasticity and impact resistance (the P values were 0.0349 for flexural strength, <0.0001 for modulus of elasticity and 0.0038 for impact resistance) than the composites reinforced by thin branches. The tensile strength and Young’s modulus from the composites reinforced by regular HB and thin branches were similar.
Figure 11. The effect of the diameters of the HB (regular and small size) on flexural, impact resistant, and tensile properties. Composites were manufactured with a weight/area of 1500 g/m² and thickness of 3.2 mm, and compression molded at 185 °C for 80s. The concentration and length of the regular bines and thin branches were fixed at 60 weight % and 5 cm long. The average diameters of regular and thin branches were 2.6 mm and 1.3 mm respectively.
The better mechanical properties of composites reinforced by regular HB are mainly related to its lower bulk density and better tensile properties than the thin branches. As reported in the experimental part, the bulk density of the regular HB was 0.65 g/cm$^3$ which is lower than that of thin branches (0.69 g/cm$^3$). The regular HB also has better tensile strength and modulus than the thin branches (reported in materials part). The bulk density of the reinforcements is a very important parameter in lightweight composites. With the same weight of the reinforcing materials in the composites, the bines with low bulk density are used in a high volume, which results in composites with decreased voids between bines and PP, leading to an improvement in the mechanical properties. The better tensile properties of the regular HB than thin branches also provide better reinforcement to the composites.

**5.3.2 Effect of widths of OB on the mechanical properties of composites**

As shown in Figure 12, the composites reinforced by OB with 2mm width possessed better mechanical properties than the composites reinforced by 4mm and 6mm wide OB. Compared with the composites reinforced by 4 mm OB, the composites reinforced by 2 mm wide OB have significantly higher flexural strength, modulus of elasticity, impact resistance and Young’s modulus (the P values were 0.0375, 0.0165, 0.0038 and 0.0009 respectively). There were no statistical difference in mechanical properties between the composites reinforced by 4 mm OB and those reinforced by 6 mm OB.
Figure 12. The effect of the widths of OB on flexural, impact resistant and tensile properties. Composites were made with a weight/area of 1500 g/m² and thickness of 3.2 mm, and pressed at 185 °C for 80s. The concentration and length of the OB were fixed at 60 weight % and 5 cm long. The widths of OB were chosen as 2, 4, and 6 mm.
Increasing the width without changing the length of the OB decreased the aspect ratio of the bark. The aspect ratio of the reinforcing materials is a critical factor in determining mechanical properties of composites (Huda and Yang, 2008a and 2009 a, b). A larger aspect ratio leads to a better adhesion between reinforcements and matrix materials, and results in the improved mechanical properties. Although the mechanical properties of composites reinforced by 4 mm wide OB and 6 mm wide OB did not show significant difference, the standard deviations for the composites reinforced by the OB with 6 mm width were larger than the composites reinforced by the OB with 4 mm width. The larger standard deviations are because of the increased size of OB from 4 mm to 6 mm. The increased size makes the OB more difficult to be spread homogeneously on the PP webs, resulting in larger deviations of the properties.

5.3.3 Effect of length of OB on the mechanical properties of composites

As shown in Figure 13, the modulus of elasticity and tensile strength significantly increased when the lengths of the OB increased from 5 cm to 7 cm (the P values were 0.0053 and 0.0322). However, other properties did not show statistically significant differences between the composites reinforced by 5 and 7 cm long OB. When the length of the OB further increased from 7 to 9 cm, none of the mechanical properties show statistically significant differences. However, the standard deviations of the properties from the composites reinforced by 9 cm were larger than those properties from the composites reinforced by 7 cm OB.
Figure 13. The effect of the lengths of OB on flexural, impact resistant, and tensile properties. Composites were developed had a weight/area of 1500 g/m$^2$ and thickness of 3.2 mm, and pressed at 185 °C for 80s. The concentration and width of the OB were fixed at 60 weight % and 2 mm wide. The lengths of the OB were chosen as 5 cm, 7 cm and 9 cm.
Increased length with unchanged width of the OB increases the aspect ratio. As discussed above, the aspect ratio of the reinforcing materials plays an important role in determining mechanical properties of composites. The larger aspect ratio results in better adhesion between reinforcements and matrix materials leading to the increase in mechanical properties. However, in this study, many properties, such as flexural strength, impact resistance and Young’s modulus, did not show significant increases, and the reasons are not clear. When the lengths of OB were further increased from 7 to 9 cm, the distribution of the OB on PP webs becomes less homogeneous due to the larger size of the barks. Composites with poor homogeneity of the reinforcing and matrix materials lead to defects, therefore large standard deviations of the mechanical properties were observed.

5.3.4 Effect of length of HF on the mechanical properties of composites

As shown in Figure 14, the flexural strength, modulus of elasticity, impact resistance, tensile strength and Young’s modulus increased significantly when the fiber length increased from 4.3 to 7.9 cm (the P values were 0.0063, <0.0001, 0.0028, <0.0001 and 0.0004 for flexural strength, modulus of elasticity, impact resistance, tensile strength and Young’s modulus respectively). When the fiber length further increased from 7.9 to 14.6 cm, the tensile strength and Young’s modulus decreased significantly (the P values were 0.0050 and 0.0462). The flexural strength, modulus of elasticity and impact resistance did not show statistical differences between the
composites reinforced by 7.9 cm long HF and 14.6 cm long HF.
Figure 14. The effect of lengths of HF on flexural, impact resistant, and tensile properties. Composites were made with weight/area of 1500 g/m² and thickness of 3.2 mm, and pressed at 185 °C for 80s. The concentration of HF was 60%. The averages of fiber lengths were 4.3, 7.9 and 14.6 cm.
Increasing the length of HF from 4.3 to 7.9 cm led to an increased aspect ratio, and therefore, an improved adhesion between the reinforcements and matrix materials, and improved mechanical properties. When the length of HF further increased from 7.9 to 14.6 cm. Although fibers had increased aspect ratio, the longer fibers were more likely to entangle with each other, and posed difficulties in the carding and mixing processes leading to poor homogeneity and, therefore, inferior mechanical properties.

5.3.5 Comparison of the mechanical properties among HB-PP, OB-PP and HF-PP composites

As shown in Table 5, the OB reinforced composites generally have better mechanical properties than the composites reinforced by HF and regular HB. Compared with the composites reinforced by HF (7.9 cm long), the composites reinforced by OB (7 cm long and 2 mm wide) had significantly higher flexural strength, modulus of elasticity and Young’s modulus, significantly lower impact resistance, and similar tensile strength. Compared with the composites reinforced by regular HB, the composites reinforced by OB had significantly higher flexural strength, modulus of elasticity, impact resistance, tensile strength and Young’s modulus.
Table 5. Comparison of mechanical properties of composites from regular HB (5 cm long), OB (7 cm long and 2 mm wide), HF (7.9 cm long) and jute fibers. All composites were manufactured with a weight/area of 1500 g/m² and thickness of 3.2 mm (0.47 g/cm³), and pressed at 185 °C for 80s. The concentration of reinforcements was 60%.

<table>
<thead>
<tr>
<th>Material</th>
<th>FS (MPa)</th>
<th>MOE (GPa)</th>
<th>IR (J/m)</th>
<th>TS (MPa)</th>
<th>YM (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Regular Hop</td>
<td>11.5 ± 1.0</td>
<td>1.4 ± 0.1</td>
<td>76.6 ± 10.2</td>
<td>2.0 ± 0.5</td>
<td>349.1 ± 40.2</td>
</tr>
<tr>
<td>Bine-PP</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Chemically Extract</td>
<td>5.8 ± 0.8</td>
<td>0.7 ± 0.1</td>
<td>193.9 ± 23.4</td>
<td>6.8 ± 0.7</td>
<td>410.2 ± 34.5</td>
</tr>
<tr>
<td>Bark Fiber-PP</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Outer Bark-PP</td>
<td>13.0 ± 0.9</td>
<td>1.6 ± 0.1</td>
<td>163.8 ± 18.9</td>
<td>6.7 ± 0.9</td>
<td>733.6 ± 56.8</td>
</tr>
<tr>
<td>Jute-PP*</td>
<td>9.1 ± 0.4</td>
<td>1.6 ± 0.1</td>
<td>112.3 ± 8.6</td>
<td>8.9 ± 0.8</td>
<td>469.3 ± 30.1</td>
</tr>
</tbody>
</table>

FS = Flexural Strength, MOE = Modulus of Elasticity, IR = Impact Resistance, TS = Tensile Strength, YM = Young’s Modulus, * Jute data were from Huda and Yang 2009.
The better mechanical properties, except the impact resistance, for the composites reinforced by OB compared to those reinforced by HF are mainly due to the lower bulk density of OB (0.58 g/cm$^3$) than that of HF (1.03 g/cm$^3$). With the same weight of the reinforcing materials in the lightweight composites, the lower bulk density of the OB allows the OB to be used in a higher volume which leads OB to be packed tightly with fewer voids in the composites. During compression molding, the voids between OB and PP are reduced, due to the highly packed OB, leading to improved mechanical properties. However, the higher impact resistance from composites reinforced by HF than the composites reinforced by OB is difficult to explain.

The better mechanical properties from the composites reinforced by OB than that reinforced by regular HB are mainly because of the larger aspect ratio and better tensile properties of OB than regular HB. Although the bulk density of OB is similar to that of regular HB, the cross section of the OB (an average of 2 mm wide and 0.4 mm thick) is much smaller than that of regular HB (about 2.6 mm in diameter). The flat configuration of the barks allows them to cover more area on the PP webs than the cylindrical HB and results in decreased amount of voids. The small cross section of OB also results in a large aspect ratio and improved mechanical properties. Meanwhile, the better tensile properties of OB than regular HB also help to better reinforce the mechanical properties.
5.3.6 Comparison of the mechanical properties between hop-PP and jute-PP composites

Compared with the jute-PP composites, the composites reinforced by OB have 43% higher flexural strength, 46% higher impact resistance, 56% higher Young’s modulus, similar modulus of elasticity and 33% lower tensile strength based on the data of jute-PP composites reported by Huda and Yang (2009b). Although the tensile properties of OB were much worse than that of jute fiber as shown in Table 6. The main reason for the better mechanical properties of composites reinforced by OB is the low bulk density of OB. As discussed above, the low bulk density of the reinforcement is a critical factor in determining the mechanical properties of lightweight composites.
Table 6. Properties of chemically extracted natural cellulose fibers from hop barks compared with cotton and jute.

<table>
<thead>
<tr>
<th>Fiber Properties</th>
<th>Denier</th>
<th>Length, cm</th>
<th>Strength, g/den</th>
<th>Elongation, %</th>
<th>Modulus, g/den</th>
<th>Moisture regain, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hop Bark Fibers</td>
<td>48 ± 19</td>
<td>3-20</td>
<td>4.1 ± 1.9</td>
<td>3.3 ± 1.2</td>
<td>161 ± 57</td>
<td>8.3 ± 0.4</td>
</tr>
<tr>
<td>Cotton</td>
<td>3-8</td>
<td>1.5-5.6</td>
<td>2.7-3.5</td>
<td>6.0-9.0</td>
<td>55-90</td>
<td>7.5-8.0</td>
</tr>
<tr>
<td>Jute</td>
<td>13-27</td>
<td>15-36</td>
<td>3.2-3.5</td>
<td>0.9-1.2</td>
<td>190-200</td>
<td>13.80</td>
</tr>
</tbody>
</table>

Data for cotton are from Batra (1998), jute data are from Reddy and Yang (2005).
Although the HF shows better tensile properties and similar bulk density as jute fibers, the mechanical properties (except the impact resistance) of jute-PP composites are significantly better than that of the composites reinforced by HF. The reason for the poor mechanical properties of HF-PP composites might be that some HF were entangled together after the chemical treatment and mechanical opening. The entangled fibers resulted in poor adhesion between HF and PP, and led to poor mechanical properties. However, the reason for the high impact resistance of the composites reinforced by HF is still unclear at this time.

5.3.7 Comparison of the sound absorption properties between hop-PP and jute-PP composites

As seen in Figure 15, the composites reinforced by HF have similar sound absorption performance to the composites reinforced by jute fibers. The composites reinforced by regular HB have slightly lower sound absorption behavior than jute-PP composites. However, the composites reinforced by OB showed the best sound absorption property among the composites, especially when the frequency of the sound ranged from 1.6 to 3.0 kHz.
Figure 15. The comparison of the sound absorption properties among lightweight composites reinforced by HB, OB, HF and jute fibers, respectively. Composites were manufactured with weight/area of 1500 g/m² and thickness of 3.2 mm, and pressed at 185 °C for 80s. The concentration of reinforcements was 60%. The length of regular HB and jute fibers was 5 cm. The OB was 7 cm long and 2 mm wide. The HF was 7.9 cm long.
The similar properties between the composites reinforced by HF and jute fibers are mainly due to the similar bulk density of fibers, similar sound absorption properties of cellulose fibers and similar structure of composites since both types of composites were made by the same manufacturing method. Although the bulk density of OB is similar to that of regular HB, OB has larger aspect ratio and a flat configuration which make the barks cover a larger area on the PP webs and distribute more homogeneously in the composites. The composites reinforced by OB are more compact with fewer amounts of voids than the composites reinforced by regular HB. Thus, the sound energy is more likely to be absorbed when the sound waves go through different phases of the matrix and reinforcement materials rather than travel through the voids directly. However, the composites with good sound absorption property at low frequency (below 1.5 kHz) are desired for automotive interiors because this frequency zone corresponds to noise from tires, the running engine, road, conversations, and wind. Thus composites reinforced by regular HB, OB and HF have similar sound absorption behavior as the jute-PP composites in the frequency zone between 0 to 1.5 kHz.
CHAPTER 6: CONCLUSIONS

6.1 Wheat Straw Reinforced Composites

In this research a novel method has been developed for combining of long WS with PP nonwoven webs to make composites. For whole WS-PP composites, WS at 60% weight ratio and 5 cm long are the optimal conditions for mechanical properties among the conditions studied during the compression molding at 185°C for 80 s. These same conditions used with mechanically split WS result in the best mechanical properties of composites compared to other split configurations. Compared with whole WS-PP composites, mechanically split WS-PP composites have 69% higher flexural strength, 39% higher modulus of elasticity, 18% higher impact resistance properties, 69% higher tensile strength and 26% higher Young’s modulus. This is attributed to the increased surface area and aspect ratio of split WS compared with whole WS. Composites from mechanically split wheat straws have 114% higher flexural strength, 38% higher modulus of elasticity, 10% higher tensile strength, 140% higher Young’s modulus, 50% lower impact resistance properties and better sound absorption property within the range of 0.3 to 2.2 KHz of sound frequency compared with jute composites. Except for impact resistance, the superior properties of wheat straw-PP, compared with jute-PP composites, provide an opportunity for immediate application of WS composites in structural parts of automotive interior where natural fibers are currently being used. Substantial cost advantage can also be materialized from composite manufacturing operation with WS. Utilization of WS in composites
will potentially lead to reduction in environmental pollution and provide a higher profit margin for the wheat farmers.

6.2 Switchgrass Stem Reinforced Composites

In this research, a newly developed method was applied to make lightweight composites from long SG stems and PP nonwoven webs. When the manufacturing parameters are set at 60% concentration of SG stems and 5 cm long with split configuration of stems, the SG-PP composites showed the best mechanical properties among the conditions studied during the compression molding at 185°C for 80 s. Composites from mechanically split SG stem have 56% higher flexural strength, 19% higher modulus of elasticity, 15% higher impact resistance, 63% higher Young’s modulus and 52% lower tensile strength and similar sound absorption property compared with jute-PP composites. Although the tensile properties of SG stem are much worse than jute fiber, SG stems with low bulk density are found to better reinforce the lightweight composites. Except for the tensile strength, the superior mechanical properties of SG-PP compared with jute-PP composites, provide an opportunity for applications of SG composites in automotive interiors where composites reinforced by natural fibers are currently being used as support layers, and in the construction industry with the products such as ceiling tiles and office panels.

6.3 Hop Bines Reinforced Composites

In this research, lightweight composites were developed using HB and OB as
reinforcement materials and non-woven PP webs as matrix material, the OB-PP showed better properties than similar PP composites reinforced with jute fibers. In addition, natural cellulose fibers obtained from the hop bines were also used as reinforcement in the composites. The OB-PP composites showed the best mechanical properties among the conditions studied when the OB had length of 7 cm and width of 2 mm, and the concentration of OB was set at 60% by weight. One of the findings of this research is that lightweight composites with better properties can be achieved using barks directly rather than fibers chemically extracted from the barks. In addition to better mechanical properties, using barks directly would also lead to substantial simplifications in manufacturing and decreases in costs. Composites from OB and PP webs had 43% higher flexural strength, 46% higher impact resistance, 56% higher Young’s modulus, similar modulus of elasticity and 33% lower tensile strength, and better sound absorption property compared with jute-PP composites. Although the tensile properties of OB were much worse than jute fibers, OB with low bulk density was found to better reinforce the lightweight composites. The superior mechanical properties of OB-PP, compared with jute-PP composites, provides an opportunity for applications of OB-PP composites in automotive interiors where composites reinforced by natural fibers are currently being used as support layers, and in the construction industry for products such as ceiling tiles and office panels.
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United State Department of Agriculture.

