Performance Comparison of Different Plant Fiber/Soybean Protein Adhesive Composites

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To compare the properties of different plant fiber/soybean protein adhesive composites, six types of plant fibers (rice straw, wheat straw, peanut straw, rice husk, wheat husk, and peanut shell) were selected as reinforced materials, and soybean protein adhesive was used as the adhesive. Six types of different bio-composites were prepared by the compression molding process. The Fourier transform infrared (FTIR) spectra, mechanical properties, moisture absorption, and thermal stability of the composites were measured. The tensile cross-section microstructure of the composites was examined. Results showed that the peanut straw/soybean protein adhesive composite contained more hydrophilic groups. The wheat fiber-based composites possessed more hydrogen bonds, leading to the best binding interface compatibility and mechanical properties. The wheat straw/soybean protein adhesive composite had the highest tensile strength, flexural strength, and impact strength, which were 337.7%, 638.6%, and 483.4%, compared to those of the rice husk/soybean protein adhesive composite, respectively. The peanut shell/soybean protein adhesive composite's equilibrium moisture content was the lowest (8.70%). The rice husk/soybean protein adhesive composite had the highest equilibrium moisture content (14.23%), and the best thermal stability as the initial temperature of pyrolysis was 283.4 °C and the residual mass was 34.45%.

Keywords: Biomass; Soybean protein adhesive; Mechanical property; Moisture absorption; Thermal stability

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INTRODUCTION

As a bio-based adhesive that has been used as wood adhesives for more than a century, soybean protein-based adhesives have become popular again in making environmentally friendly materials (He 2017). The use of renewable resources and biodegradable polymers to produce environmentally friendly materials is of great interest (Mantia and Morreale 2011; Zhang and Peng 2015; Torres-Tello *et al*. 2017). In this field, the study of bio-composites has become a very popular research direction due to its increasing application in building materials, interior decoration, and transportation (Pan *et al*. 2014). A bio-composite is a kind of composite material made from reinforced material with some accessories or adhesives, and the reinforced material can be carbonaceous materials (from animals, plants, or microorganisms) or biodegradable products and waste residue from agriculture or industry (Mohanty *et al*. 2002; European Parliament 2009; Liu *et al*. 2015). Bio-composites can effectively reduce the consumption of non-renewable

resources and are of great importance in environmental protection.

As a natural polymer material, plant fiber is a pollution-free and renewable natural resource. Under certain natural conditions, it can be completely decomposed into CO_2, H_2O, and other substances (Satyanarayana 2015), but the value of the materials has not received sufficient attention. Though they have been used for feed, activated carbon, building materials, and gas by chemical or biological methods, most are abandoned or burnt, exacerbating pollution (Sun *et al*. 2009). However, due to a great similarity in chemical composition with wood, other plant material can be used as a substitute for wood raw material (Xu *et al*. 2016). The main chemical components of plant fiber include cellulose, hemicellulose, lignin, and organic solvent extracts (Yan *et al*. 2014). Compared with carbon fiber and glass fiber, the presence of hydroxyl groups makes the surface of plant fiber polar and hydrophilic (Faruk *et al*. 2012; Liu *et al*. 2016). Due to a higher cellulose content, plant fibers generally have a low density, a high modulus ratio, high strength, and good toughness (Zhou *et al*. 2014; Wang *et al*. 2017a; Yao *et al*. 2017). An appropriate amount of hemicellulose acts as an adhesive during molding, which prevents the composite material from producing large deformations when used (Fu *et al*. 2010). Traces of lignin also can be used as an effective binder in improving the strength of the material in high pressure and high temperature processes (Li and Sarkanen 2017). The above studies illustrate the utility of the preparation of plant fiber composite materials.

With increasing environmental consciousness and the refinement of national environmental protection laws, many manufacturers have begun to consider natural biological adhesives, such as soybean protein adhesive, to replace synthetic adhesives (Liu 2013). Soybean protein adhesive is an environmentally friendly adhesive composed of defatted soybean powder and soybean isolated protein (Liu *et al*. 2017). It provides strong tensile, cohesive, and osmotic forces (Wang *et al*. 2016). The adhesive is prepared using defatted soybean powder as base material (Johnson *et al*. 1984). In the study of wood particleboard cemented by soybean-based adhesive, when the moisture content of surface wood core layer was less than 1%, the core layer adhesive dosage was 6% to 8%, and the water proofing agent dosage was 1% (Li *et al.* 2017). As a result, the density of particleboard was 0.65 g/cm^3 to 0.75 g/cm^3 (Li *et al.* 2017). The main physical and mechanical properties of soybean-based wood particleboard can meet the requirements of the national particleboard (GB/T 4897 2015) used in different conditions (Li *et al.* 2017). In the study of wet strength compared with soybean protein adhesive, the wet strength of the WPU-SPI adhesive, which was made by dispersing soy-oil-based waterborne polyurethane (WPU) into soy protein isolate (SPI) slurry, increased by 65%. The increase was mainly due to the interaction of small molecules with the proteins in the WPU (Liu *et al.* 2016). Additionally, -CH and -OH groups produced by the reaction between melamine and glyoxal may react with soybean-based adhesives. Therefore, the water resistance of soybean-based adhesives obtained by the preparation of the nontoxic melamine-glyoxal resin as the crosslinking agent is obviously improved (Wu *et al.* 2016). Inspired by the strong adhesion ability of mussel proteins, renewable and robust soy-based composite films were prepared from two soybean-derived industrial materials: soluble soybean polysaccharide (SSPS) and catechol-functionalized soy protein isolate (SPI-CH). The bio-mimetic adherent catechol moieties were successfully bonded in the polymeric network based on catechol cross-linking chemistry. Consequently, the films exhibited favorable water resistance and gas (water vapor) barrier performance (Wang *et al.* 2017b). In a comparative study of the properties of composites prepared by four types of biological glues and wheat straw, the mechanical properties of wheat straw composites that were

prepared with a soybean protein adhesive were obviously higher than those prepared by other biological adhesives, including those of plastic matrix composites. Additionally, they exhibited a low moisture absorption rate (Wang *et al.* 2016).

Plant fibers and soybean protein adhesive are both environmental friendly materials. They can be used to produce plant fiber/soybean protein adhesive composites, and their wastes can decompose completely into carbon dioxide, water, and plant amino acids under certain conditions. In this paper, six types of plant materials (rice straw, wheat straw, peanut straw, rice husk, wheat husk, and peanut shell) were selected to produce composites with soybean protein adhesive. The properties of the six types of plant fiber/soybean protein adhesive biomass composites were compared and analyzed.

EXPERIMENTAL

Materials

The soybean protein adhesive was supplied by TianTi KuangYe Co. (Henan, China). It had been mechanically crushed and dehydrated as a white and opaque powder with non-crystalline beads. Six types of plant fragments (rice straw, wheat straw, peanut straw, rice husk, wheat husk, and peanut shell) were collected from Lianyungang, China. The straw size was between 5 mm and 15 mm. The husk size was more than 1 mm. Plant fragments that were too long or too short were removed to reduce its random effects on the properties of the bio-composites. And the size distributions were similar between different straws and different husks, respectively. Analytically pure glycerin was purchased from Nanjing Jinling Chemical Co., Ltd. (Nanjing, China).

Preparation of Plant Fiber/soybean Protein Adhesive Composites

The crops had been washed and dried before they were disintegrated, and the soil sticking on the surface of the crops had been removed. The impurities (sand, soil, and dust) from six types of plant fragments were removed by oscillatory separation and sieving. The materials were oven-dried at 100 °C for 5 h. Oven-dried plant fragments, dried soybean protein adhesive, and waterproofing agent (glycerin) were mixed at the mass ratio of 50:10:1, respectively, and stirred to uniformity at room temperature. The pattern die was filled with mixed material uniformly. A plate vulcanization machine (XLB-0; Shunli Rubber Machinery Co., Huzhou, China) was used to compress the material at 160 °C and 6 MPa for 6 min until cooling to ambient temperature. The size of the bio-composites was 120 mm × 100 mm × 5 mm.

Performance Measure and Characterization of Plant Fiber/Soybean Protein Composites

FTIR analyses

The infrared spectrum analysis was conducted with a Nicolet iS-10 (ThermoFisher Scientific, Waltham, MA, USA). The 0.002 g sample powder was ground and dispersed with 0.2 g KBr, followed by compression to form average sections. The FT-IR spectra was recorded in a range from 4000 cm^{-1} to 400 cm^{-1}, at a resolution of 4 cm^{-1} with 32 scans.

Moisture absorption

Specimens were measured with a constant temperature and constant humidity box (HPX-160BSH-III; Xin Miao Medical Equipment Co., Shanghai, China) to determine the

equilibrium moisture of the bio-composites according to the GB/T 20312 (2006). The specimens that had been processed and dried were placed on the container baffle until the weight was constant. The temperature and relative humidity were set to 23 ± 0.5 °C and 90%, respectively. The specimens were weighed after 0 h, 6 h, 18 h, 42 h, 66 h, 90 h, 114 h, and 138 h. The maximum water absorption was reached when the qualities of the specimens no longer changed notably. Finally, the equilibrium moisture was obtained by averaging the results from three trials.

Mechanical properties measurement

The measure was carried out using an electronic universal testing machine (SANS CMT6104; MTS, Shanghai, China). Both tensile strength and tensile modulus of the composites were measured according to GB/T 1040.1 (2006). Flexural strength and flexural modulus were measured following GB/T9341 (2008). Finally, impact strength was measured in accordance with GB/T1043.1 (2008). The final results of each group were identified by averaging the results from three trials.

Micromorphology analysis

The microstructure of the material tensile section was examined with a laser microscope (OLS4100; DaoJin Enterprise Management Co., Ltd., Shanghai, China).

Thermal stability analysis

Thermogravimetric analysis (TG) and derivative thermogravimetric analysis (DTG) curves of six types of plant fiber/soybean protein adhesive composites were conducted using a simultaneous thermal analyzer (NETZSCH STA 449 Jupiter; NaiChi Science Instrument Business Co., Shanghai, China) at a heating rate of 20 °C/min from 30 °C to 600 °C. Argon was selected as protecting gas with the flow rate of 10 mL/min.

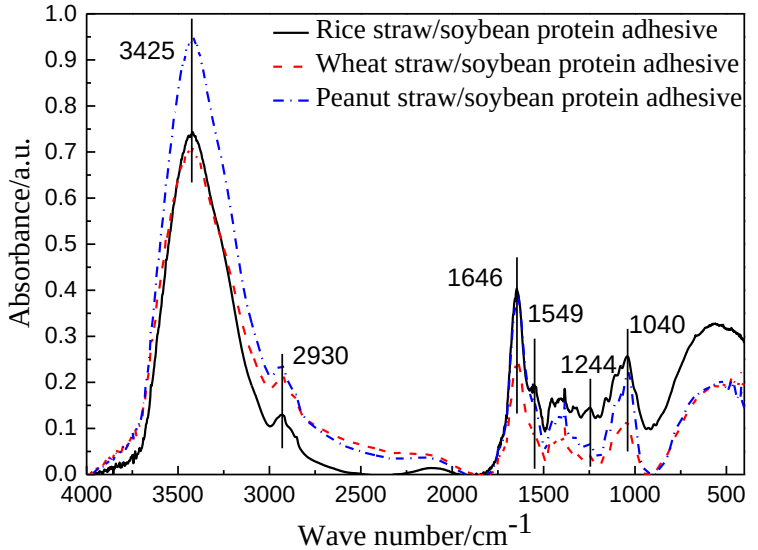
RESULTS AND DISCUSSION

FTIR Spectra of Plant Fiber/Soybean Protein Adhesive Composites

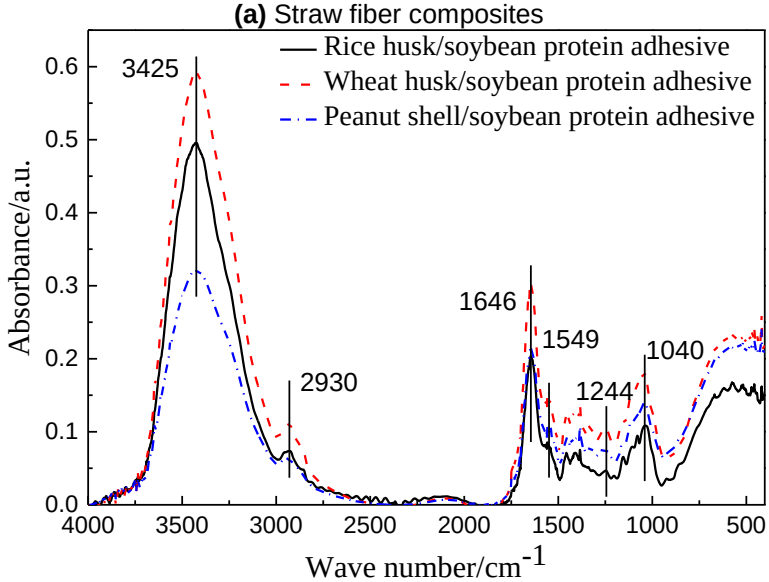
The FTIR curves of the six types of plant fiber/soybean protein adhesive composites are shown in Fig. 1. The main absorption peaks of the six types of composites were similar. The fibers of straw and husk were chosen in this experiment. The absorption peak of the composites prepared with the same type of fiber had a much closer position and more similar intensity. The wide absorption peak located at 3425 cm^{-1} represents the stretching vibration of the -OH group of cellulose, hemicellulose, or lignin and the $-NH_2$, -NH, and -OH groups of soybean protein adhesive. The absorption peak at 2930 cm^{-1} represents the absorption region of methylic $-CH_2$ groups. The absorption peaks located at 1646 cm^{-1}, 1549 cm^{-1}, and 1244 cm^{-1} represent the absorption band of proteinic Amide I, Amide II, and Amide III (containing the absorption region of the C=O groups' stretching vibration) (Meng and Ma 2001). The absorption peak located at 1040 cm^{-1} represents the absorption region of C-O in the lignin (Wang and Xu 2005).

The bio-composites included mainly hydrophilic groups such as $-NH_2$, -NH, -OH, and C=O. Comparing the intensity of each absorption peak, there were noticeable differences in the wave intensity at 3425 cm^{-1} and 1646 cm^{-1}. This result showed that straw fiber composite, especially the peanut straw/soybean protein adhesive composite, contained more hydrophilic groups. Particularly, -OH and $-NH_2$ groups had stronger

polarity. Hydrogen bonds could be combined by -OH and -NH_2 to enhance the interfacial binding force of composites, which could affect the mechanical properties and moisture absorption property of composites (Wu and Xie 2011).



(a) Straw fiber composites



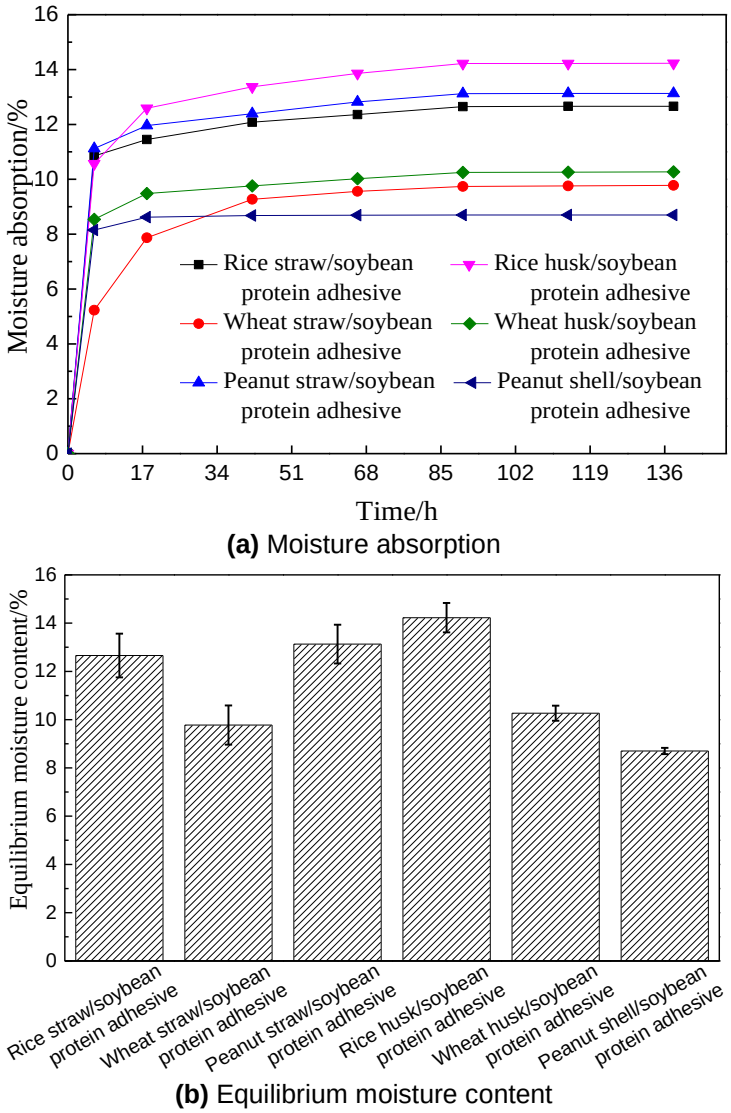
(b) Husk fiber composites

Fig. 1. FTIR spectra of plant fiber/soybean protein adhesive composites

Moisture Absorption of Plant Fiber/Soybean Protein Adhesive Composites

Figure 2 shows the moisture absorption property of the six types of plant fiber/soybean protein adhesive composites. The hygroscopicity curves of all composites were almost the same in Fig. 2(a). The moisture absorption amount between 0 h to 6 h increased quickly. Later, the moisture absorption rate decreased, and the curves tended to plateau from 18 h to 90 h. After 90 h, the moisture absorption remained largely consistent and reached saturation point. The equilibrium moisture contents of the six types of bio-composites were generally high. The rice husk/soybean protein adhesive composites had the largest equilibrium moisture content (14.23%). The peanut shell/soybean protein adhesive composites had the lowest equilibrium moisture content (8.70%). The bio-

composites have more hydrophilic groups than the traditional medium density fiberboard, but greater interface bonding force leading to the similar equilibrium moisture content in the same relative humidity (Yu *et al*. 1995).



(a) Moisture absorption

(b) Equilibrium moisture content

Fig. 2. Moisture absorption of plant fiber/soybean protein adhesive composites

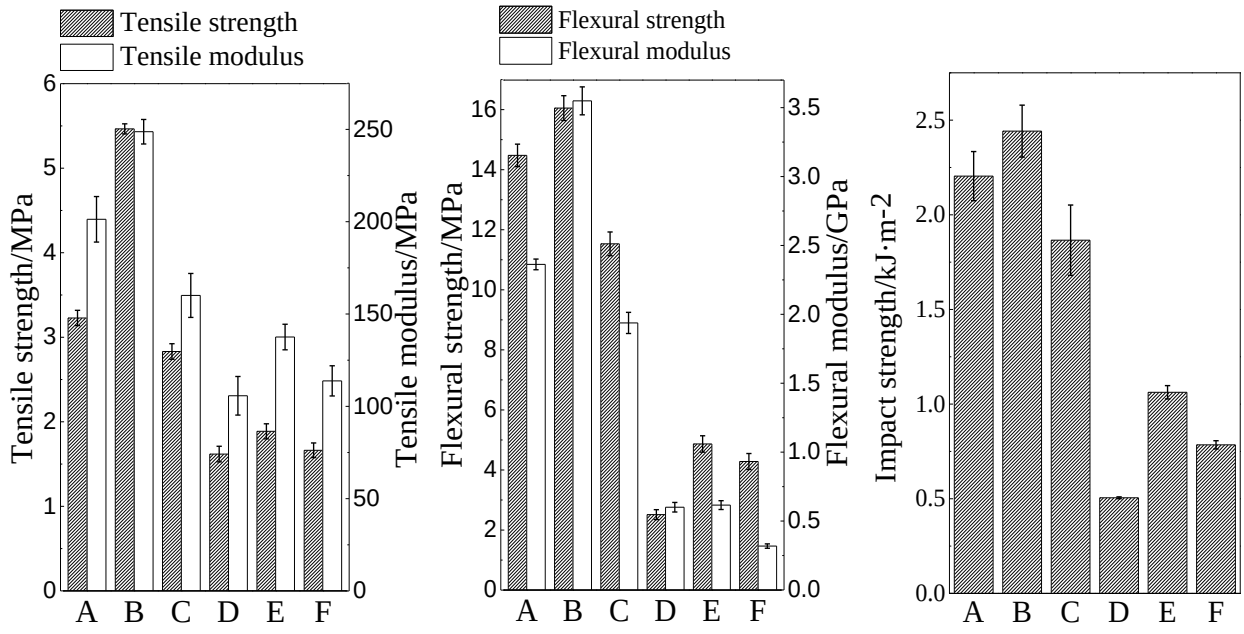
Compared with the husk fiber composites, the straw fiber composites generally had a larger equilibrium moisture content. This could be due to the fact that the straw plant fiber contained a capillary or more porous structure, which leads to capillary phenomena, accelerating the absorption of water molecules (Gong *et al*. 2017). Wheat straw/soybean protein adhesive composites had a much lower equilibrium moisture content and moisture absorption rate than the other two straw fiber composites, though the three types of straw fiber composites had similar amounts of hydrophilic groups. This was due to the strength of the hydrogen bond formed in the wheat straw/soybean protein adhesive composites, which improved its interface compatibility. There were fewer pores in the composites, and the water was not able to penetrate easily. The crystallinity of cellulose and hemicellulose in plant fiber was higher (Zhang *et al*. 2013), and there were fewer exposed hydrophilic

groups. Thus, the equilibrium moisture content was low. The rice husk/soybean protein adhesive composites had less hydrogen bonding and more interfacial pores, so the equilibrium moisture content and moisture absorption rate were higher. These results suggest that the main factors determining the moisture absorption property were the structure of the plant fibers, the interfacial compatibility of composites, and the quantity of hydrophilic groups.

Mechanical Properties of Plant Fiber/Soybean Protein Adhesive Composites

Figure 3 shows the tensile strength, tensile modulus, flexural strength, flexural modulus, and impact strength of the plant fiber/soybean protein adhesive composites. All of the mechanical properties of the wheat straw/soybean protein adhesive composites were better than in the other bio-composites. The tensile strength and tensile modulus were 465 MPa and 248.7 MPa, respectively. The flexural strength and flexural modulus were 16.05 MPa and 3.549 GPa, respectively. The impact strength was 2.442 $kJ \cdot m^{-2}$. Among the three types of husk plant fiber composites, the wheat husk/soybean protein adhesive composite had the best mechanical properties. The tensile strength, tensile modulus, flexural strength, flexural modulus, and impact strength were 1.887 MPa, 137.6 MPa, 4.868 MPa, 0.616 GPa, and 1.062 $kJ \cdot m^{-2}$, respectively. The tensile properties of the composites made from the similar plant fibers of rice and peanut crops were similar to each other. The mechanical properties of rice husk/soybean protein adhesive composite were the worst. Consequently, the mechanical properties of straw fiber composites were higher than those of husk fiber composites. The mechanical properties of the composites made of straw fiber were obviously higher than those made of the husk fiber from the same type of crops. The mechanical properties of the wheat fiber-based composites were better than those of the other two types of crop fiber/soybean protein adhesive composites. Compared with the traditional medium density fiberboard, the bio-composites have more hydrophilic groups and are more fragile, but the lower density results in higher specific strength (Huang and Pan 2014).

The mechanical properties of plant fiber/soybean protein adhesive composites mainly depend on the morphology of the plant fiber, the content of main components of plant fiber (cellulose, hemicellulose, and lignin), and interfacial adhesion between plant fiber and protein adhesive. The position and orientation of the plant fiber in the composites was random. Plant fibers were randomly connected to form a three-dimensional network structure so that a composite formed by being adhered to by the protein adhesive. However, the shape and length of the different plant fibers affected the mechanical properties of the composites. For the same type of crop, its straw fiber is usually longer than its husk fiber, and the both straws and husks have similar components of the plant fibers, so the mechanical property of the straw fiber composite was better than that of the husk fiber composite. Similarly, because of the long fiber of wheat, the mechanical properties of the wheat fiber-based composites were quite good. The hydrogen bonding between the protein and the fiber was strong, which caused better interface compatibility. Cellulose acts as a skeleton in plant fibers and was bound together by hemicellulose and lignin to form plant fibers. The wheat plant contained more cellulose and hemicellulose, and the degree of binding was higher. As such, the strength of the fiber was higher. Because of poor interfacial compatibility and low interfacial binding force, the rice husk/soybean protein adhesive composite had the weakest mechanical properties.



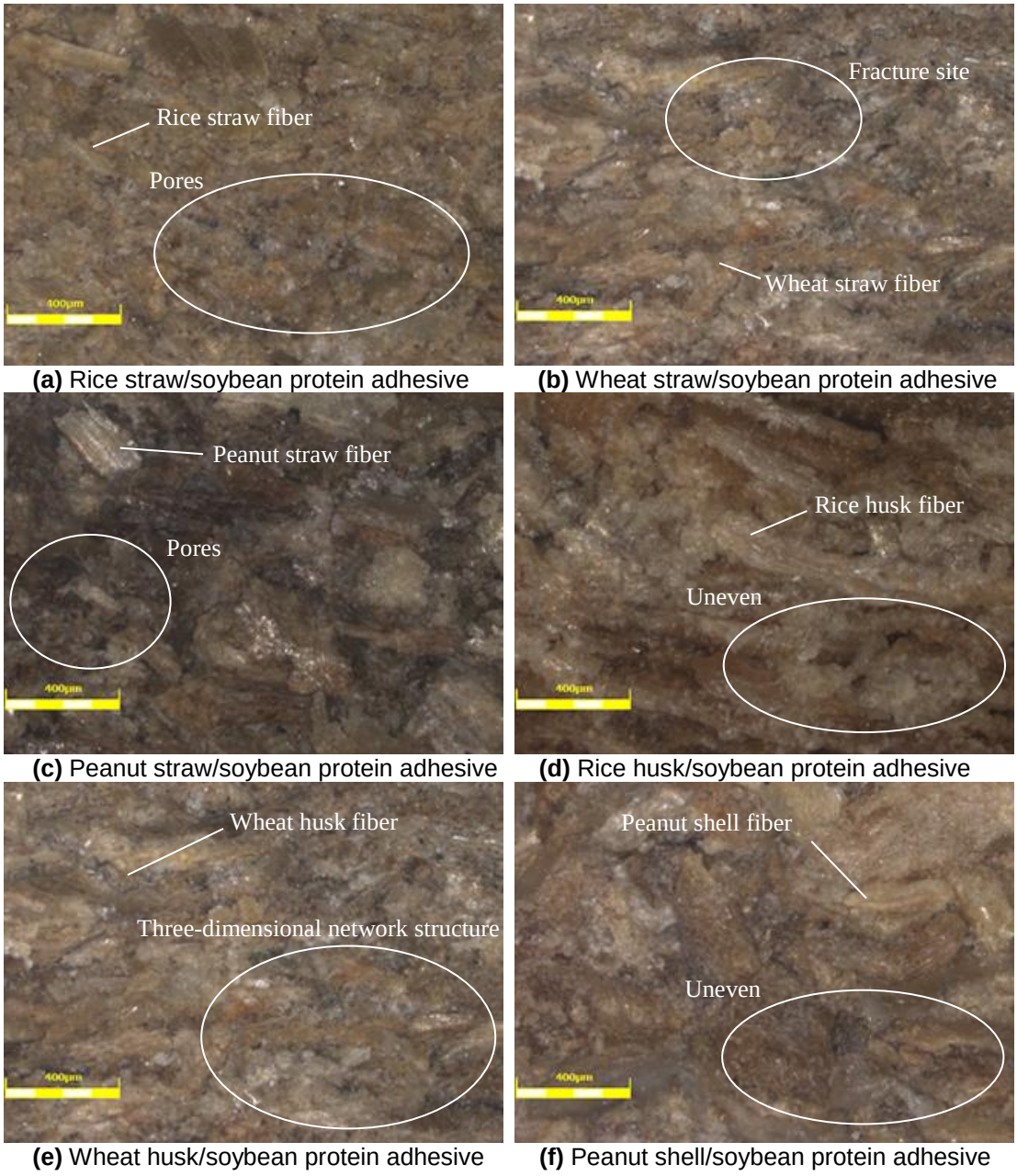
(a) Tensile Properties **(b)** Flexural Properties **(c)** Impact Properties

A-Rice straw/soybean protein adhesive; B-Wheat straw/soybean protein adhesive;
C-Peanut straw/soybean protein adhesive; D-Rice husk/soybean protein adhesive;
E-Wheat husk/soybean protein adhesive; F-Peanut shell/soybean protein adhesive

Fig. 3. Mechanical properties of plant fiber/soybean protein adhesive composites

Microstructure Surface of Plant Fiber/Soybean Protein Adhesive Composites

Figure 4 shows a microcosmic cross-section of the six types of plant fiber/soybean protein adhesive composites. The interfacial density of rice straw/soybean protein adhesive composite had an unfavorable appearance, and a lot of pores were found on the surface. However, the three-dimensional network structure formed well due to the slenderness of the fiber; the overall section was even (Fig. 4a). There were fewer pores on the surface of the wheat straw/soybean protein adhesive composites and the plant fiber was strong and long. In addition, the less exposed plant fiber at the fracture showed that the interface bonding force was larger (Fig. 4b). Due to the small interface bonding force of peanut straw/soybean protein adhesive composites, most of the fracture sites were located at the interface between peanut straw and protein adhesive and the material had pores (Fig. 4c). The surface of the rice husk/soybean protein adhesive composites had more pores. This showed that the plant fiber was relatively slender and stout. However, the interfacial binding force was small, and the fracture surface was very uneven (Fig. 4d). The wheat husk/soybean protein adhesive composites had a relatively flat cross-section, and their plant fiber was slender, sturdy, and had a good three-dimensional network structure (Fig. 4e). The interfacial compatibility of peanut shell/soybean protein adhesive composites was bad because the fiber was stout but short (Fig. 4f). In conclusion, compared with the composites prepared with the husk, the fiber in the composites prepared with the straw was longer and formed a better three-dimensional network structure.



(a) Rice straw/soybean protein adhesive **(b)** Wheat straw/soybean protein adhesive

(c) Peanut straw/soybean protein adhesive **(d)** Rice husk/soybean protein adhesive

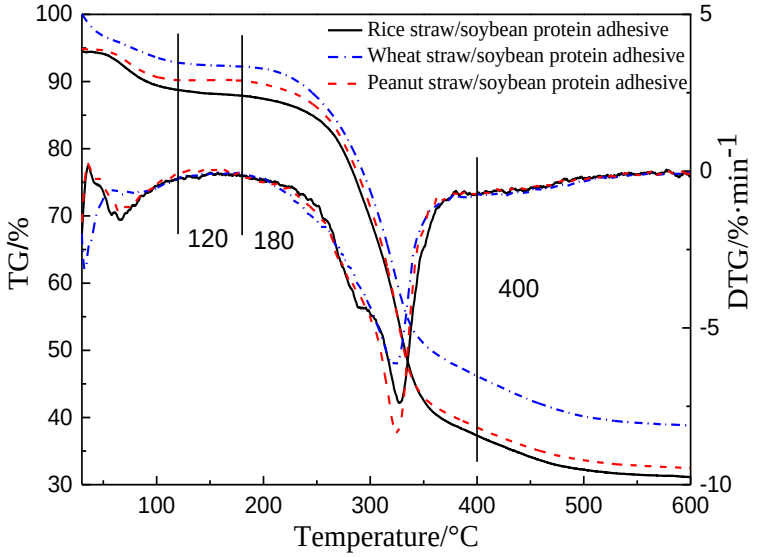
(e) Wheat husk/soybean protein adhesive **(f)** Peanut shell/soybean protein adhesive

Fig. 4. Microstructure surface of plant fiber/soybean protein adhesive composites

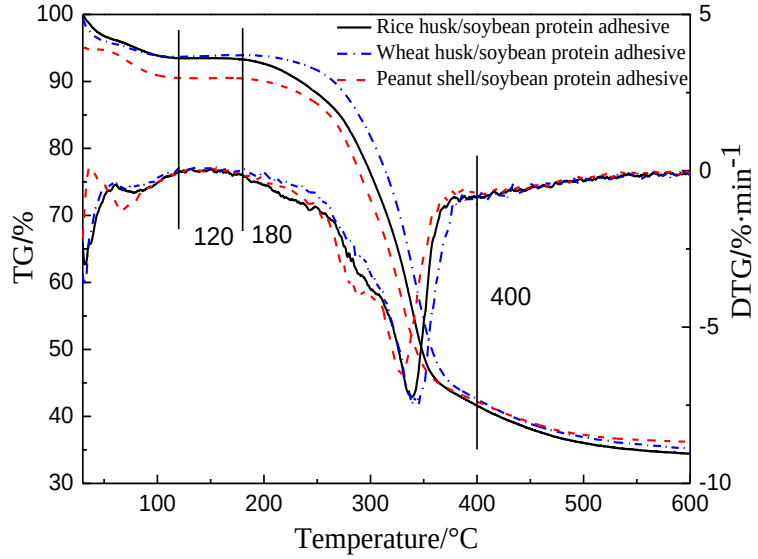
Thermal Stability of Plant Fiber/Soybean Protein Adhesive Composites

Figure 5 (parts a and b) displays the thermogravimetric analysis (TG) and derivative thermogravimetric analysis (DTG) curves for the six types of plant fiber/soybean protein adhesive composites. Table 1 shows the thermal stability of the six types of plant fiber/soybean protein adhesive composites. The TG/DTG curves of each of the six bio-composites were similar. In the range of 30 °C to 120 °C, there was a small amount of weight loss due to the large change in the TG curve of the rice husk/soybean protein adhesive composite, which had a high equilibrium moisture content. These results suggest that water evaporation resulted in the observed weight loss. Between 120 °C and 180 °C, the curved lines remain relatively unchanged with the increasing temperature. The pyrolysis stage of the six types of bio-composites was between 180 °C and 400 °C. The

pyrolysis of plant fiber main components began at unique temperatures. More specifically, cellulose began from 230 °C to 310 °C, hemicellulose began from 180 °C to 240 °C, and lignin began from 300 °C to 400 °C (Zeriouh and Belkbir 1995). The pyrolysis of protein was distributed between 300 °C and 600 °C (Shen *et al*. 2016). More precisely, the curved line indicated that the pyrolysis of the soybean protein adhesive peaked between 180 °C to 400 °C. Lastly, from 400 °C to 600 °C, the curved lines of DTG approached 0%·min^{-1}, and the residue was charred in the high temperature.



(a) Straw fiber composites



(b) Husk fiber composites

Fig. 5. TG and DTG curves of plant fiber/soybean protein adhesive composites

Table 1 indicates that the residual mass, initial decomposition temperature, and final thermal decomposition temperature of each of the six bio-composites were relatively similar, yet there were a few differences that may have been caused by the different proportions of main components in plant fiber. More specifically, as the pyrolysis of hemicellulose started at a low temperature, so did the pyrolysis of the composites that

contained more hemicelluloses. After pyrolysis, the residual mass of cellulose was lower than that of the other components, so the composites with more cellulose were lighter than other composites after pyrolysis. Finally, comprehensively comparing the initial pyrolysis temperature of six bio-composites, the thermal stability of the rice husk/soybean protein adhesive composite was the best of all, and the peanut straw/soybean protein adhesive composite was the worst.

Table 1. Thermal Stability of Plant Fiber/Soybean Protein Adhesive Composites

Plant Fiber	Temperature (°C)		Residual Mass (%)
	Onset	Termination	
Rice Straw	278.3	350.6	31.12
Wheat Straw	281.9	344.6	32.49
Peanut Straw	271.8	345.7	38.81
Rice Husk	283.4	360.8	34.45
Wheat Husk	274	354.3	38.19
Peanut Shell	282.6	367.5	35.22

CONCLUSIONS

1. The basic functional groups of the six types of plant fiber/soybean protein adhesive composites (rice straw, wheat straw, peanut straw, rice husk, wheat husk, and peanut shell) were quite similar. The peanut straw/soybean protein adhesive composite contained more hydrophilic groups, such as $-NH_2$, -NH, -OH, and C=O. The wheat fiber-based composites possessed more hydrogen bonds, leading to the best binding interface compatibility.
2. The properties of the wheat fiber-based composites were better than those of the others. Wheat straw/soybean protein adhesive composite had the best mechanical properties with a tensile strength, flexural strength, and impact strength of 69.3%, 10.8%, and 10.78% higher, respectively, than that of the rice straw/soybean protein adhesive composite. The properties of tensile strength, flexural strength, and impact strength of the wheat straw/soybean protein adhesive composite were 337.7%, 638.6%, and 483.4%, compared to those of the rice husk/soybean protein adhesive composite, respectively.
3. The equilibrium moisture content of the peanut shell/soybean protein adhesive composite was the lowest (8.70%). The rice husk/soybean protein adhesive composite had the highest equilibrium moisture content (14.23%) and the best thermal stability. Its initial temperature of pyrolysis was 283.4 °C, and the residual mass was 34.45%.

ACKNOWLEDGMENTS

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