

## STRAW BASED PARTICLEBOARD BONDED WITH COMPOSITE ADHESIVES

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Environmentally friendly particleboard was prepared with wheat straw, an inexpensive material. The particleboard was produced by a mixing process, using a composite adhesive comprised of urea-formaldehyde (UF) adhesives and EPU. The performance of the board was evaluated by measuring internal bonding strength (IB), thickness swelling, modulus of rupture (MOR), modulus of elasticity (MOE), and formaldehyde emission. The experimental results showed that maximum of dry and wet internal bonding strength, modulus of rupture, modulus of elasticity were 0.45MPa, 0.18MPa, 31.80MPa, and 5043MPa, respectively. The thickness swelling (TS2h) and thickness swelling (TS24h) were 3.9% and 10.7%, respectively. The composite adhesives and particleboards were measured by differential scanning calorimetry (DSC), dynamic mechanical analyzer (DMA), X-ray photoelectron spectroscopy (XPS) and scanning electron microscope (SEM) measurements. The results indicated that the composite adhesive of UF/EPU could contribute to excellent mechanical, thermal, and water-resistant properties of the wheat straw particleboards.

*Keywords:* Particleboards; Wheat straw; Composite adhesive; DSC; XPS; SEM

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### INTRODUCTION

The demand for glued-wood composite products such as particleboard, medium density fiberboard, and plywood has increased dramatically throughout the world in recent years (Sellers 2001; Youngquist 1999; Zheng et al. 2007). At present, increasing demand of particleboards from forest resources in housing construction and furniture manufacturing sectors have led to a shortage of wood supply (Zheng et al. 2007; Singha. 2008). On the other hand, large quantities of agricultural residues, including harvesting residues, barks, lumber and furniture plant residues, residues of pulp plants, and recycled paper are available for woodware production in place of wood. Wheat straw (WS) is a renewable agricultural by-product. In China, more than  $1.2 \times 10^{12}$  tons of WS is produced annually, with a large quantity of the WS being incinerated or discarded in the environment. Although the chemical composition of WS is similar to that of wood, comprising cellulose structures in an amorphous matrix of hemicelluloses and lignin, its structure is looser and its strength is lower; it also contains less lignocellulosic materials and more ash and extractives with lower molecular weights than wood. Furthermore, silica and waxy substances on the exterior surfaces of WS tend to interfere with its bonding to other materials when using conventional adhesives such as urea formaldehyde (UF) or phenol formaldehyde (PF) (Yao et al. 2003; Mo et al. 2003; Liu et al. 2002;

Wang et al. 2009). The advantages of straw particleboards include built-in insulation, sound suppression characteristics, and low cost (Boquillon 2005). In addition, several types of adhesive resins have been used to make straw particleboard. Among these adhesives, urea-formaldehyde is the most economical and useful adhesive because of its low cost and easy production (Guru et al. 2005). But there are also some disadvantages, such as poor water resistance, especially intolerance boiling water, and less durability. These problems are compounded by formaldehyde emission, resulting in harm to the environment and the human body. On the other hand, isocyanates are highly reactive to amines and urethane, which are sensitive to nucleophilic reagents under both acidic and basic mediated condition (Blencowe et al. 2006). The advantages of isocyanate adhesives include shorter time and lower temperature for hot pressing, and less dry materials cost, which could improve productivity of wheat straw-based composites (Gallbraith 1986). The cost of isocyanate, which may be used as an adhesive for wood and wheat straw, is substantially greater than other wood adhesives such as urea formaldehyde, phenol-formaldehyde, and melamine-formaldehyde, based on weight (Han et al. 1998). Making advantageous use of isocyanate and urea-formaldehyde resins to achieve low-cost production of high-performance, environmentally friendly particleboard is an innovative point of this study (Kang 2001; Mo et al. 2001). And the mechanical properties of wheat straw particleboards bonded by composite adhesive with UF and EPU (emulsifiable pMDI) can be improved greatly without removing wax and ash from the wheat straw surface through bleaching with oxidizing agents and alkali.

The aim of this study was to investigate the effect of mechanical properties and water resistance of particleboard made from wheat straw, depending on the ratio of composite adhesive. All chaff, nodes, leaves, and broken kernels do not need to be removed for preparing particleboards with composite adhesive. Furthermore, the composite adhesives and the boards were investigated by using differential scanning calorimetry (DSC) and dynamic mechanical analyzer (DMA), respectively. The study of morphology and chemical state requires a reliable method to analyze wheat straw particleboards. X-ray photoelectron spectroscopy (XPS) and SEM provide a chemical analysis and morphology of the glue line substrate in the boards. As described in this paper, a variety of characterization procedures for wheat straw particleboard can provide a strong theoretical basis for production practices.

## **EXPERIMENTAL**

### **Materials**

Wheat straw raw material was obtained from a local farm. The stems were first cut into 100 to 150 mm lengths by a drum chipper and further refined into 10 mm using ring flakers. All of the 10 mm straw was further oven-dried at 80 °C to about 4% moisture content and stored in plastic bags, which were kept in the wood based panel laboratory.

Urea-formaldehyde (UF) is the dominant adhesives for making the particleboard. The UF resin was synthesized by our laboratory; one of the mole ratios was 1.3 of the E<sub>2</sub>

type. The resin solid content and urea/formaldehyde molar ratio of the UF resin were 60-62% and 1.3, respectively.

Polymeric methane diphenyl diisocyanate (pMDI) was supplied by Bayer Polymer Ltd. Co. (branch company, China); it contains 32.9% of NCO with an average functionality of 2.7. Ammonium chloride, used as a hardener, was purchased from TianJin BoDa Chemical Co. Other agents such as formic acid, sodium hydroxide, urea, and formaldehyde and sodium bisulphate were purchased from TianJian Huabei Co. The EPU was prepared by the reaction of the pMDI with the NP40.

### Straw Particleboards Manufacturing

The UF resin was synthesized with mole ratio (U/F) of 1.3. The solids content in the adhesive solutions was 62%. The composite adhesive involved adding EPU with different ratios into the UF resin, achieving highly stable mixtures. The composite adhesive was sprayed in the form of particles in a blender at 10% resin content based on the oven-dried weight of particles. Particleboards of 400×400×12 mm were manufactured at a target density of 750±10 kg/m<sup>3</sup> with an adhesive content of 10%, 6 min of hot-press time, and 170 °C of hot-press temperature.

### Preparation of composite adhesive

#### *Emulsifiable polyisocyanate adhesive preparation*

Depending on the desired molar ratio of –NCO and –OH, a certain quality of pMDI and polyether polyols were placed into a three-necked bottle and churned up at a rotational speed of 500r/s. Meanwhile nitrogen gas was injected into the reactor for 30 minutes, preparing the system to react for 5 hours in an oil bath at the temperature of 70±2°C. When the reaction was finished, the reaction system was allowed to cool to room temperature (40±3 °C) and the products of reaction were taken out.

#### *Preparation of the composite adhesive*

After preparing the emulsifiable polyisocyanate and urea formaldehyde mixture according to a certain ratio, a small quantity of accessory ingredient was added and the mixture was blended rapidly for 10 to 15 minutes until a light yellow emulsion adhesive was obtained.

### Straw Particleboards Evaluation

Mechanical properties, including modulus of rupture (MOR), modulus of elasticity (MOE), internal bond strength (IB), and water resistance are very important specifications for particleboards. They were measures of finished particleboard. The straw particleboards were cut into test specimens according to national standard of GB/T 9846. The dimensions of the sample were 5.0×5.0cm square shape for internal bond measurement and for boiling water internal bond measurements. The dimensions were 5.0×17.0 cm for three-point flex measurement of MOR and MOE, respectively. All specimens were equilibrated at a temperature of 20 °C and 65% relative humidity (RH) before testing. Mechanical properties were determined by using a testing machine and following ASTM standard method GB/T 9846.1, 9846.8 (2004). Water absorption and thickness swell were measured according to the ASTM standard methods. The 50×50

mm pieces of the particleboard were soaked in water at room temperature for 2h. The thickness and weight were measured immediately after soaking. The dimensional size and weight measured before and after soaking were obtained. Then the average results were calculated from four specimens.

### **Differential Scanning Calorimetry Measurements (DSC)**

To examine the curing behavior of each UF/EPU resin, differential scanning calorimetry (DSC) (Netzsch 204 Instruments, Germany) and a high-pressure pan were used. Each composite adhesive used for DSC measurement were the same samples as those used to assess gel time, and they were scanned at a heating rate of 5 °C/min, with a temperature range of 30 to 200 °C.

### **Dynamic Mechanical Analysis Measurements (DMA)**

The samples of the straw particleboard can be studied by the DMA technique (No and Kim 2005). A three-point bending mold was employed for the test. The dimensions of samples were 40×8×2.5 mm. A fixed displacement mode with 60 mm amplitude and 10 Hz oscillation frequency was used. The temperature program started with an equilibration waiting period at room temperature, followed by increasing temperature at the same rate of 5 °C/min to 290 °C. DMA instrumental analysis was used to obtain the rigidity (storage modulus,  $G'$ ) and tan delta ( $\delta$ ,  $G''/G'$ ) curves.

### **X-ray Photoelectron Spectroscopy (XPS) and Scanning Electron Microscope (SEM) Measurements**

XPS measurements were performed at room temperature with monochromatic AlK $\alpha$  radiation (1486.6eV) using a K-Alpha X-ray photoelectron spectrometer (supplied by Thermo Fisher Scientific Co., Ltd). The X-ray beam was a 100-W, 200mm-diameter beam raster over a 2mm by 0.4 mm area on the sample. High-energy photoemission spectra were collected using pass energy of 50eV and resolution of 0.1eV. For the Ag3d5/2 line, these conditions produced an FWHM of 0.80eV. A Quanta 200 scanning electron microscopy (supplied by FEI Co., Ltd) was used for the investigation of the morphology of the wheat straw particleboards.

## **RESULTS AND DISCUSSION**

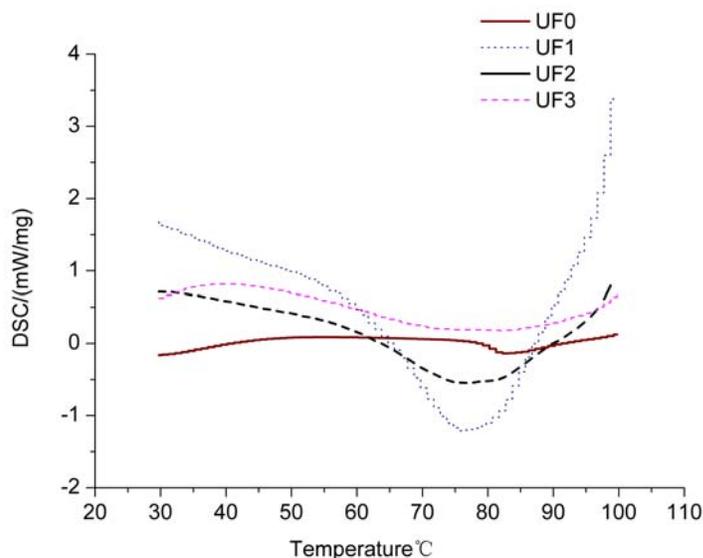
### **DSC Characteristics of Composite Adhesive with UF Resin Modified by EPU**

Differential Scanning Calorimetry (DSC) analysis has been broadly applied for UF resin curing research. Because of the fact that the urea-formaldehyde resin was added at different ratios of EPU, its curing conditions would not necessarily be constant. For evaluation of the composite effect of urea-formaldehyde resin and the EPU in bonding, DSC can accurately determine whether the modified resin can fully fill the role of an adhesive for this type of product.

With other conditions fixed, to change the add EPU ratio, the composite adhesive were characterized by DSC, as shown in Fig. 1 and Table.1

**Table 1.** Cure Temperature of UF Resin Modified by EPU

Label	Onset/ $^{\circ}\text{C}$	Peak/ $^{\circ}\text{C}$	End/ $^{\circ}\text{C}$	Area/J/g
UF0	78.6	82.9	98.5	-29.2825
UF1	61.3	77.2	86.8	-61.435
UF2	57.8	76.5	85.2	-127.717
UF3	51.7	76.7	85.0	-58.098

**Fig. 1.** DSC curves of UF resin modified by EPU

From the chart it is clear that the cured properties of composite adhesives exhibited significant differences. As the addition of EPU gradually increased, the lower initial temperature of the DSC curve, as well as the curing, began at a lower temperature. From the point of view of the peak temperature and the end of the temperature curve, its highest conversion rate of curing and temperature of curing began with the same trend. This implies that the urea-formaldehyde resin modified by EPU can promote urea-formaldehyde resin curing speed and facilitate complete curing. Judging from the area of the UF2 maximum heat release, heat was released due to the EPU and urea-formaldehyde resin curing or water reaction. And with the addition of EPU the amount of heat released gradually increased. When the UF/EPU ratio was 70/30, there was no exothermic peak in the DSC curve; this was attributed to the fact that isocyanate addition increased. These considerations may induce faster reaction with the water than urea-formaldehyde resin, possibly covering up the UF resin curing characteristics peak, thus giving rise to a wide exothermic peak. To sum up, the use of DSC method can provide a very good characterization of urea-formaldehyde resin curing properties, and it is clear from the spectra that EPU mixed with the urea-formaldehyde resin can promote the curing reaction.

### Properties of Wheat Strawboards

The properties of UF/EPU mixtures bonded particleboards comprising different ratios of UF/EPU are presented in Table 2. Glue-mixes composed of UF/EPU weight ratios of 70/30, 80/20, 90/10, and 100/0 (UF control) were prepared. To all the glue mixes were added 5% ammonium chloride aqueous solutions on UF resin. The results in Table 2 indicate that the performance of strawboards was significantly improved by the increasing addition of EPU. This is because both reactions of isocyanate with the water and hydroxyl groups in the straw can generate crosslinked polyureas or polyurethanes, leading to strong mechanical bonding. Straw particleboards bonded using UF resin exhibited poorer mechanical properties than those with added EPU. With increasing addition of isocyanate, straw boards of MOE, MOR, and IB did not follow the same trend, but the results met or exceeded national standards, and free formaldehyde also stayed below E0-type limits. Because of the increasing addition of isocyanate will increase the cost, a ratio of 80/20 composite adhesive was selected.

As shown in Table 3, the ratio of urea-formaldehyde resin to EPU was fixed at 80/20, to change the adhesive amount of composite (9%, 10%, 11%, 12%) based on dried straw. With the increase amount of composite adhesive, straw board MOE, MOR, and IB gradually improved, and straw board dimensional stability, which depended on the precise nature of the increased use of composite adhesive. However, straw board of formaldehyde emission was also increased with the amount of composite adhesive increase. From the results from Tables 2 it is evident that by the use of common urea-formaldehyde resin ( $F/U = 1.3$ ) and isocyanate composite adhesive, when the UF / EPU ratio is 80/20, an amount adhesive of 11% achieved excellent performance in meeting the E0 straw board specifications.

**Table 2.** Properties of Particleboard with Different Ratio of Composite Adhesive

UF/ EPU	density (g/cm <sup>3</sup> )	MOE (MPa)	MOR (MPa)	IB <sub>D</sub> (MPa)	IB <sub>W</sub> (MPa)	TS (2h)(%)	TS (24h)(%)	FF mg/100g
70/30	0.70	4303	26.95	0.36	0.11	9.0	24.8	0.24
80/20	0.74	5010	35.04	0.72	0.16	4.7	13.7	0.89
90/10	0.78	4783	28.56	0.55	0.14	5.6	13.9	0.24
100/0	0.72	3164	13.6	0.12	0.08	15.0	35.7	1.12

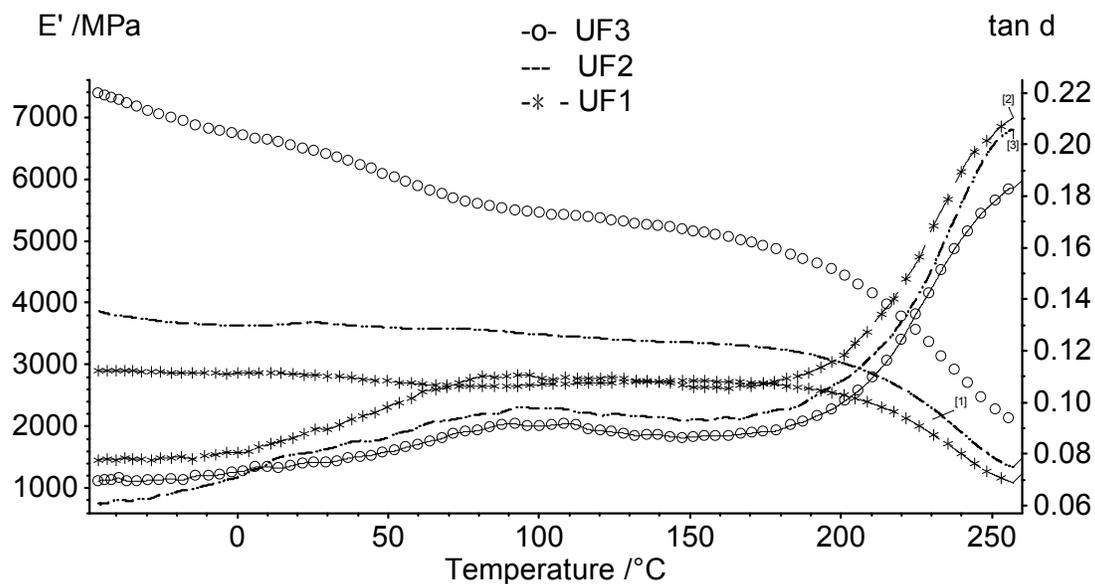
**Table 3.** Properties of Particleboard with Different Amount of Adhesive at the Ratio of 80/20

Amount of adhesive (%)	density (g/cm <sup>3</sup> )	MOE (MPa)	MOR (MPa)	IBD (MPa)	IBW (MPa)	THS (2h)(%)	THS (24h)(%)	FF mg/100g
9	0.74	4944	35.33	0.51	0.10	6.4	16.6	0.09
10	0.74	5010	35.04	0.72	0.16	4.7	13.7	0.89
11	0.76	5043	31.80	0.45	0.18	3.9	10.7	0.50
12	0.76	4829	32.00	0.56	0.05	3.8	10.0	1.49

### DMA Measurement

DMA tests were used to investigate the effect of different composite adhesive proportions on the thermomechanical properties of the prepared straw particleboards, as shown in Fig. 2. The DMA results of UF/EPU composite were conducted at 70/30, 80/20, and 90/10 ratios. From Fig. 2 it is clear that the preparation of straw particleboard with the ratio of 90/100 had a lower storage modulus. As the addition of isocyanate increased, its storage modulus increased markedly. It is noticeable that the UF/EPU 70/30 mix reached a maximum value of the modulus of 7000MPa. The reason for the observed enhancement in curing with the isocyanate has been ascribed to the higher reactivity of isocyanate than urea, thus achieving a stronger adhesion to straw. Therefore, we can conclude that isocyanate can accelerate the curing reaction of urea-formaldehyde resin, especially enhancing the strength of straw particleboard bonding. However, the tan-delta curves of all straw particleboards decreased slightly as the composite ratio increased from 90/10 to 70/30, and the loss modulus for 90/10s particleboard was distinctly greater than that of the 80/20 and 70/30 particleboards.

The results in Figs. 2 and 3 could not confirm that a mutual reaction of UF with EPU indeed occurs to a greater or smaller extent, but leads to a thermally stable structure formed by a main product that is different than what were previously small isocyanate molecules. Overall, the optimized system with a composite ratio of 70/30 exhibited a high storage modulus. However, the ratio of 80/20 complex conditions would be able to meet the requirements. But from cost considerations, we can choose the proportion of 80/20 preparation of wheat straw particleboard.



**Figure 2.** Storage modulus and tan delta of straw particleboards with different proportions of UF/EPU

### X-ray Photoelectron Spectroscopy (XPS) Analysis

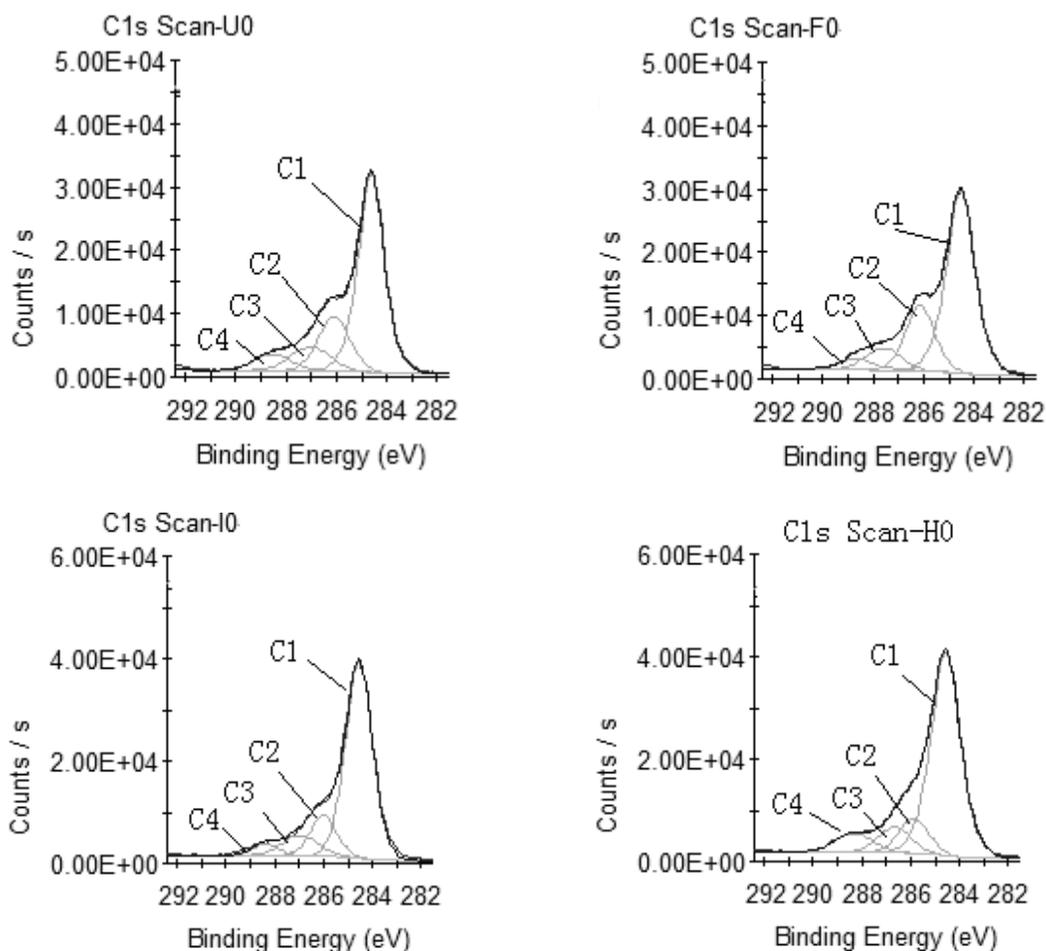
X-ray photoelectron spectroscopy (XPS) analysis was used to characterize the chemical changes of the wheat straw particleboards. In the high resolution spectra of C1s, as shown in Fig. 4 and Table 4, it is evident that the C1s spectra of the samples consisted of four well-resolved peaks. The curve-fitting simulation was carried out after subtracting the baseline correction.

The peaks of carbon atoms at 285.0eV, 286.2-286.4eV, 287.2-287.9eV, and 289.3-290.7eV corresponded to the carbon atoms that can be described in abbreviated form as C-C or C-H, the carbon atoms singly bonding to oxygen (ether group C-C-O), the carbon atoms double-bonded to oxygen and nitrogen in the urethane linkage (-O-C=O), and the carbon atoms double-bonded to oxygen and nitrogen (-N-C=O) in the imides group, respectively. The peak-fitted C1s spectra correspond to wheat straw particleboards with the composite ratios UF/EPU of 100/0, 90/10, 80/20, and 70/30, respectively. The peak fitting of the C1s spectra from the data set is illustrated in Fig 3. The carbon chemical state demonstrated a slight decrease of the ether group with an increase of amount of EPU, suggesting a modification of the glue line of wheat straw particleboards.

**Table 4.** Binding Energy (eV) and Relative Intensity (%) of the High-Resolution C1s Peak Fitting Signals for the Reference UF and EPU

Sample UF/EPU	C1(285.0eV) (C=C/C-H)	C2(286.3-286.4eV) C-O-C	C3(287.3-287.4eV) O-C-N- -N-C-N-	C4(288.8-289.1eV) -N-C=O O-C=O
100/0(U)	63.29	19.62	10.76	6.33
90/10(F)	63.29	22.15	8.86	5.70
80/20(I)	70.46	14.49	9.42	5.62
70/30(H)	71.43	11.43	9.28	7.86

In the present study the variation in the ratio of UF/EPU of composite adhesive was attained by changing the content of EPU. An increase in the ratio of UF/EPU resulted in an increase in the content of carbon to carbon single or carbon to hydrogen bonds (C1-C or C1-H). On the other hand, it appears that the sum of the content of the contributions of carbon atoms involved in single bonds with oxygen increased 3% first and subsequently decreased gradually by 6.19%. This fact may be attributed to the large interchange benzene ring association and hence an increase in C=C/C-H bond strength was more due to a higher UF/EPU ratio. In addition, the benzene ring can improve the cohesion of the straw layer with glue line. Then, the main contribution to C3 would essentially be due to the -N-C3-N- bond (i.e. methylene bridges) instead of -N-C3-O- (methylene ether bridge) contributions. But the percent content of C3 does not represent a major change. On the other hand, with increasing EPU content, the urethane (-N-C=O) functional group increased, which led to increased cohesion and markedly improved strength of wheat straw particleboards. Thus, it can be concluded that the main impact of the ratio of UF/PEU is due not to physical but rather to chemical medications.

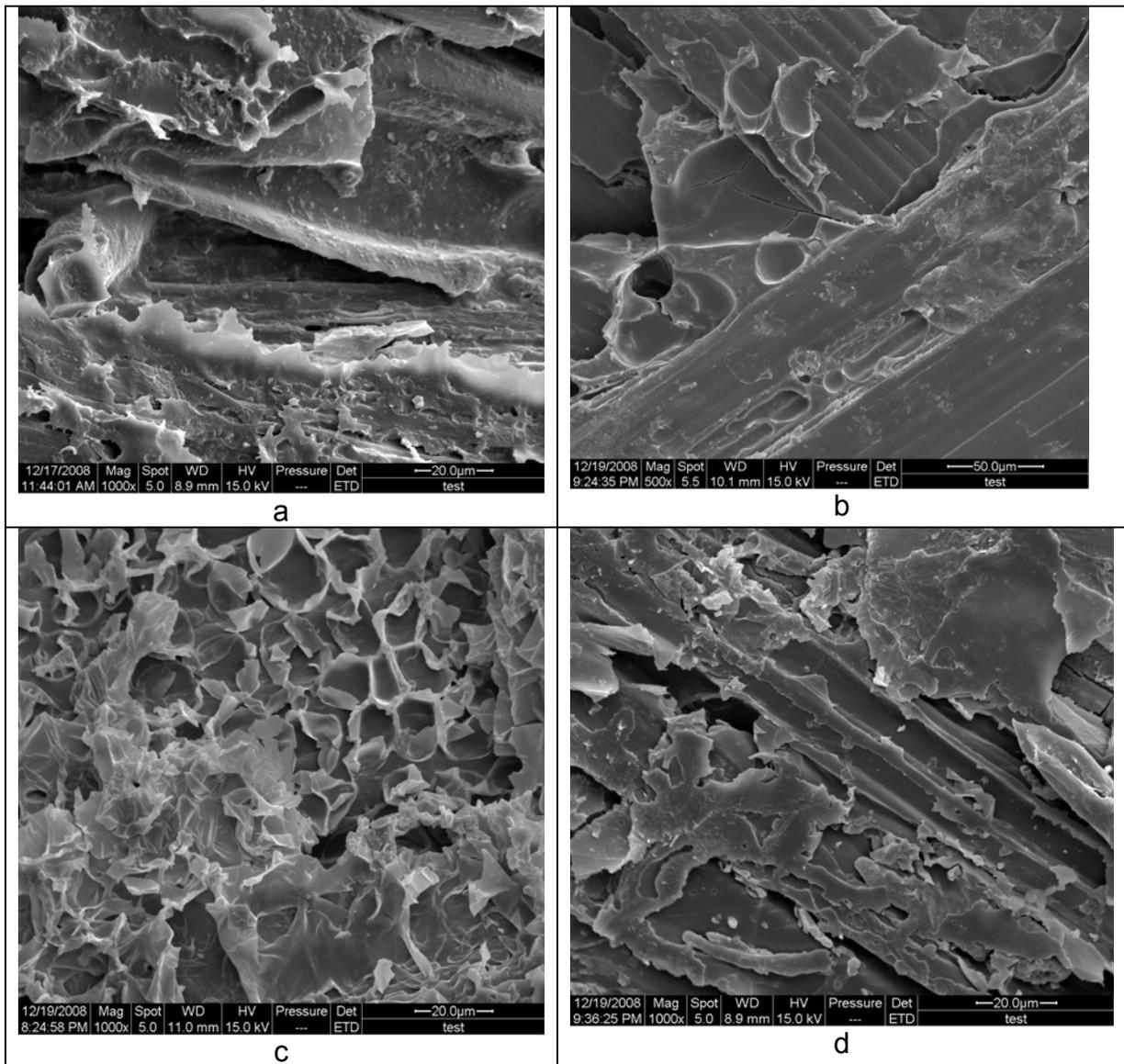


**Fig. 3.** Carbon 1s peak-fitting signal of the particleboards with (UF/EPU)

In fact, EPU can enhance the reaction conditions of the wheat surfaces by high reactivity to the wheat surface. Therefore, physical and chemical changes of wheat surfaces interfere with adhesion strength of the adhesive consequently. From the results of XPS analysis, it was clear that the urethane groups ( $-\text{NH}-\text{C}=\text{O}$ ) attached onto the surface of the wheat straw particleboards, and these groups were useful for improved the strength of polymers to make wheat straw particleboards. As a result, the composite adhesive of UF/EPU could contribute the excellent mechanical, thermal, and water-resistant properties to the wheat straw particleboards.

### Observation of Wheat Straw Particleboards by Scanning Electron Microscopy (SEM)

SEM pictures of wheat straw particleboards with different proportions of UF/EPU are shown in Fig 4.



**Fig. 4.** SEM micrographs of straw particleboards with different proportions of UF/EPU: (a) proportion of 100/0; (b) proportion of 90/10; (c) proportion of 80/20; (d) proportion of 70/30

From the SEM pictures it is apparent that the morphology of wheat straw particleboards with urea formaldehyde resin was modified by EPU. Particleboards made from different ratios (100/0, 90/10, 80/20, and 70/30) of composite adhesive were investigated. The wheat straw particleboard specimens treated by sputter-coating with gold provided good analysis of scanning electron microscopy, as shown in Figs. 4(a) through 4(d). The wheat straw particleboard that was prepared with a 100/0 ratio of UF/EPU (unmodified UF) clearly shows the interfacial structure between wheat straw and urea formaldehyde resin. A large number of solidified particles of cured UF resin could be exfoliated and well dispersed into to the straw cell wall, but the distribution was non-uniform. There were relatively few glue spots of particleboard with UF resin. With a mixture ratio of 90/10 the amount of glue after curing the adhesive layer was slightly less

than in the case of the 100/0 ratio, and the distribution was very good. Cross-sectional observations from the sample prepared with an 80/20 ratio showed only a few fissures and a more even distribution into the straw catheter holes and thin-walled elements, playing a role similar to that of nails. In addition, it can be seen in Fig. 5(d) with the ratio of 70/30 UF/EPU; there was a great deal of glue left in the fractured surfaces of boards. The reason for this behavior is that the content of the EPU adhesive increased, which led to a rapid increase of the viscosity of composite adhesive. Furthermore, the highly reactive EPU contains the –NCO functional group, which causes uneven distribution in the glue line of boards. As a result, the SEM results revealed that UF/EPU composites using 80/20 had good particle dispersion and particle-matrix adhesion.

## CONCLUSIONS

1. In conclusion, the results of this study indicate that wheat straw is a suitable raw material for making particleboards with special composite adhesive. The results of differential scanning calorimetry measurements indicated that emulsifiable pMDI (EPU) could accelerate the cure rate of the UF resin effectively, achieving better thermo-stability of the boards. All the particleboards made in this study exceeded the minimum mechanical property requirement of national standards.
2. Regarding the binding ability and cost, better performance of wheat straw particleboard bonded with composite adhesive can be obtained at a UF/EPU ratio of 80/20, a hot-press temperature of 170 °C, a density of 0.75g/cm<sup>3</sup>, a hot press time of 6 min, and adhesive amounts of 10% based on the dry amounts of wheat straw.
3. From the results of DMA, XPS, and SEM analysis, it was clear that the urethane groups (-NH-C=O) were attached onto the surface of the wheat straw particleboards, and these groups were useful for improving the strength and thermal stability of wheat straw particleboards. Also, the composite adhesive of UF/EPU could contribute the excellent mechanical, thermal, and water-resistant properties to the wheat straw particleboards.
4. All in all, 80/20 of UF/EPU ratio particleboard prepared with the best overall performance. Thus the preparation and characterization of wheat straw particleboard, as described in this paper, provides a theoretical basis for the application of wheat straw.

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