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## **COVER SHEET**

**Title: Compare between Flax Shive and Extracted Flax Shive Reinforced PP Composites**

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## **ABSTRACT**

Flax shive (FS), the woody part composing of over 70 wt% of the flax straw, is a waste product left after removing the fibers from flax straw. Although the FS is supposed to have lower strength than that of flax fiber, however, its low cost and greater availability could be advantages for the production of low cost composites. Recently, FS has been extracted using environmentally benign processes and thus hemicelluloses and lignin were effectively removed from FS. In this work, FS and extracted flax shive (EFS) were characterized and further used as reinforcing materials for polypropylene (PP) composite. The effect of cellulose content on the composite properties, such as thermal stability and mechanical properties was examined. Compatibilizer was added to get the best compatibility between the FS and the PP matrix. It was shown that EFS present better thermal stability than FS because of its lower flammable noncellulose ratio. FS appears to behave as filler for composite even with compatibilizer. However, with the presence of coupling agent E43, EFS could be upgraded to be a reinforcing material for PP composites. With 30 % EFS, the tensile strength and the modulus of composite can increase almost 8% and 100% over than PP, respectively.

## **INTRODUCTION**

Flax is remarkable with its high specific strength and modulus among the different kinds of natural fibers as reinforcing material for polymer composites, because of the good mechanical properties and environmental friendly performance [1]. However, flax shive (FS) is always considered to be waste product left after the cellulosic flax fiber removal from flax straw. It is the woody part of straw. There is 2.5 tons of shives

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shives produced for every one ton of fiber production [2]. To make use of flax shives has been an attractive research subject for economical and ecological reason. There are many studies on fractionation of flax shive into constituent biopolymers and their further conversion into biofuels and other bioproducts [3-5].

So far, there has been no study on flax shive as reinforcing materials for composites. The possible reasons are its low mechanical properties resulted by its high non-cellulose portions, and also the shortcomings as natural fibers, such as hydrophilic nature, high moisture absorption and low thermal resistance, in the light that the properties of composite material are mainly decided by the property of reinforcing material and the adhesion between the reinforcement and the surrounding matrix. Flax fiber possesses ~73% of cellulose, ~13% of hemicelluloses and ~5% of lignin [6]. While Flax shives possess high lignin (~24%), hemicellulose (~24%) and less cellulose (~53%) contents. Recently, FS has been extracted using environmentally benign processes such as pressurized low polarity water and pressurized aqueous ethanol. After these processes, hemicelluloses and lignin were effectively removed from FS. The extracted flax shive (EFS) could be consisted of ~29.1% lignin, ~60.0% cellulose, and ~2.3% of hemicelluloses [4, 5]. The cellulose content increase ~7% and most of hemicelluloses was removed. Therefore, its applicability to be reinforcing material is more promising.

On the other hand, flax shive also present the hydrophilic nature of flax, which could lead to a bad adhesion between flax shive and matrix. To enhance interfacial adhesion of flax / polymer composites, modifications on fiber and matrix with chemical treatments or by adding coupling agent have been achieved. The aim is to activate hydroxyl groups or introduce new moieties that could effectively interlock with the matrix. Maleic anhydride-polypropylene (MAPP) copolymer is one kind of effective compatibilizers for nature fiber / PP composites [7-9].

In this work, flax shive and extracted flax shive were characterized and further used as reinforcing materials for polypropylene (PP) composite. The effect of cellulose and hemicelluloses concentration on the composite properties, such as thermal stability and mechanical properties was examined. Compatibilizer was added to get the best formulation of flax shive / PP composite.

## **2. EXPERIMENTAL**

### **2.1. Materials**

Flax shive and extracted flax shive were obtained from Pacific Agri-Food Research Center, Agriculture and Agri-Food Canada. Homopolymer Pro-fax PDC 1274 (PP) ( $\bar{M}_w = 250 \text{ Kg / mol}$ ) obtained from Basell was used as Polypropylene matrix. Epolene 43 (E43) ( $\bar{M}_n = 9,100$ ; ~ 4.81 wt% of MA) obtained from Eastman Chemicals was used as compatibilizer in this study.

### **2.2. Compounding with PP**

FS and EFS were dried in a vacuum oven at 100°C for 6h before their compounding with PP / E43 according to the formulation shown in Table I in a Plastimeter of the C.W. Brabender Instrument Inc.. All the components were mixed totally

for 10 min at 60 rpm at 190°C. The obtained composites were kept in a vacuum oven at 100°C for 24h before moulding. Compressing of the composites was carried out in a Carver compressing moulding machine.

TABLE 1. FORMULATION OF FLAX SHIVE / PP COMPOSITES

SAMPLE	FS (G)	EFS (g)	E43 (G)	PP1274 (g)
PP				50
PPFS30	15			35
PPEFS30		15		35
PPFS30E	15		1	34
PPEFS30E		15	1	34

### 2.3. DSC Study

DSC-7 Perkin-Elmer calorimeter was applied to study the melting and crystallization behaviour of PP and flax shive / PP composites at a heating and cooling rate of 10°C / min during all the process. The sample was first heated to 200 °C and kept for 5 min. And then the sample was cooled down to room temperature and performed the second heat scan thereafter. The crystallinity of the PP matrix in the composites was determined using the following equation:

$$X_c = \Delta H_m / (f_p \times \Delta H_f^0) \times 100 \% \quad (1)$$

where  $\Delta H_m$  (J/g) is the enthalpy of melting of composites,  $f_p$  is the PP weight fraction in composites, and  $\Delta H_f^0$  is the enthalpy of melting of pure crystalline PP (207.1 J/g [10]).

### 2.4. X-ray diffraction

Bruker Discover 8 diffractometer operating at 40 KV, 40 mA with Cu Ka radiation in reflection mode using a horizontal Bragg-brentano focusing geometry was applied for the wide angle X-ray diffraction measurement to characterize the PP crystalline phases.

### 2.5. SEM Observation

JEOL JSM-6100 SEM at a voltage of 10KV was utilized to analyze the dispersion of flax shive in the PP matrix using polished surfaces, and the interface between flax shive and PP matrix using the fractured specimens. Before SEM observation, the samples were coated with Au / Pd to ensure good conductivity.

### 2.6 TGA study

Thermogravimetric analysis was carried out from 25-700°C using a TG 96 SETRAM TGA, at a heating rate of 10°C / min with inert atmosphere.

### 2.7. Mechanical Characterization

Flexural properties were measured according to ASTM test methods D790, and Instron 5500R machine was used. Five specimens were tested for each sample.

## RESULTS AND DISCUSSION

### 3.1 Characterization of FS and EFS

#### 3.1.1 FTIR

The FTIR spectrum of FS and EFS is shown in Fig. 1. The characteristic peak of  $\text{--OH}$  is between  $1000\text{--}1500\text{cm}^{-1}$ , and the peak at  $1730\text{ cm}^{-1}$  presents the refraction of carboxylic group on hemicellulose. The peak at about  $1250\text{ cm}^{-1}$  belongs to a C-O stretching vibration of the acetyl group in hemicelluloses and lignin. The observed peak at  $898\text{cm}^{-1}$  indicates the presence of the  $\beta$ -glycosidic linkages between the monosaccharides. All these characteristic absorbance peaks were similar with other natural fibers, such as hemp, sisal, jute, and kapok [11-13]. The FTIR spectrum of EFS was also shown in Fig. 1. The vanish of the peak at  $1730\text{ cm}^{-1}$  after the extraction process was due to the removal of the carboxylic group on hemicelluloses. The reduction of the peak at about  $1250\text{ cm}^{-1}$  demonstrates that the extraction removed the hemicelluloses and lignin partially. These changes proved the extraction process removed effectively a large amount of non-cellulose portions in flax shive.

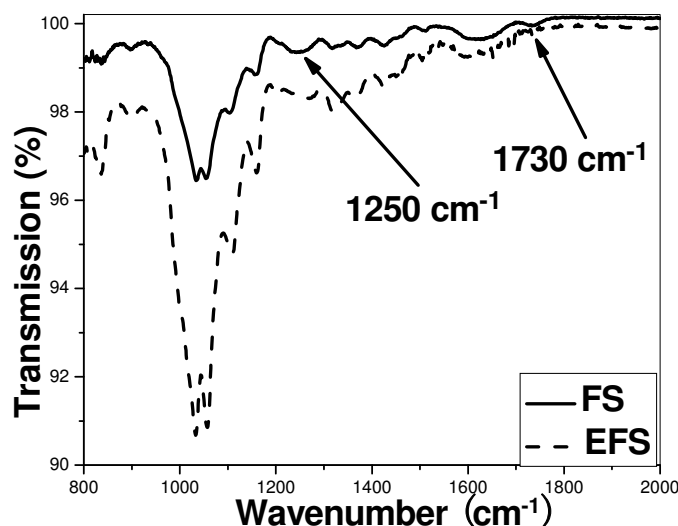


Figure 1. FTIR spectra of FS and EFS.

#### 3.1.2 SEM

Fig. 2 shows the SEM images of FS and EFS surface. It can be seen from Fig. 2(a) that FS was in a porous structure with big tunnels, without fiber observation easily, mainly composed of thick layer of pectin and lignin. EFS showed similar porous structure with comparably smaller porous tunnel than those of FS, as shown in Fig.

2(b), which maybe resulted by the lower ratio of hemicelluloses and lignin. This porous structure endows the FS and EFS weak mechanical properties, however, EFS should present better mechanical properties than FS, in the light of its higher ratio of cellulose and smaller size of porous tunnel.

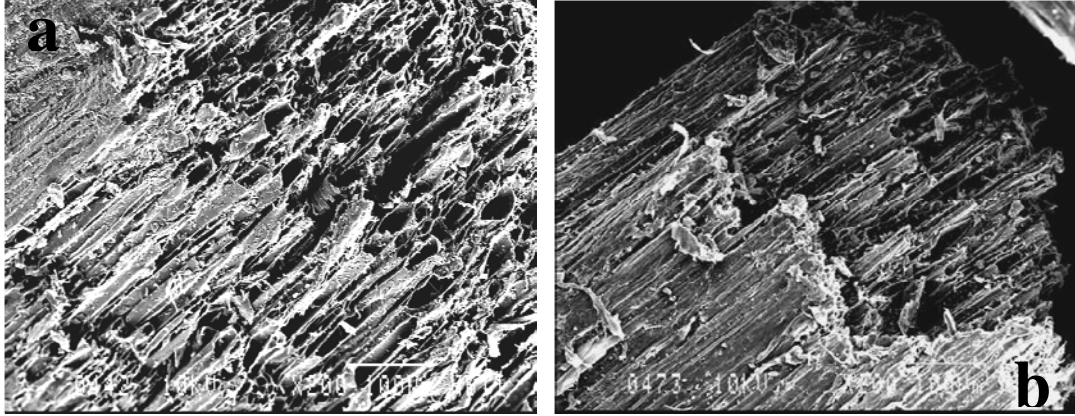


Figure 2. SEM images of (a) FS and (b) EFS.

### 3.1.3 TGA

Thermogravimetric curves for FS and EFS are shown in Fig. 3. It is interesting to observe the TGA curve (Fig. 3(a)) of FS lies below that of EFS, and the temperature of DTG peak (Fig. 3(b)) is lower for FS. This indicated FS has worse resistance to thermal degradation at lower temperature range, which is the main application temperature range for natural material reinforced polymer composites. Table II summarized the decomposition temperature differences for FS and EFS. FS had a surprising low 5% of weight loss temperature ( $T_5$ ) at 92.2°C. This should be resulted by its high water absorption ratio. While  $T_5$  of EFS is 207°C, and  $T_{10}$  is 283°C, which is much higher than that of FS and proper for most polymer composite processing conditions. This is supposed to be resulted by the lower content of non-cellulose portions of EFS, in the light that the thermal stability of non-cellulose portions, such as pectin, lignin, and hemicellulose is lower than that of cellulose [14]. However, EFS presented about 20°C lower  $T_{max}$  than that of FS (340°C). This is maybe resulted by the extraction process, which probably hurt the cellulose structure to some extent.

TABLE II. THERMAL PROPERTIES OF FLAX SHIVE AND EXTRACTED FLAX SHIVE

	$T_5$ (°C)	$T_{10}$ (°C)	$T_{max1}$ (°C)	$T_{max2}$ (°C)
FS	92	265	290	362
EFS	207	283		340

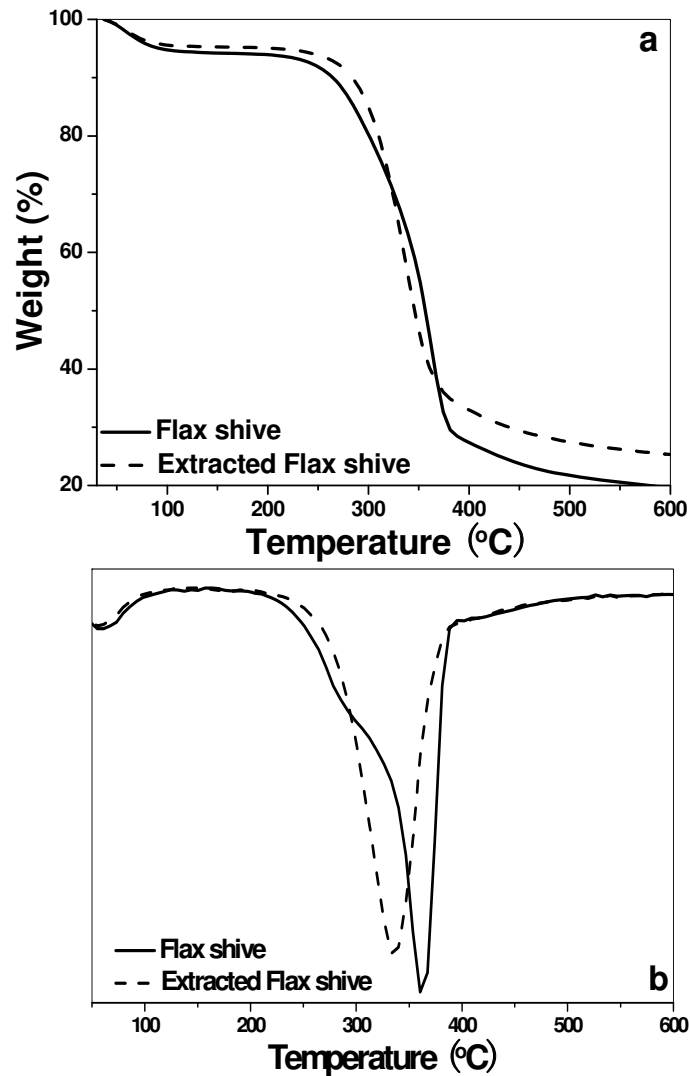


Figure 3. TGA of FS and EFS (a) and DTGA spectra of FS and EFS (b).

### 3.2 Characterization of FS and EFS / PP Composites

#### 3.2.1 DSC

The melting and crystallization behaviour of composites were studied using DSC. According to the crystallization and second heating curves of FS / PP composites (Fig. 4), the composites presented similar crystallization behaviours with PP. Table III shows the value of melting temperature of the second scan ( $T_m$ ), the crystallization temperature ( $T_c$ ) and crystallinity ( $X_c$ ) of samples. It is shown that  $T_m$  for all the composites was similar. However,  $T_c$  increased from 113°C to more than 117°C after the flax shive was inserted into pure PP. This was resulted by the shive that acted as nucleating agent for PP and caused the PP crystallized at a higher temperature during cooling process, when 30% of flax shive added into flax shive / PP composites. The nucleating ability of shives was similar with that of fibers which could accelerate the



crystallization process of PP [15,16]. In addition, the  $X_c$  and  $T_c$  of EFS / PP composite were even higher than those of FS / PP according to Fig. 4 and Table III. This proved that less hemicelluloses and lignin also favored the crystallization process of PP. The insertion of coupling agent with the ratio of 2 % did not increase  $T_c$  and  $X_c$  further, and even made the difference between FS / PP and EFS / PP composites minor.

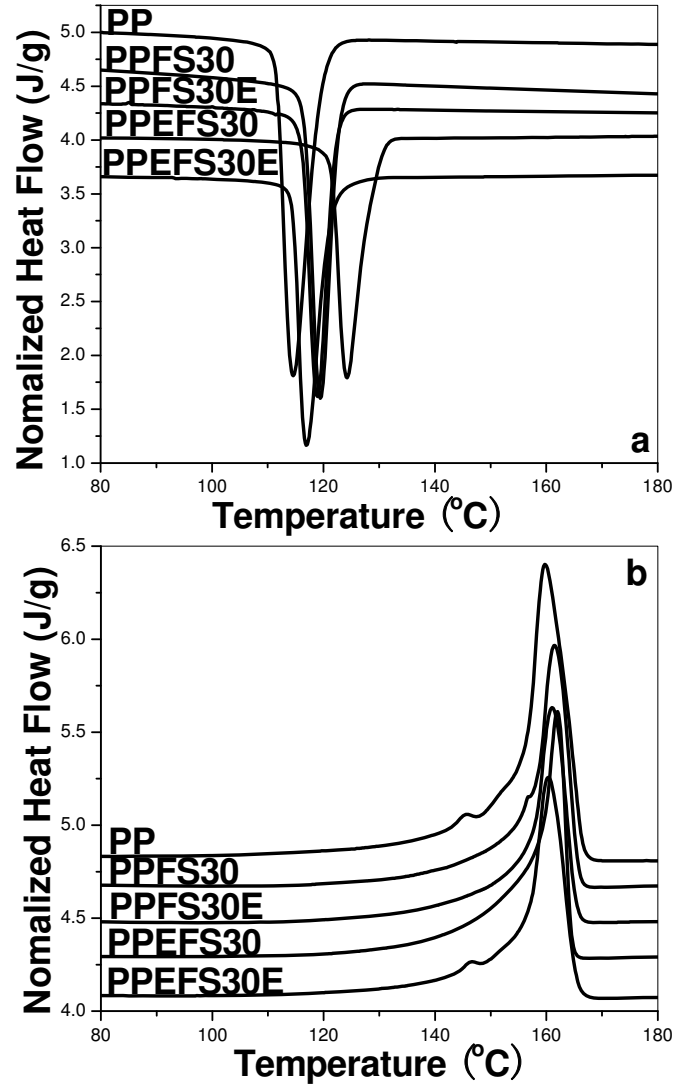


Figure 4. (a) The cooling DSC curves; (b) the second heating DSC curves of flax shive / PP composites.

TABLE III. THERMAL PROPERTIES OF FLAX SHIVE / PP COMPOSITS

	$T_5$ (°C)	$T_{10}$ (°C)	$T_{max1}$ (°C)	$T_{max2}$ (°C)	$T_m$ (°C)	$T_c$ (°C)	$X_c$ (%)
PP	423	435		455	161	113	48.9
PPFS30	280	313	364	462	162	119	48.6
PPEFS30	308	330	338	462	162	124	52.8
PPFS30E	276	310	361	462	161	119	48.7
PPEFS30E	300	324	339	462	160	117	49.9

### 3.2.2 TGA

Fig. 5 shows the TGA thermograms of flax shive / PP composites. Temperature of 5% weight loss ( $T_5$ ) and 10% ( $T_{10}$ ) of all the samples were shown in Table III. There was a sudden decrease that began at about 250 °C for the four composites in thermogravimetric curves (Fig. 5 (a)). This decrease was corresponding to the thermal depolymerization of hemicelluloses, which was more apparent by the centering minor peak at about 290°C of FS composites in Fig. 5 (b). In addition,  $T_5$  and  $T_{10}$  of EFS composites were always higher than that of FS composites, which proved further that more flammable non-cellulose portion, such as hemicelluloses, lignin, etc. existed in FS than in EFS.

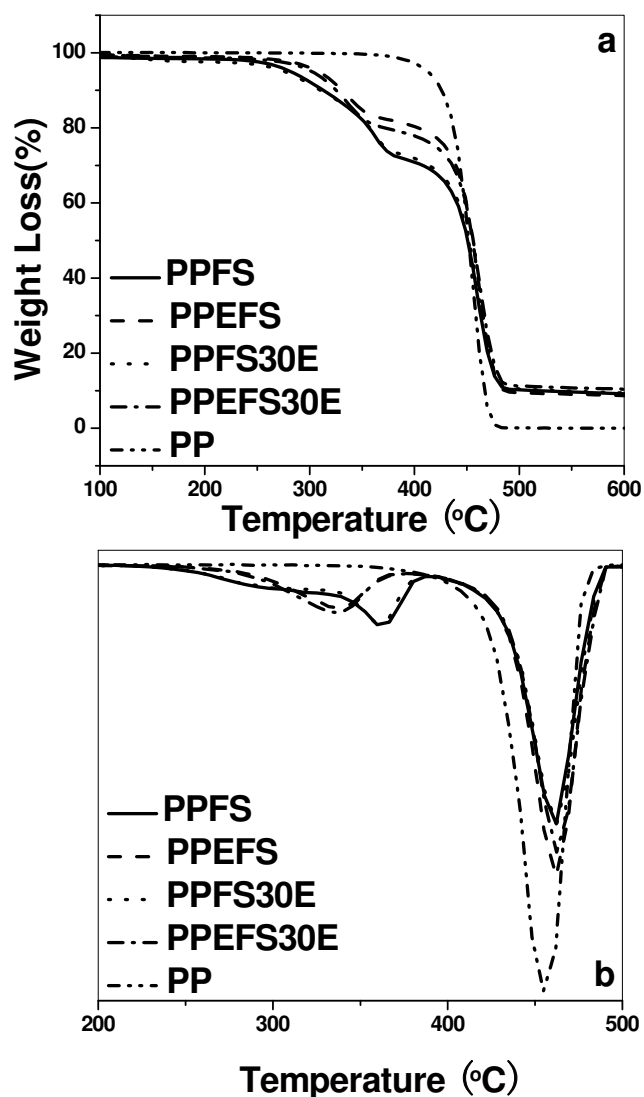


Figure 5. TGA (a) and DTGA spectra (b) of FS and EFS reinforced PP composite.

In the DTG curves of these composites (Fig. 5(b)), two peaks were obtained. The minor peak at about 350 °C was related to the degradation of cellulose, and the higher

peak at about 462 °C was corresponded to the degradation of polypropylene and dehydro cellulose [16]. It is shown in Fig. 5 (b) that the EFS composites degraded at lower temperature for  $T_{max1}$  than FS, though EFS has lower ratio of noncellulose portions. This may be resulted by the extraction process of hemicelluloses and lignin which hurt the cellulose structure by some extent. There was no much difference for the major peak  $T_{max2}$  between FS and EFS composites, because the degradation temperature of dehydro cellulose and PP could not be affected by the cellulose ratio. Furthermore, all the composites degraded at comparably higher temperature than PP, which means the thermal stability of composites was better than that of matrix. It was supposed that the flax shive / matrix interactions resulted in this higher thermal stability of composites.

### 3.2.3 XRD

WAXS patterns of PP and flax shive / PP composites are shown in Fig. 6. The observed diffraction curves were all characteristic of the  $\alpha$  – monoclinic crystal structure of polypropylene with main reflections at about 14°, 16.8°, 18.5° and 21° of 2 $\theta$  corresponding to the (110), (040), (130) and (111) planes, respectively. In addition, there was significant difference in the intensity of the reflections of  $\alpha_1$  (110) and  $\alpha_2$  (040) between composites and PP. This difference on intensity indicated some orientation of PP  $\alpha$ -crystallite in the composites [17], which was probably related to the orientation of flax shive induced by the compression molding. Similar orientation phenomenon was also found in PP / clay / coupling agent nanocomposites [18].

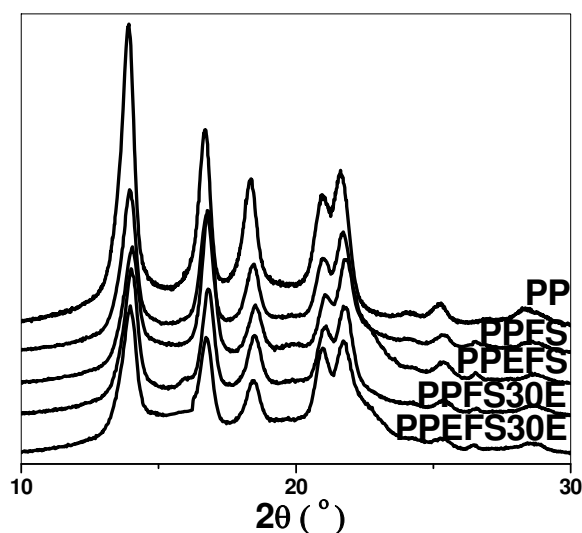


Figure 6. XRD spectra of flax shive / PP composite.

### 3.2.4 MECHANICAL PROPERTIES

The flexural strength, modulus and short beam shear strength of FS and EFS reinforced PP composites with E43 are shown in Fig. 7. Flexural strength is a

combination of the tensile and compressive strengths. Flax shive insertion decreased the flexural strength compared to pure PP for both FS and EFS. When compatilizer E43 was added into composites, the flexural strength became higher than that of neat PP, and even could increase almost 8 % compared with pure PP for EFS / PP composite (Fig. 7(a)), which indicates an improvement of interface adhesion and effectively stress transfer from the PP matrix to flax shive. The scattering of short beam shear strength of FS and EFS reinforced PP composites was similar with that of flexural strength, though all the value was still lower than that of PP (shown in Fig. 7 (c)). This was supposed to be related to the modification on the molecular level of fibers due to chemical bonding between fiber components and coupling agents. The hydroxyl groups on cellulose fiber can react with the anhydride functionality of maleated PP into ester bond [7,9,16]. Coupling agent E43 in these composites was supposed to provide the formation of such bonds between shive and matrix and resulted in an improved interfacial property of composites.

All composites showed remarkably higher flexural modulus than pure PP as shown in Fig. 7 (b). The flexural modulus increased 67% compared with PP for FS / PP composite, and almost 100% for EFS / PP composite. The reason should be the higher modulus of FS and EFS than PP. Surprisingly, the addition of coupling agent E43 did not result in an even higher modulus of composites than those without E43. Thus, the extracted flax shive could be used as reinforcing material of PP composite, with both strength and modulus much increased.

On the other hand, as shown in Fig. 7, the strength and modulus of EFS / PP composites were always higher than those of FS / PP composites. It is supposed that the mechanical properties of EFS were higher than that of FS, which was resulted by the higher cellulose ratio in the EFS. There were more hemicellulose and lignin remained on FS surface that would affect the mechanical property of composites, and also prevent the interaction between flax shive and matrix produced by the coupling agent. Therefore, according to all the above factors, the mechanical property of EFS / PP composites was better than that of FS/ PP composites. EFS was concluded to be kind of reinforcing material for PP, while FS could only be used as filler for composite.

### 3.2.5 SEM OBSERVATION

The effect of compatibilizer E43 for the interfacial property of flax shive / PP composites was investigated by SEM observation on the fracture surfaces of flexural test specimens, as shown in Fig. 8. It is shown very clearly in Fig. 8 (a) and (b) that the interfacial adhesion between flax shive and PP matrix of composites without E43 was poor proved by much more pulled out shives and fibers, the gap at the interface between shive and PP matrix, and the clean shive surface without much adhering polymer. While in Fig. 8 (c) and (d), the adhesion of composites with E43 was much better. There was no obvious gap between fiber and PP matrix, and the flax shive was coated and adhered by PP. The PP matrix of EFS composite was even stretched by the flexural test, which proved the strong adhesion between shive and matrix. One could even not differentiate flax shive from PP matrix for EFS composite as shown in Fig. 8 (d). The interface between EFS and matrix was proved to be better than that between FS and matrix with less cracks and shive sticking out observation and stretched matrix. This is supposed to be resulted by the more hemicellulose, lignin on the shive

surface of FS which may decrease the interaction between fiber and matrix further bonded by the coupling agent. The good interfacial adhesion could make the stress transfer well from the fiber to matrix, and thus the failure mainly happened in matrix, not on the shive surface.

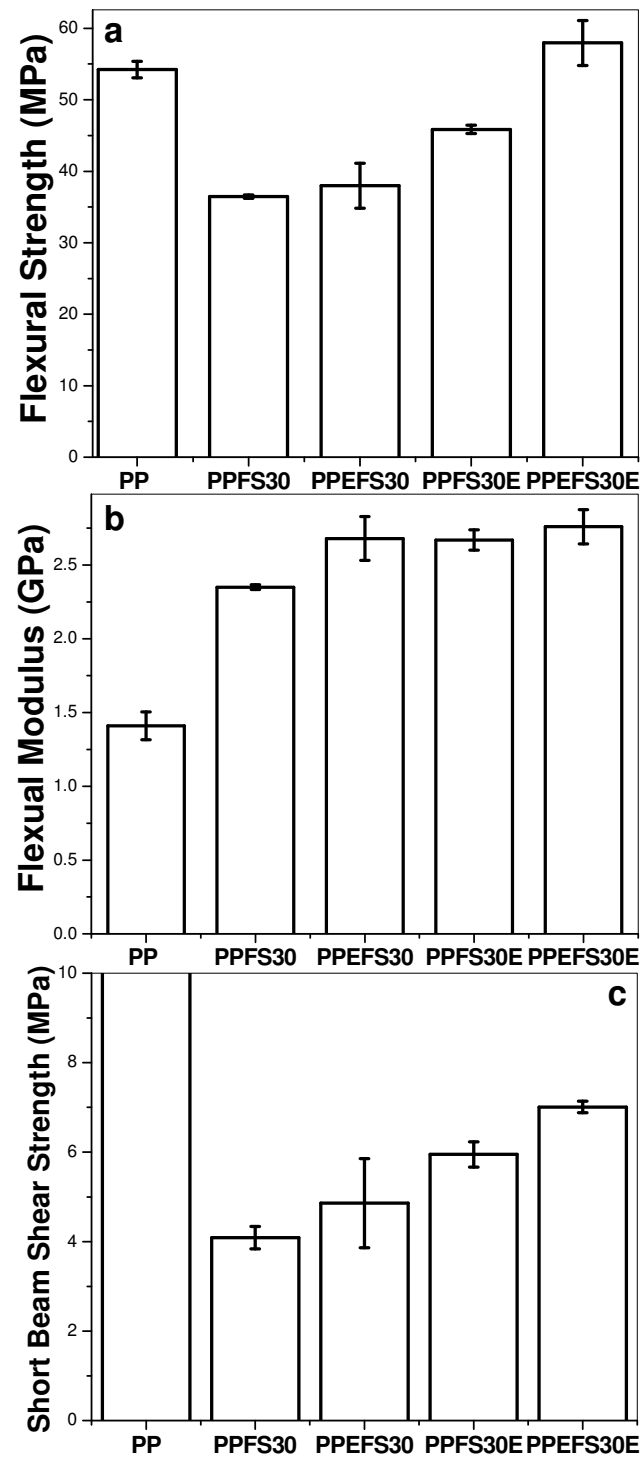


Figure 7. Flexural strength (a) and flexural modulus (b) and short beam shear strength (c) of flax shive/PP composites as a function of formulation.

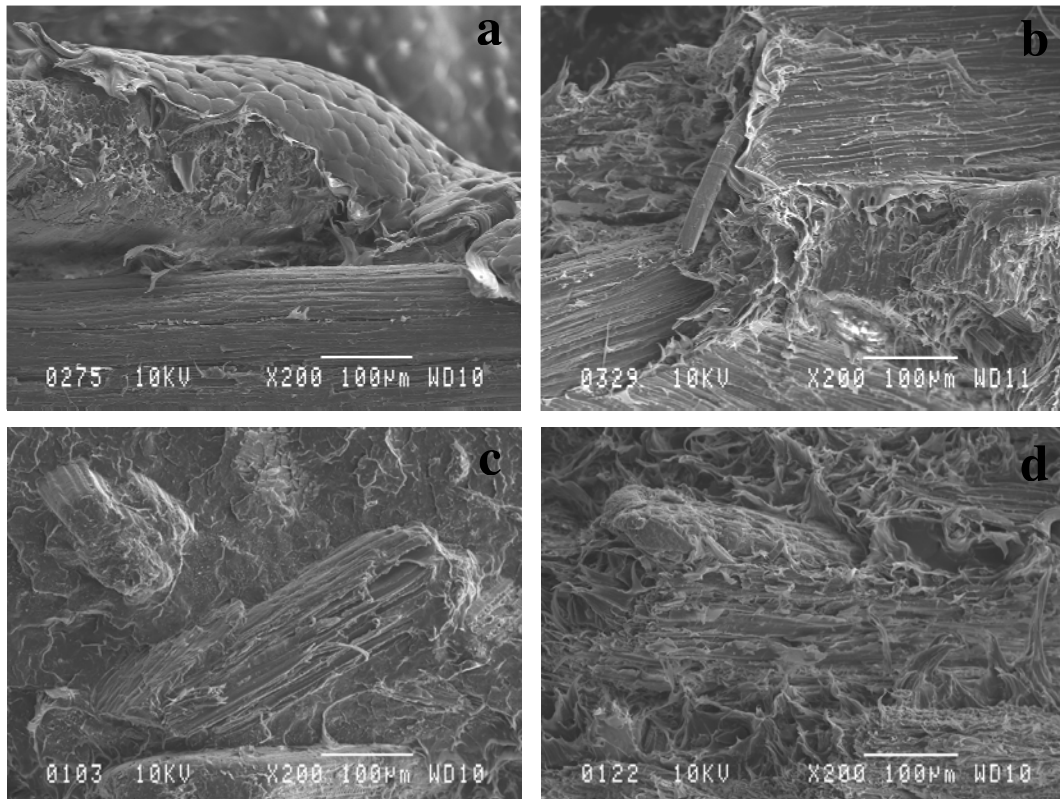


Figure 8. SEM images of composites: a. PPFS, b. PPEFS, c. PPFS30E, d. PPEFS30E.

## CONCLUSION

FS is the waste product left after the removing of the fibers from flax straw. It is woody part composing of over 70 wt% of the straw. After the special extraction process, hemicelluloses and lignin of FS were effectively removed and the cellulose content of EFS increase ~7%. Both FS and EFS were characterized and further used as reinforcing materials for PP composite. Compatibilizer was added to get the best compatibility between the FS and the PP matrix. It was shown that EFS presented much less flammable hemicelluloses and lignin content, and thus better thermal stability than FS at normal compounding temperature range. The porous tunnel of EFS was also shown to be smaller than FS by SEM observation, which should endow EFS better mechanical properties than FS. When they are applied in PP composites, the nucleating ability of flax shives was similar with that of fibers which could accelerate the crystallization process of PP. And the observed XRD diffraction curves of composites were all characteristic of the  $\alpha$  – monoclinic crystal structure of PP. FS appears to behave as filler even with compatibilizer. However, with the presence of coupling agent E43, EFS could perform as a reinforcing material for PP composites. When 30 % EFS was added in the composite, the tensile strength and the modulus can increase almost 8% and 100% over than PP, respectively. The thermal stability of EFS / PP composites was also higher than that of FS / PP composites.

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