Effects of Rice Straw Fiber Morphology and Content on the Mechanical and Thermal Properties of Rice Straw Fiber-High Density Polyethylene Composites

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ABSTRACT: Rice straw fiber-high density polyethylene (HDPE) composites were prepared to investigate the effects of rice straw fiber morphology (rice straw refined fiber, rice straw pellet, rice straw strand), fiber content (20 and 40 wt %), and maleic anhydride polyethylene (MAPE) concentration (5 wt %) on the mechanical and thermal properties of the rice straw fiber-HDPE composites in this study. Rice straw refined fiber exhibited more variability in length and width, and have a higher aspect ratio of 16.3. Compared to the composites filled of rice straw pellet, the composites made of the refined fiber and strand had a slightly higher tensile strength and lower tensile elongation at break. The tensile and flexural strength of the composites increased slightly with increasing rice straw fiber content up to 40 wt %, while the tensile elongation at break decreased. With addition MAPE, the composites filled with 20 wt % rice straw fiber showed an increase in tensile, flexural and impact strength and a decrease in tensile elongation at break. Differential scanning calorimetry showed that the fiber addition and morphology had no appreciable effect on the crystallization temperature of the composites but decreased the crystallinity. The scanning electron microscopy observation on the fracture surface of the composites indicated that introduction of MAPE to the system resulted in promotion in fiber dispersion, and an increase in interfacial bonding strength. Fiber breakage occurred significantly in the composites filled with refined fiber and strand after extruding and injection processing. J Appl Polym Sci 121: 2900–2907, 2011

Key words: composites; fibers; mechanical properties; polyethylene (PE); morphology

INTRODUCTION

In recent years, polymer composites reinforced with natural fiber such as wood, corn stalk, flax, hemp wheat straw, newspaper fiber, sunflower stalk, and bagasse fiber have become popular due to their renewable, recyclable, and biodegradability.[1–6] These composite products are characterized by a unique combination of excellent durability, superior dimensional stability, high rigidity, and relatively low density.[7,8] Wood fiber has traditionally been the major natural fiber used for the manufacturing of natural fiber-reinforced plastic composites. In recent years, other types of natural fiber such as wheat straw, corn stalk, flax, and hemp have also been used as fillers for the manufacturing of thermoplastic composites. The utilization of agriculture fibers as an alternative to wood fiber for natural fiber plastic composites can reduce the consumption of wood fiber and thus help protect forest and environment.

In processing of natural fiber and polymer composites, the properties of the polymer composites are influenced by fiber characteristics such as fiber content, fiber size, and fiber manufacturing processing. In our previous study of wheat straw fiber-polypropylene (PP) composites,[9] it was reported that the PP composites made of wheat straw fines had a slightly higher tensile elongation at break and tensile strength than those made of the longer wheat straw fiber. Stark et al.[10] reported the effect of different sizes of wood flour particles and refined fiber on the mechanical properties of wood-flour-filled PP composites, and suggested the use of higher aspect ratio wood fibers for increasing the strength of the resultant composites. Panthapulakkal et al.[11] presented wheat straw and corn stem filled polypropylene composites, and found that high shear compounding of wheat straw fibers exhibited similar properties to that produced by the milled wheat straw, due to fiber breakage occurred during the high shear compounding that results in a similar aspect ratio to that of milled straw. It was interesting to note that small wood powder particles lead to improved properties, even

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without addition of maleic anhydride polypropylene (MAPP). For example, the PP composite containing 20 wt % wood powder (< 38 μm) had Young's modulus and elongation at break similar to those of the pure PP, but a higher stress at break than the pure PP.[12] During the processing of polymer composites, natural fibers have been widely used for the polymer composite manufacturing in the form of pellets or powder form rather than in the fibrous form.[13,14]

Rice straw fiber has also considered as potential filler for the manufacturing of thermoplastic composites. Precious studies have mainly focus on rice husk and milled rice straw-reinforced thermoplastic composites. Park et al.[15] explored the rice husk surface modified with MAPP and silane. Tajvidi et al.[16] reported the water absorption behavior of rice hull filled PP. Panthapulakkal et al.[17] studied the extruded rice-husk-filled high density polyethylene (HDPE) composite affected by coupling agents. Yao et al.[18] studied the mechanical properties and crystallization behavior of the milled rice straw reinforced HDPE composites affected by fiber type and loading. Madhoushi et al.[19] presented the withdrawal strength of rice straw fiber-thermoplastic composites under dry and wet conditions. So far very limited studies have examined the rice straw refined fiber reinforced thermoplastic composites.

The objectives of this study are to investigate the relationships of the tensile, flexural and impact properties, thermal behavior and surface morphology of the rice straw fiber-HDPE composites with the following variables: (1) the rice straw fiber content (20 and 40 wt %); (2) rice straw fiber manufacturing processing (refined fiber, pellet, and strand); and (3) the addition of MAPE (5 wt %).

EXPERIMENTAL

Materials

Rice straw refined fiber (RF) samples for this study were obtained from the pilot plant of Engineering Research Center of Fast-growing Trees and Agrifiber Materials, Jiangsu, under the following refining conditions: steam pressure of 1.2 bar, and 8.0% moisture content. Rice straw pellet samples for this study were prepared with a hammer miller, and samples were produced with the screen system equipped with 60 and 80 mesh screens. Rice straw strand samples for this study were prepared with a chipper, and samples were produced with the screen system equipped with 20 and 40 mesh screens. Figure 1 (a–d) showed the appearance of rice straw, rice straw refined fiber, rice straw pellets, and rice straw strand, respectively, at the same magnification.

The high density polyethylene (HDPE) used in this work, was a homopolymer pellet, grade 5000S, ρ = 0.95 g cm^{-3}, melt flow index (MFI) 0.8–1.2 g/10 min (190°C/2.16 kg), kindly supplied by Sinopec Yangzi Petrochemical Company Ltd. Maleic anhydride polyethylene (MAPE) has 0.85 wt % maleic anhydride (MAPE), grade PE-G-1, supplied from Deba Chemical Co.Ltd., Nanjing, was used as compatibilizer.

Processing of rice straw fiber-HDPE composites

The HDPE, MAPE, rice straw fiber was dried at 105°C to constant weight before extruding process. The polymer, fibers and compatibilizer were preblended in a mixer and then extruded using a two screw extruder with a length-to-diameter ratio *L/D* of 36. The barrel temperatures were 140°C (Zone I, feeding), 130 C (Zone II, melting), 110°C (Zone III, pumping), and 161°C at die, respectively, and the screw speed was 50–70 rpm. The extrudate was granulated using a chipper setting at the die. Afterwards, the granules were molded into test specimens by an injection molder, Chen de plastics machinery, at a molding temperature of 140°C. A flow chart of sample preparation and characterization was given in Figure 2. Compositions of the rice straw fiber-HDPE composites prepared were summarized in Table I according to a previous article.[9]

Morphology characterization

The sizes of rice straw fiber, including length and diameter were measured with an imaging system. An imaging system consists of Nikon microscope (a Nikon SMZ800) with camera, and a computer. Each fiber sample was evenly placed on a glass dish under the microscope and the fiber images were taken with a camera and recorded with Imaging MVS3000 software. Then, Image MVS3000 was used to measure the length and width of individual fibers. The aspect ratio was calculated as the ratio of the fiber length and width for each fiber. Hundred counts were taken for each sample.

Tensile testing

Tensile properties of the rice straw fiber-HDPE composites were performed with a SANS CMT 6104 tester. The tensile properties were determined in accordance with the National Standards of the People's Republic of China GB/T 1040-1992 procedure at a cross head rate of 2 mm min^{-1}. Six specimens were tested in each run.

Flexural testing

Flexural test of 80 × 10 × 4 mm^3 test pieces were also performed with a SANS CMT 6104 tester. The flexural properties were determined according to the National

Figure 1 The morphology of rice straw (a) rice straw, (b) rice straw refined fiber, (c) rice straw ground pellets, and (d) rice straw strand. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Standards of the People's Republic of China GB/T 9341-2000 in the three point bending mode at a cross head speed of 2 mm min^{-1} and with a span of 67.4 mm. Six specimens were tested in each run.

Impact testing properties

The unnotched charpy impact test of 80 × 10 × 4 mm^3 test pieces were performed with a SANS ZBC 1251-1 tester. The impact properties were determined according to the National Standards of the People's Republic of China GB/T 16420-1996. Six specimens were tested in each run. All the tests were performed at 25 ± 2°C.

Differential scanning calorimetry

Differential scanning calorimetry (DSC) measurements were performed on a DSC 200 F3 (NETZSCH) with ~ 5 mg sample on nitrogen atmosphere. Samples test procedures were as follows: (1) heating from 25°C to 150°C at 10°C · min^{-1} and with hold for 5 min to eliminate the thermal history effect; (2) cooling from 150°C to 25°C at 5°C · min^{-1} for determining the crystallization temperature (T_c); and (3) reheating to 150°C at 10°C · min^{-1} to determine the melting temperature (T_m). The degree of crystallinity (X_c) of the PE component was determined from the following equation:

$$X_c = \frac{\Delta H}{\Delta H^{\circ}} \times 100\% \qquad (1)$$

where ΔH and ΔH° are the heat of fusion of the rice straw fiber-HDPE composites and 100% crystalline polyethylene, respectively. In this calculation, ΔH° is taken to be 293 J g^{-1}.[20]

Scanning electron microscopy

To better understand the interfacial adhesion between the rice straw fiber and matrix, the fracture surfaces of the impact tested samples were observed

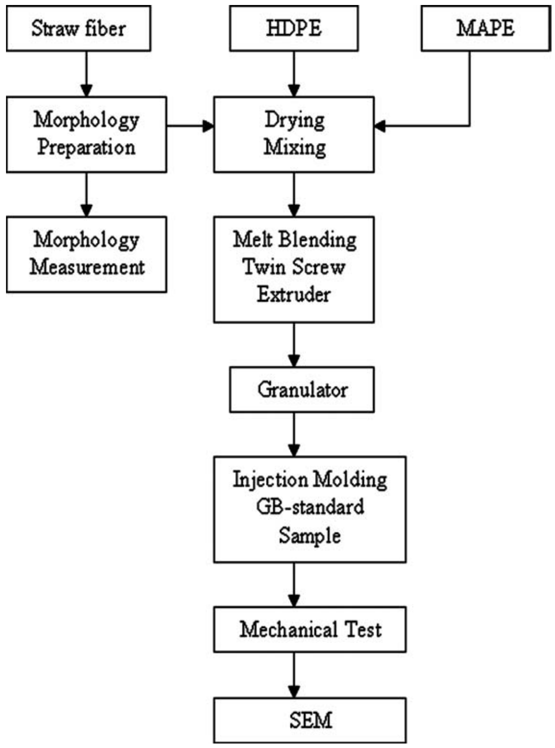

Figure 2 Flow chart of sample production and testing.

using a QUANTA 200 SEM (FEI Company) at an accelerating voltage of 17 kV. The surfaces were coated with gold before examination.

RESULTS AND DISCUSSION

Fiber dimension distribution

The rice straw fiber generated from the three kinds of manufacturing process exhibited distinct morphological traits. Size measurements (i.e., length and width) were listed in Table II. The length and width was affected significantly by the rice straw fiber manufacturing processing. After grinding, rice straw pellet had a homogeneous morphology, with 0.36–2.89 mm in length, and 86.76–395.16 μm in width. Rice straw pellet indicated the "square-like"[18] feature with aspect ratio of 6.19. RF derived from refining process, was consisted of fiber bundle, fines, and powder, resulting in fibrous-looking [Fig. 1(b)]. RF exhibited highest variability, with a range of length from 0.18 mm to 1.14mm, and a range of width from 31.92 μm to 223.26 μm. However, the rice straw strand showed an appreciable increase in the length (from 2.81 mm to 21.19 mm) and width (from 281.24 μm to 1500.56 μm) in processing of chipping.

Tensile properties

The tensile properties of the rice straw fiber-HDPE composites were influenced by rice straw fiber morphology and content, as shown in Figure 3. Figures 3(a,b) showed that at the same proportion of HDPE and MAPE, the tensile strength [Fig. 3(a)] followed the order: RF > pellet > strand, and the tensile elongation at break [Fig. 3(b)]: pellet > strand > RF. The improvement in tensile properties of the HDPE composites reinforced with RF most likely due to a higher aspect ratio of 16.31 and intrinsic tensile strength of RF comparing to that of pellet and strand. The higher aspect ratio enhanced stress transfer from matrix to the fiber. It was also reported that maximum strength and modulus occurred in PP composites filled with higher aspect ratio wood flour.[10]

The tensile properties of the HDPE composite increased with addition of the rice straw fiber to HDPE matrix. With increasing rice straw fiber content from 20 to 40 wt %, the tensile strength of the HDPE composites increased slightly for RF, while it decreased for pellet and strand. The tensile elongation at break of the composites decreased to nearly 7.0% when the rice straw fiber increased to 40 wt %. Reduction in tensile elongation may be due to the decreased deformability of a rigid interphase between the rice straw fiber and the matrix.

To improve the mechanical properties of the rice straw fiber-HDPE composites, 5 wt % MAPE were recommended to add to the system of rice straw fiber-HDPE composites.[9,21] Addition MAPE to the

TABLE I
Composition of Rice Straw Fiber/HDPE Composites (by Weight)

Sample code	Conditions	Fiber species	PE	Rice straw fiber	MAPE
1	HDPE	–	100	0	0
2	PE80R20	Refined fiber	80	20	0
3	PE75R20M5	Refined fiber	75	20	5
4	PE55R40M5	Refined fiber	55	40	5
5	PE80P20	Pellets, 60–80 mesh	80	20	0
6	PE75P20M5	Pellets, 60–80 mesh	75	20	5
7	PE55P40M5	Pellets, 60–80 mesh	55	40	5
8	PE80S20	Strand, 20–40 mesh	80	20	0
9	PE75S20M5	Strand, 20–40 mesh	75	20	5
10	PE55S40M5	Strand, 20–40 mesh	55	40	5

TABLE II
The Lengths, Widths, and Aspect Ratios of the Fiber Produced from Different Processing

Fiber species	Length (mm) Min	Length (mm) Max	Length (mm) Mean	Width (μm) Min	Width (μm) Max	Width (μm) Mean	Aspect ratio
RF	0.179	2.906	1.137 (0.610)	31.92	223.26	82.50 (37.77)	16.31 (10.68)
Pellets	0.355	2.890	1.039 (0.444)	86.76	395.16	185.77 (56.00)	6.19 (3.44)
Strand	2.811	21.190	10.545 (4.152)	281.24	1500.56	809.78 (270.47)	14.48 (7.32)

The values in parentheses are the standard deviations.

fiber-HDPE composites had the higher tensile strength for three morphology of 20 wt % rice straw fiber, which represented an increase by 21.0% for RF, 16.2% for rice straw pellet, and 4.6% for rice straw strand, respectively. It is noted that MAPE gave high tensile strength in HDPE composites indicated the high interfacial adhesion between the rice straw fiber and matrix. Furthermore, MAPE addition decreased the tensile elongation at break, and the tensile elongation at break reduced by 9.3% for RF, 7.3% for rice straw pellet, and 9.3% for rice straw strand, respectively. The stronger interfacial bonding between the HDPE matrix and rice straw fiber reduced the ductility of the HDPE matrix and the tensile elongation of the rice straw fiber-HDPE composites at break.[9]

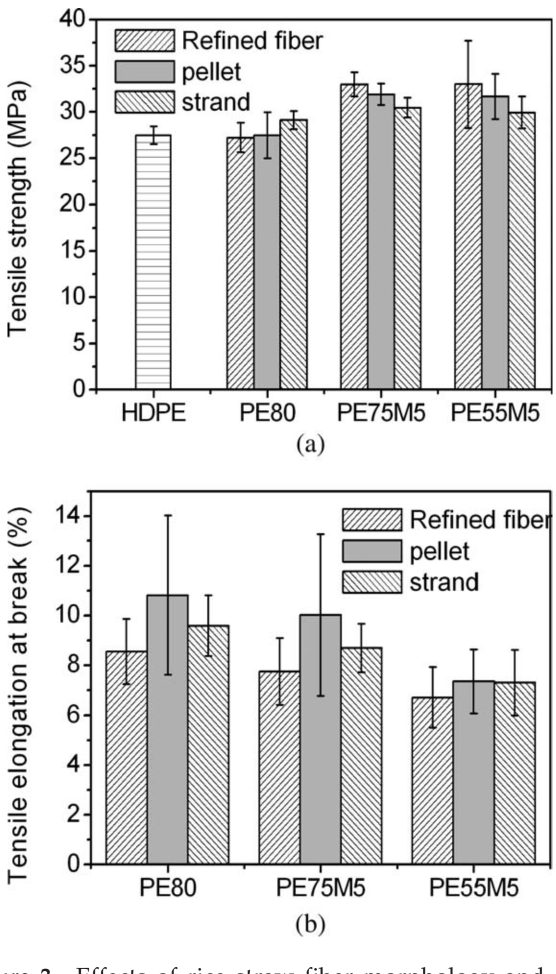

Figure 3 Effects of rice straw fiber morphology and content on the tensile properties of the composites (a) tensile strength, and (b) tensile elongation at break.

Flexural properties

Figure 4 showed the flexural properties of the rice straw fiber-HDPE composites in relation to rice straw fiber content, morphology and MAPE addition. As rice straw fiber content increased from 20 to 40 wt %, the flexural strength of the HDPE composites increased by 4.7% for RF, 6.6% for pellet, and 2.7% for strand, respectively. The increased flexural strength may result from the fact that the stiffness of the rice straw fiber is higher than that of the HDPE matrix and the reinforcement introduced by the rice straw fiber enables stress to be transferred from the matrix to the rice straw fiber.[22] Moreover, the morphology and size of the rice straw fiber affected the flexural property of the composites. At the system of HDPE and MAPE composites filled with 20 and 40 wt % rice straw pellet, the composites both showed highest flexural strength of 33.1 MPa and 35.3 MPa. This was probably less stress transferred caused by the "square-like" shape of pellet, with the lowest aspect ratio of 6.19. This finding was consistent with that of a previous study.[18] Figure 4 also

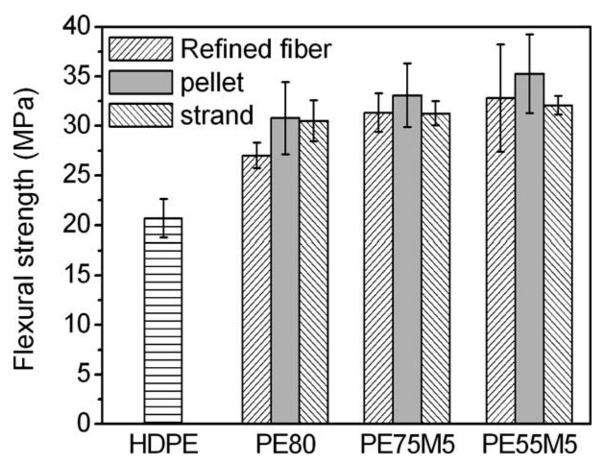

Figure 4 Effects of rice straw fiber morphology and content on the flexural properties of the composites.

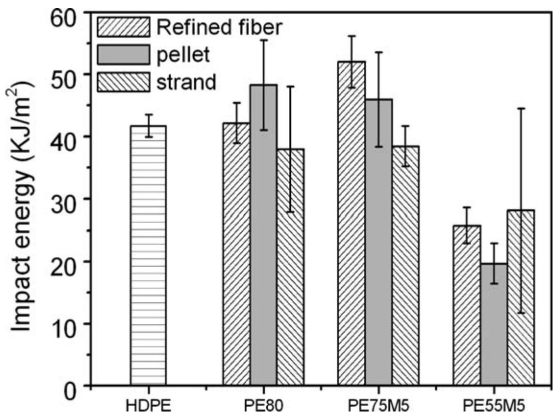

Figure 5 Effects of rice straw fiber morphology and content on the impact strength of the composites.

showed the flexural property of RF and strand composites at 20 wt % and 40 wt % filler levels, respectively, with or without the addition of MAPE. The use of RF and strand had little effect on flexural property in our study. This was possibly due to the breakage of RF and strand with larger size and higher aspect ratio in processing of extrusion and injection. Similar to the tensile strength, the flexural strength of the HDPE composite increased with addition of MAPE to the system. The 5 wt % MAPE giving the higher flexural strength in the HDPE composites reinforced with 20 wt % rice straw fiber indicated the higher interfacial adhesion between the rice straw fiber and matrix.

Charpy impact properties

The Charpy unnotched impact strength of the composites was presented in Figure 5. It was seen that the impact strength of the HDPE composites filled with 20 wt % RF and pellet were found to be higher than that of the pure HDPE. It was because the additional mechanisms of energy absorption effective during fracture in the composites with 20 wt % RF and pellet.[23] It also showed that the impact strength of HDPE reinforced with 20 wt % strand decreased in comparison to that of pure HDPE and composites reinforced with 20 wt % RF and pellet, and it mainly due to energy dissipated by the rice straw strand with more rigid. As the rice straw fiber content increased from 20 to 40 wt %, the impact strength of the filled HDPE composites was reduced. As a rigid material, rice straw fiber had a higher stiffness than pure HDPE, addition of 40 wt % rice straw fiber thus led to a higher tensile and flexural strength of the rice straw fiber-HDPE composites. However, the HDPE composites with 40 wt % rice straw fiber became more rigid and fiber agglomeration also increased, creating regions of stress concentration, thus, decreasing the impact strength of composites.[23] Furthermore, with the addition of 5 wt % MAPE composites, impact strength increased the most for 20wt% RF-HDPE composites at around 23.3%. This was due to MAPE provided better

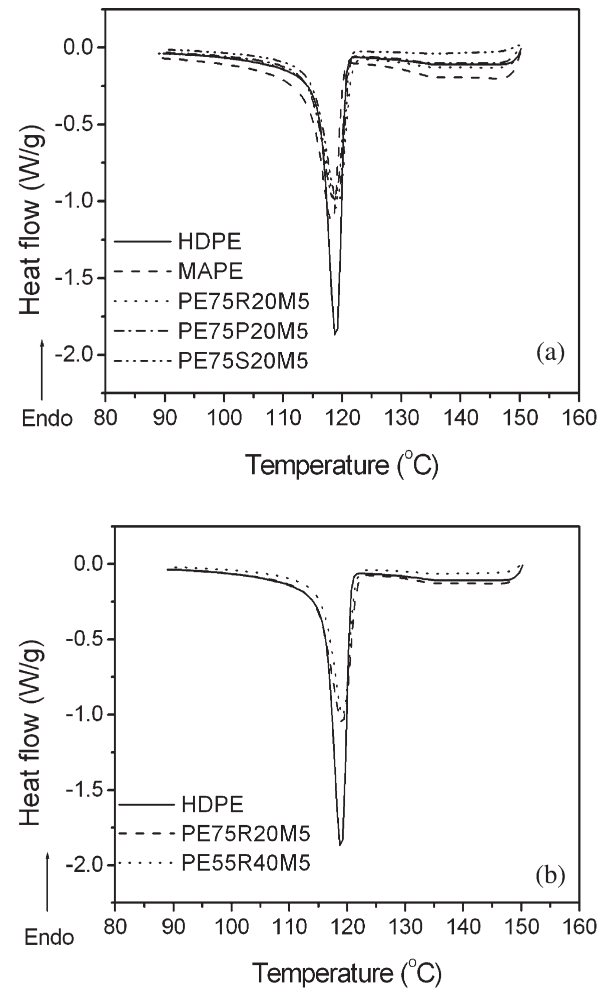

Figure 6 The DSC thermograms of rice straw fiber/HDPE composites during cooling affected by (a) fiber morphology, and (b) fiber addition.

TABLE III
Summary on DSC of the Rice Straw Fiber/HDPE Composites

Sample code	T_c °C	ΔH_c J/g	T_m °C	ΔH_m J/g	X_c %
HDPE	119.0	130.5	133.3	151.9	44.54
PE80R20	119.2	122.9	136.3	133.1	41.95
PE75R20M5	119.1	108.5	136.2	120.5	37.03
PE55R40M5	119.2	97.56	135.1	107.5	33.30
PE80P20	118.8	118.8	135.9	133	40.55
PE75P20M5	118.7	106.8	136.8	116.7	36.45
PE55P40M5	118.8	91.74	135.1	99.77	31.31
PE80S20	119.2	122.2	135.3	136.3	41.71
PE75S20M5	119.0	107.1	136.5	119.6	36.55
PE55S40M5	119.2	89.73	134.8	95.45	30.62
MAPE	118.2	122.9	131.8	142.6	41.95

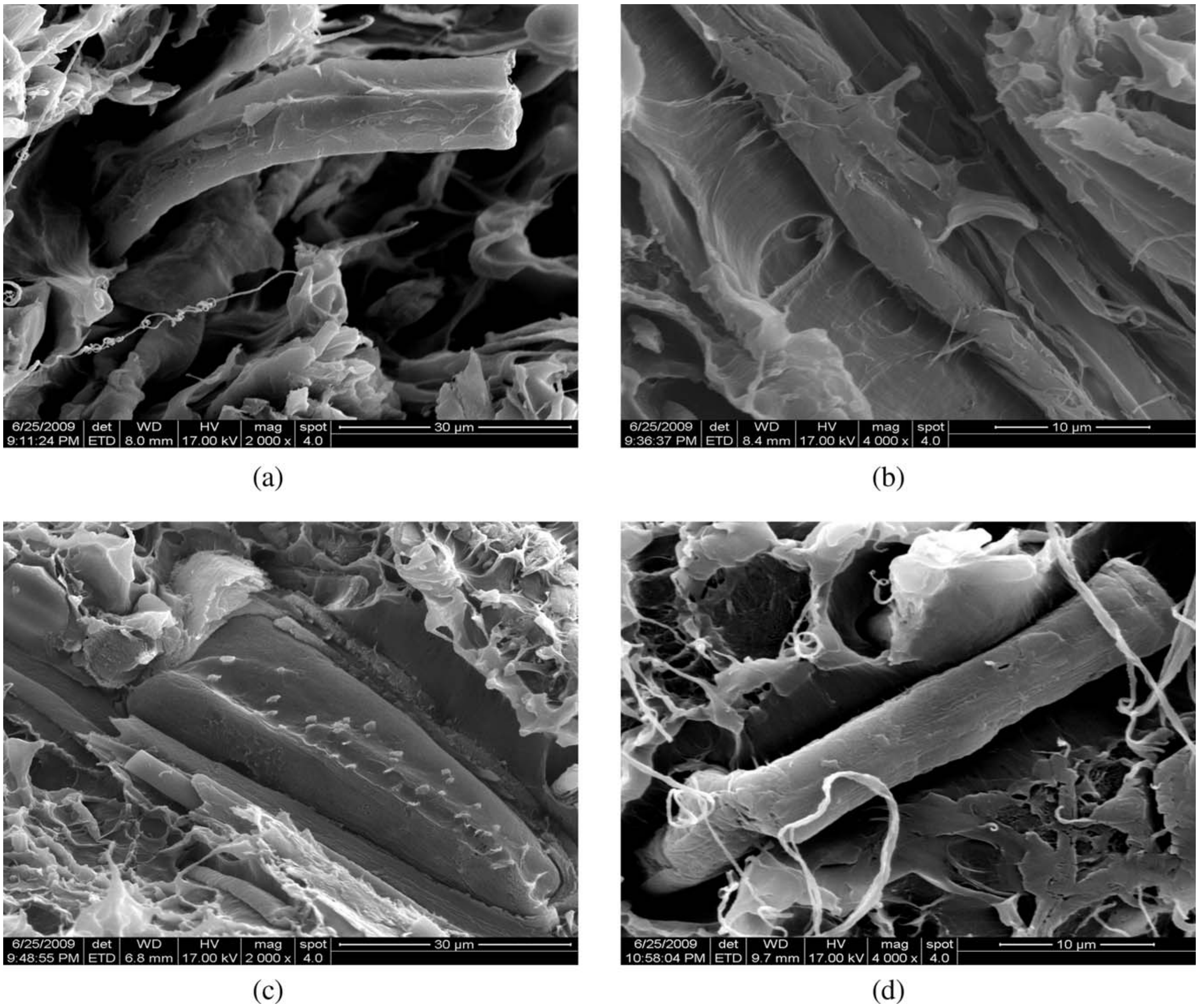

Figure 7 SEM observations of the composites (a) PE80R20, (b) PE75R20M5, (c) PE75P20M5, (d) PE75S20M5.

adhesion between the matrix and RF, and that required more energy to initiate or propagate a crack.

Thermal and crystallization analysis

The thermal properties and crystallization behavior of composites affected by rice straw fiber morphology and addition were analyzed by DSC. The results of crystallization temperature (T_c), heat of crystallization (ΔH_c), melt temperature (T_m) and the degree of crystallinity from DSC were summarized in Table III.

Figure 6(a) showed thermograms for the crystallization of the HDPE composites filled with 20 wt % rice straw fiber and 5 wt % MAPE. Generally, the introducing of natural fibers increased the crystallization temperature (T_c) of polymer with an assumption that the natural fibers act as efficient nucleating agents for the crystallization of polymer and consequently increase its crystallization growth rate during the cooling from the molten state.[24,25] As shown in Figure 6(a), this influence was not significantly observed in the HDPE composites filled with rice straw RF and strand. With regard to the degree of crystallinity (X_c), calculated according to eq. (1) for the HDPE composites, fiber morphology influenced X_c of HDPE significantly. The DSC curves during cooling clearly demonstrated that the rice straw RF had a higher X_c and that was 37%. It indicated more nucleation when compared with rice straw pellet and strand. Figure 6(b) showed thermograms for the crystallization of pure HDPE and the composites filled with rice straw RF addition. In the presence of rice straw RF from 20 to 40 wt %, the value of X_c of HDPE decreased by 10.8%. This could be attributing to the dilution effect of the fiber.[26]

As shown in Table III, the addition of rice straw pellet to HDPE resulted in a slight decrease of T_c

and X_c of the polymer matrix. This was possibly contributing to the dilution of the rice straw pellet during crystallization when compared with rice straw RF and strand. With regard to the melting temperature of the HDPE composites, the addition of the rice straw fiber in the polymer matrix caused a slight increase, and no essential correlation of the results with the rice straw fiber content and morphology could be established, as shown in Table III.

Scanning electron microscopy

The morphology of the fracture surface of the HDPE composites filled with 20 wt % rice straw fiber were presented in Figure 7(a–d). Figure 7(a) clearly showed the rice straw RF pullouts in the composites without MAPE. This indicated a reduction in interfacial bonding strength led to an increase in fiber pullout. From Figure 7(b), the rice straw RF was uniformly coated by layers of PE matrix. The addition of MAPE led to the formation of interfacial bonding between the HDPE and fiber, and the physical entanglement of PE molecule for the matrix and MAPE. From Figure 7(c,d), the rice straw pellet and strand could be embedded in the matrix. Furthermore, the fiber breakage was very obviously observed in composites filled with RF and strand, corresponding with higher aspect ratio.

CONCLUSIONS

The tensile, flexural, and impact mechanical properties and crystallization behavior of the rice straw fiber-HDPE composites have been investigated in this study. Rice straw fiber with different morphology produced by different manufacturing process have significantly influenced on mechanical properties of HDPE composites. Rice straw refined fiber had a wide range distributing in the length and width. Rice straw pellet was in a presence of homogeneous small pellet and rice straw strand had the largest size in the appearance morphology. Rice straw refined fiber and strand both had higher aspect ratio of 16.31 and 14.48 respectively. The use of higher aspect ratio rice straw fibers for increasing the tensile strengths, and lower aspect ratio rice straw fibers for increasing the flexural strength of the resultant composites. With increasing rice straw fiber from 20 to 40 wt % in HDPE matrix, the flexural strength of the composites increased slightly, while the impact strength decreased significantly due to the stiffness of the rice straw fiber. Adding MAPE to the system, the composites filled with 20 wt % rice straw fiber showed an increase in tensile, flexural and impact strength, and resulted in a slightly lower tensile elongation at break.

DSC results showed that adding rice straw fiber to the HDPE matrix did not changed in the crystallization temperature but decreased the crystallinity of HDPE. Fiber morphology affected the crystallinity of HDPE and the use of higher aspect ratio rice straw fiber for increasing the crystallinity. The SEM observation on the fracture surface of the composites indicated that introduction of MAPE to the system improved the interfacial bonding strength. Fiber breakage was more obviously occurred in rice straw refined fiber and strand than rice straw pellet.

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