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COMPARISON BETWEEN THE TENSILE, WATER ABSORPTION AND FLAMMABILITY PROPERTIES OF RECYCLED HIGH-DENSITY POLYETHYLENE/RICE HUSK COMPOSITE FROM TWIN-SCREW EXTRUDER AND HEATED TWO-ROLL MILL

This study compares the mechanical properties of recycled high-density polyethylene (r-HDPE)/rice husk (RH) composites from a twin-screw extruder and a heated two-roll mill, and the effect of different filler loadings using different melt blending processes on the mechanical properties of r-HDPE/RH composites. Polyethylene-graft-maleic anhydride (MAPE) acts as the coupling agent to enhance interfacial bonding between the fibre and the polymer matrix. The filler loading used was in the range of 10-40 wt. %. In this work, r-HDPE/RH blends were prepared using a twin-screw extruder and a heated two-roll mill. The ratio of 70/30 twin-screw extruder compounded composites significantly showed higher tensile based on improved to about 45.5% at 11 MPa compared to those compounded in the heated two-roll mill. The same ratio showed an increment almost up to 9% of elongation at break. It has also been verified that the higher filler loading used reduced the tensile strength and elongation at break, while the Young's modulus increased. The result was evidenced by the increase in water absorption and longer burning time as the filler loading increased.

Keywords: recycled high-density polyethylene; rice husk; twin-screw extruder; heated two-roll mill

1. Introduction

In this new era of technology, wood-plastic composites industry has grown significantly. This is due to their unique properties when combined together, apart from the desirable properties, cost advantage, and the request for economically-friendly materials. Wood-polymer composites have a wide range of applications, such as automotive, furniture, building, and other industries [1]. Wood fibre is usually famous for their lignocellulosic properties that offer several advantages, such as low density, low production cost, and renewability. Hence, the mixing of fibre increases the stiffness, hardness, and dimensional stability of plastics.

Rice husk (RH) is one of the commonly used natural fibres that has a potential as a reinforcing filler for thermoplastic composites. The presence of cellulose and hemicellulose in natural fibre will make them break down at a relatively low temperature [2]. However, RH has poor resistance to moisture as it absorbs moisture easily and leads to the decrease in mechanical performance. On the other hand, recycled high-density polyethylene (r-HDPE) can improve eco-efficiency and lower energy use as it is known as one of the easiest plastic polymers to be recycled. r-HDPE also provides high durability and good water resistance. The development of wood-polymer composites with the aims of utilising lignocellulosic waste and producing low-cost recycled plastic is desirable [3]. Polyethylene-graft-maleic anhydride (MAPE) is used as a coupling agent. Furthermore, MAPE is the most common coupling agent used as the compound is able to improve the mechanical properties of materials.

As compounding ingredients must be incorporated uniformly in the polymer matrix, an extruder offers continuous mixing through the melting and pumping which is efficient to

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process large quantities of materials, which can be completed in a shorter time duration and delivers a homogenised melt. Therefore, it requires minimal dependent and supervision of operators while protecting one's health and safety due to less machinery contact and chemical inhalation during the closed-chamber process. Meanwhile, a two-roll mill is the simplest batch of compression mill mixing equipment, where additives and filler can be continuously added during open mill processing in smaller batches. Thus, the second method needs a longer processing time as the manual blending must be repeated over time. However, the two-roll mill provides stronger shear ability to induce excellent mixing effects, hence could also obtained almost uniform dispersed blends equivalent to an extruder [4].

The objective of the work is to study the effect of different melt blending methods using a twin-screw extruder and a heated two-roll mill on the mechanical properties of r-HDPE/RH composites. Furthermore, the study was carried out to investigate the effect of different filler loadings using different melt blending methods on the mechanical properties of r-HDPE/RH composites.

2. Experimental

2.1. Methodology

This section discusses the materials used and the preparation of wood-polymer composites (r-HDPE/RH). RH was used as a filler and r-HDPE as a polymer matrix. Other than that, MAPE was added as a coupling agent. r-HDPE/RH composites were prepared using a twin-screw extruder and a heated two-roll mill, and then compressed using a compression moulding machine. Several tests were carried out, such as tensile test, water absorption test, and flammability test. The results were supported by Fourier transform infrared spectroscopy (FTIR) and scanning electron microscope (SEM).

2.2. Materials

RH, which is in brownish powder form with the density of 0.5-0.7 g/cm^3, was obtained from Biocomposite Extrusion Sdn. Bhd. The RH needs to be dried in an oven for 24 h at 100°C to completely remove humidity from the materials. r-HDPE with melt flow index of 3.24 g/10 min (200°C) and density of 0.923 g/cm^3 was also supplied from Biocomposite Extrusion Sdn. Bhd. It is in the form of white pellets. MAPE with the density of 0.92 g/cm^3 was supplied by Aldrich Chemistry Co. and in the form of white beads or pellets.

2.3. Methods

Table 1 shows the formulation of r-HDPE/RH composites. The compounding of the blends was carried out by melt blending in a twin-screw extruder supplied from Lab Tech Engineering Company LTD. using four different barrel temperatures (180, 190, 200, and 190°C). The screw speed was set to 60 rpm. The extrudate was collected, cooled, and ground into pellets. In addition, the compounding of the blends was also carried out by using a heated two-roll mill supplied by Fangyuan Instrument (DG) Co. LTD, model DW5110, No. 1191. The temperature used was 180°C to melt r-HDPE for 15 min. During melting, RH was added slowly. The amount of fibre loading used was 0, 10, 20, 30, and 40 wt. %. 3 wt. % of MAPE was also added into the blends and mixed together for 10 min. The sheets were then prepared as they were taken out after 2 min. Next, granulated composite pellets were moulded using a hot press machine (model Gotech GT-7014H-P30C).

Pellets were put into a mould with 1 mm thickness. The temperature of the hot press was set at 180°C for both upper and lower press, and the pressure used was 1,000 psi. The mould was preheated for 3 min, fully pressed for 7 min, and cooled for 3 min before being cut into a dumbbell shape.

Tensile test was conducted according to ASTM D 638-03 using the samples prepared. Tensile properties were measured at room temperature using Instron 5569 machine at 40 mm/min crosshead speed. Water absorption test was conducted according to ASTM D 570-98. The samples were dried in an oven for 24 h at 100°C. Then, the samples were weighed using an analytical balance with the precision of 1 mg. The water absorbed was determined by immersion of the samples in distilled water at room temperature for 15 days and the increase of weight was measured. The amount of water absorbed by the composites was calculated according to the following equation:

$$\text{Water Absorption}\,(\%) = \\ = \frac{\text{Final Weight} - \text{Original Weight}}{\text{Original Weight}} \times 100\%$$

Flammability test was carried out to measure the properties of the composites in response to heat and flame under controlled laboratory conditions. The test results represent flaming plus glowing time in seconds under the conditions of the test, in which ASTM D 5078-90 (Procedure B: Test of plaque specimens) was followed. Ignition time, smoke colour, and dripping of materials during combustion were recorded. Next, Fourier transform infrared spectroscopy (FTIR) was performed by using a Perkin-Elmer 2000 spectrometer and attenuated total reflection (ATR) method was used to identify the functional groups present in the samples. ATR was used for strong absorption of thick samples, which often produced intense peaks when measured by transmission. Lastly, an SEM (model Hitachi TM3000 Tabletop Microscope) that used a beam of highly energetic electron was utilised in this study to examine objects on a very fine scale. The SEM was used to check the topography, morphology, and composition of fracture samples. The samples were coated using platinum to improve the imaging of the samples, and every sample was observed in 200× and 500× magnification to observe the fracture surface of the samples.

TABLE 1

Formulation of r-HDPE/RH composites

Process	Recycled High-Density Polyethylene (r-HDPE)	Rice Husk (RH)	Polyethylene-Graft-Maleic Anhydride (MAPE)
Twin-Screw Extruder	100	0	0
	90	10	3
	80	20	3
	70	30	3
	60	40	3
Heated Two-Roll Mill	100	0	0
	90	10	3
	80	20	3
	70	30	3
	60	40	3

3. Results and discussion

3.1. Tensile Properties

3.1.1. Tensile strength

The tensile strength of r-HDPE/RH composites is shown in Figure 1. It can be seen that all blends in both twin-screw extruder and heated two-roll mill showed significantly decreased tensile strength with the increase of filler loading. This is due to the presence of RH that influenced the crystalline structure of r-HDPE as RH has amorphous characteristics. The increase in RH loading blocked the ordered arrangement of r-HDPE and reduced the crystallinity, which reduced the tensile strength value [5]. A similar result was also observed by Yang et al. [6] who studied the properties of lignocellulosic material filled with polypropylene bio-composites made from different manufacturing processes. In addition, the tensile strength of r-HDPE blend is higher for the heated two-roll mill in the ratios of 90/10 and 80/20 but demonstrated lower tensile strength for the ratios of 70/30 and 60/40 compared to the twin-screw extruder. As

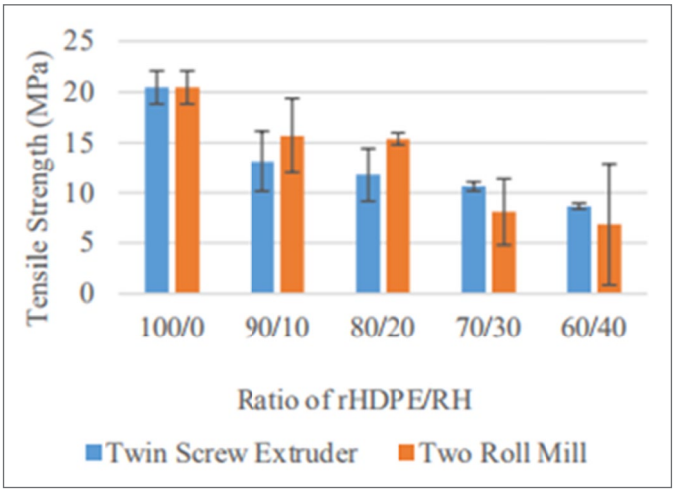

Fig. 1. Tensile strength of r-HDPE/RH composites

the filler increases, the composite produced using the heated two-roll mill will go through difficulty of the process due to non-homogeneous of the composite that resulted in decreased tensile strength. The reduction in tensile strength might also be resulted from the molecular structure of materials, which has a great impact on the tensile strength of the materials [7]. Molecular structure responsible for the intermolecular forces is formed in the material. These intermolecular forces are responsible for binding different molecules of the materials together, hence whenever there is a change in the molecular structure, the tensile strength of the material differs greatly [8].

3.1.2. Elongation at break

Figure 2 displays the elongation at break of r-HDPE/RH from a twin-screw extruder and a heated two-roll mill. From the figure, the elongation at break is higher in the twin-screw extruder compared to the heated two-roll mill. r-HDPE/RH in the ratio of 70/30 from the twin-screw extruder has the highest value of elongation at break, whereas the sample with similar blend ratio from the heated two-roll has the lowest value of elongation at break. Incorporation of the filler resulted in the abrupt drop of elongation at break compared to the r-HDPE matrix. Elongation at break was seen to diminish with RH concentration [9]. Moreover, the addition of a coupling agent increased the interlocking between two phases, thus increased the interfacial adhesion between the phases in polymer state. This would lead to low mobility of the chains of r-HDPE/RH blend interface and reduced the elongation at break [10]. Besides, pure r-HDPE has higher elongation at break, which indicates that the material is of high ductility.

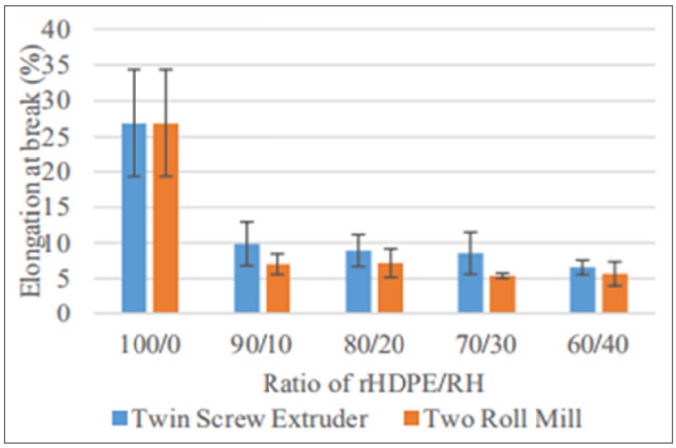

Fig. 2. Elongation at break of r-HDPE/RH composites

3.1.3. Young's modulus

Figure 3 shows the Young's modulus of r-HDPE/RH from a twin-screw extruder and a heated two-roll mill. Based on the figure, r-HDPE/RH in the ratio of 80/20 using different blending methods has similar Young's modulus values. Meanwhile,

r-HDPE/RH in the ratio of 60/40 from the heated two-roll mill has the highest value of Young's modulus, whereas r-HDPE/RH with a similar blend composition from the twin-screw extruder has the lowest value of Young's modulus. This indicates that lower filler loading makes the composites stiffer when processed using the heated two-roll mill compared to the twin-screw extruder. As the filler loading increased, the degree of obstruction increased, which in turn increased the stiffness. This statement is supported by Rahman et al. [11]. In addition, the incorporation of fibre into the polymer matrix reduced matrix mobility, resulting in the stiffness of the composites. As a result, Young's modulus increased with the increase of filler content of the composites [12].

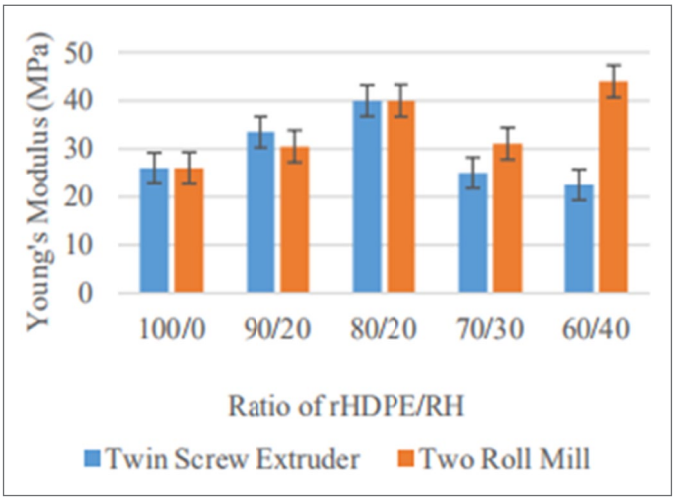

Fig. 3. Young's modulus of rHDPE/RH composites

3.2. Water Absorption Behaviour

Water absorption behaviour of the composites at room temperature for different filler loadings is shown in Figure 4. It is shown that r-HDPE/RH from the heated two-roll mill has higher water absorption compared to the twin-screw extruder. As the filler loading increased, water uptake from the composites increased. The ratio of 60/40 two-roll mill showed the highest percentage of water absorption due to poor interfacial adhesion of matrix and fibre, as depicted in Figure 7(c), (d) and Figure 8(c), (d). The non-homogeneous compounding during the process leads to poor bonding of these two materials. Thus, the OH group inside the rice husk actively absorbed water [13]. Different blending methods give different results for water absorption. By using the heated two-roll mill, the blending is more affected by the environmental condition. The roll mill provides a rapid release of volatiles during the mixing and at the same time allows oxygen from the air to affect the melt. The degree of shear stress is considerably lower for the two-roll mill than in the twin-screw extruder; therefore, it can be assumed that the degree of homogenisation of the composites will also be smaller [14]. Thus, the result for water absorption is better for the twin-screw extruder as two-roll milling causes more severe damage to the fibres and leads to a narrower fibre distribution than twin-screw extrusion [15]. It is proven in the figure that water absorption increased with an increase in filler loading. The results after 15 days of immersion are presented in Table 2. The presence of natural fibre increased water absorption of the composites. Natural fibre has a hydrophilic nature that lowers the compatibility with hydrophobic polymeric matrices. Cellulose fibres contain many hydroxyl groups (-OH) and readily interact with water molecules by hydrogen bonding [16].

TABLE 2

Data collected for water absorption for 15 days of immersion

Process	r-HDPE/RH	Original Weight (g)	Final Weight (g)	Water Absorption (%)
Twin-Screw Extruder	100/0	0.4702	0.472	0.38
	90/10	0.486	0.4925	1.34
	80/20	0.5248	0.5363	2.19
	70/30	0.633	0.66	4.27
	60/40	0.5946	0.6224	4.68
Heated Two-Roll Mill	100/0	0.4702	0.472	0.38
	90/10	0.523	0.5377	2.81
	80/20	0.5098	0.5323	4.41
	70/30	0.5665	0.5963	5.26
	60/40	0.5542	0.5935	7.09

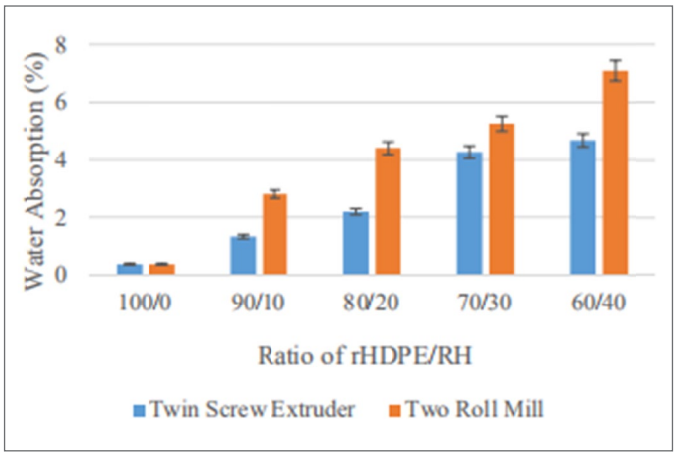

Fig. 4. Water absorption of r-HDPE/RH composites

3.3. Flammability Test

Table 3 shows the flame characteristics and ignition time of r-HDPE/RH from a twin-screw extruder and a heated two-roll mill. Based on the comparison between different melt blending methods, it is proven that by using the heated two-roll mill, the ignition time is slower compared to the composites produced using the twin-screw extruder. It can be concluded that the composites from the heated two-roll mill are more flame resistant compared to the twin-screw extruder. It is also noted that the presence of RH filler in the composites released a lot of black smoke during burning, and dripping also occurred. On the other

hand, pure r-HDPE dripped badly and released only a little black smoke during combustion. As predicted, the burning of all RH containing composites produced a lot of smoke. The presence of high lignin content (about 26%-31%) from the use of raw RH as the filler in composites can be related to this phenomenon [17]. Figure 5 shows the ignition time for r-HDPE/RH from a twin-screw extruder and a heated two-roll mill. The most significant result was exhibited through the 80/20 ignition time using twin-screw extruder. This might be influenced by the exploitation of v-orientation of fibre, which later resulted in the highest ignition time [18,19]. Hence, the results proved that the higher filler content, the longer the ignition time [20]. It is believed that with the presence of RH in composites, the flammability resistance increased with the increase of RH filler content from 10 wt. % to 40 wt. %. This improvement is assigned to the nature of silica (15%-17% in raw RH) that slows down burning [21].

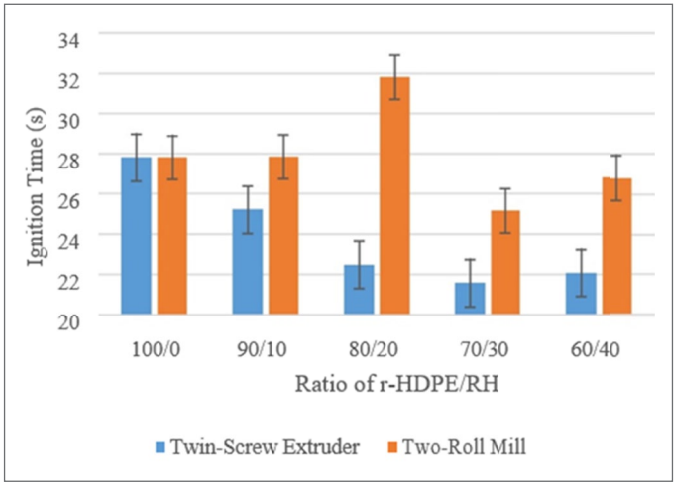

Fig. 5. Ignition time of r-HDPE/RH composites

TABLE 3

Flammability characteristics of r-HDPE/RH using different melt blending methods

Process	r-HDPE/R	Ignition Time (s)	Smoke Colour	Drip
	H			
Twin-Screw Extruder	100/0	27.8	Produce light black smoke	Drip
	90/10	25.1	Produce heavy black smoke	Drip
	80/20	22.48	Produce heavy black smoke	Drip
	70/30	21.58	Produce heavy black smoke	Drip
	60/40	22.07	Produce heavy black smoke	Drip
Heated Two-Roll Mill	100/0	27.8	Produce light black smoke	Drip
	90/10	27.84	Produce heavy black smoke	Drip
	80/20	31.81	Produce heavy black smoke	Drip
	70/30	25.15	Produce heavy black smoke	Drip
	60/40	26.81	Produce heavy black smoke	Drip

3.4. Fourier Transform Infrared Spectroscopy

Figure 6 displays the FTIR spectra of r-HDPE/RH in the ratios of 80/20 and 60/40 from a twin-screw extruder and a heated two-roll mill. There is a notable variation in the band at 3300-3400 cm^{-1}, which represents the stretching vibration of intermolecular hydrogen bonded –OH groups in cellulose fibre. A broader peak at 3310 cm^{-1} indicates the presence of stretch hydroxyl group on the RH surface. The presence of this band is due to the stretching vibration of intermolecular hydrogen bonded –OH groups in cellulose fibre [22]. The sharp and strong stretching frequency from 2850 to 3000 cm^{-1} exhibited $–CH_2$ symmetric stretching from r-HDPE. Besides, no visible peak around 2300-2500 cm^{-1} is observed, which represents CO_2 in r-HDPE/RH in the ratio of 80/20 using the twin-screw extruder

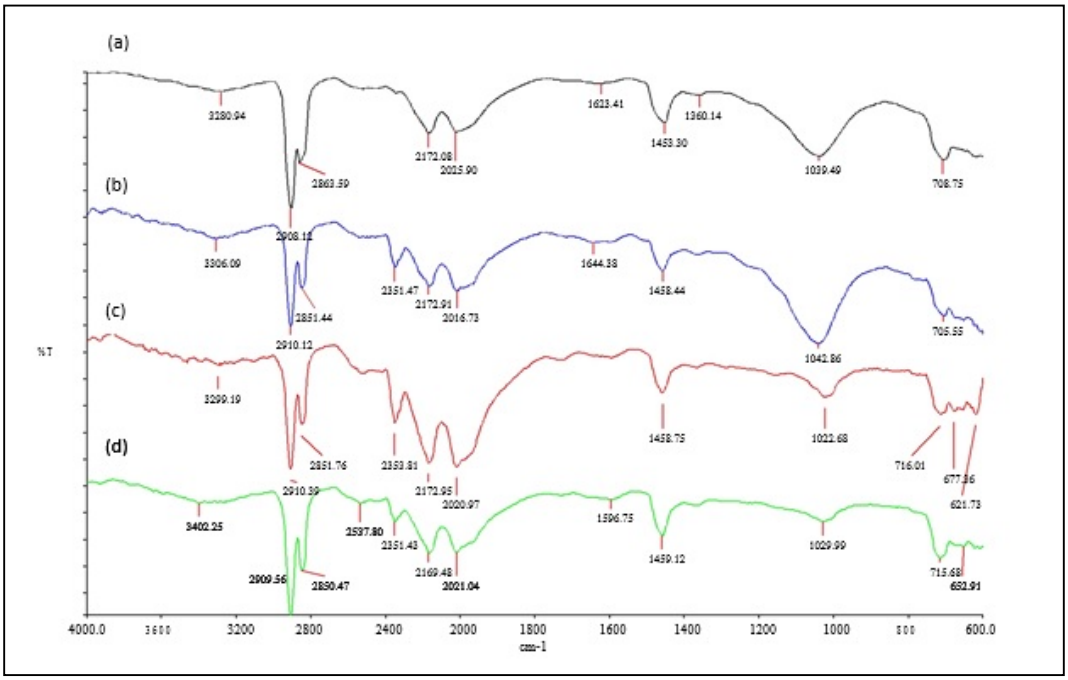

Fig. 6. FTIR spectra of (a) 80r-HDPE20RH TSE, (b) 60r-HDPE40RH TSE, (c) 80r-HDPE20RH TRM, and (d) 60r-HDPE40RH TRM

compared to other spectra, and this indicates that no r-HDPE is present in the blend. This shows that the interaction mixed well between RH and MAPE for the twin-screw extruder compared to the heated two-roll mill [23]. In average 1600 cm^{-1}, carbonyl peak C = C slightly shifted because the carbonyl bonds in hemicellulose are broken. The spectra revealed –CH2 bending deformation in the range of 1450-1460 cm^{-1}. The band at 1030 cm^{-1} is attributed to the stretching vibration of C-O of lignocellulose and Si-O-Si of RH [22]. All bands around 700-900 cm^{-1} are also assigned to C-H out-of-plane bending vibration.

3.5. Scanning Electron Microscope

The surface fracture of r-HDPE/RH in the ratios of 90/10 and 60/40 from a twin-screw extruder for 200× and 500× magnification is displayed in Figure 7. From observation, there is no clear gap between r-HDPE and RH for both compositions. This indicates good interfacial bonding between the fibre and the matrix. The fibres are perfectly bonded to the matrix and it is also evident that the fibres are strongly immersed in the matrix. This shows that the mixing efficiency attributes to a good interface between RH fibres and matrix [24].

Figure 8 presents the surface fracture of r-HDPE/RH in the ratios of 90/10 and 60/40 from a heated two-roll mill for 200× and 500× magnification. As observed, the pulled-out fibres present for the ratio of 60/40 proved that r-HDPE and RH have poor interfacial adhesion, which leads to the loss in mechanical properties. During fracture, it is possible that wood fibres are weakly attached to the polymer matrix, thus are pulled out from the matrix [25]. However, the fracture surface for the ratio of 90/10 shows that the fibres are perfectly attached to the matrix. During increasing the fibre loading using a two-roll mill, compounding becomes tense and ultimately hard to blend properly, causing a non-homogeneous composite surface. This is obviously exhibited in the respective SEM result, where a two-roll mill of 60/40 showing a large gap between the matrix and fibre. Thus, this proves that the application of a two-roll mill resulted in the lowest tensile strength as compared to other formulations [19]. The observed morphology confirms the explanation that blending

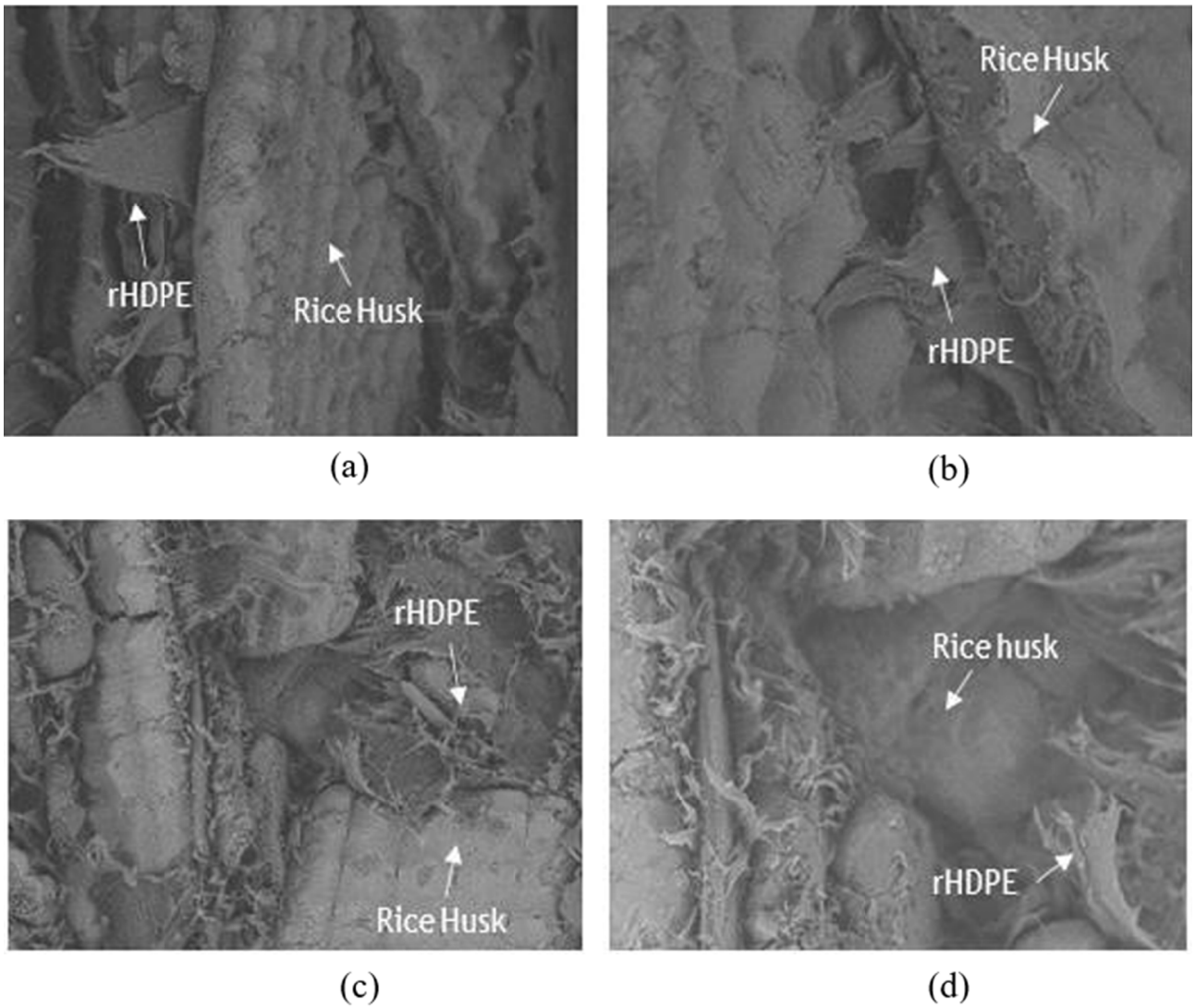

Fig. 7. SEM micrographs of the surface of r-HDPE/RH in the ratio of 90/10 for (a) 200× magnification and (b) 500× magnification, and the surface of r-HDPE/RH in the ratio of 60/40 for (c) 200× magnification and (d) 500× magnification using a twin-screw extruder

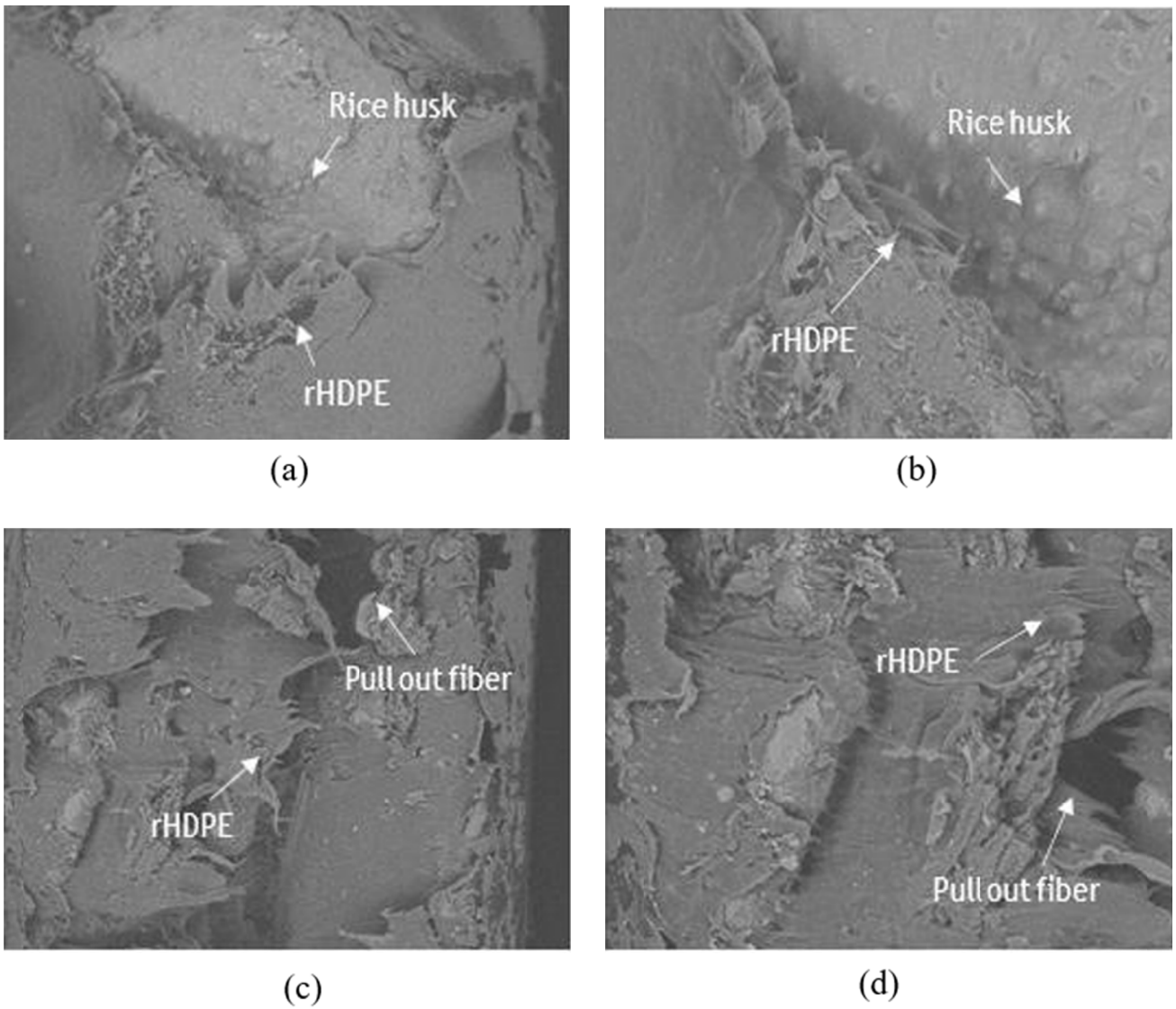

Fig. 8. SEM micrographs of the surface of r-HDPE/RH in the ratio of 90/10 for (a) 200× magnification and (b) 500× magnification, and the surface of r-HDPE/RH in the ratio of 60/40 for (c) 200× magnification and (d) 500× magnification using a heated two-roll mill

using the twin-screw extruder is better than the heated two-roll mill for the lower degree of water absorption and the higher mechanical properties of r-HDPE/RH composites.

4. Conclusion

In conclusion, the increase of filler loading will reduce tensile strength, especially for the heated two-roll mill. The interfacial bonding between the filler and the matrix reduced as the filler content increased which is influenced by the presence of agglomeration that generates flaws and creates voids between the filler and the polymer matrix. Other than that, r-HDPE shows higher elongation at break, which indicates that r-HDPE has high ductility. However, as the filler is added, the ductility of composites reduced. For Young's modulus test, r-HDPE/RH in the ratio of 80/20 shows a similar value for both blending methods. The lower filler loading makes the composites stiffer, especially when the mixing is conducted using the heated two-roll mill. The increase of filler loading increases the degree of obstruction and reduces the matrix mobility, which further increases the stiffness of composites. In the observation of water absorption behaviour of r-HDPE/RH composites, the result shows that the composites with higher filler loadings absorbed more water as RH filler has a hydrophilic nature. The flammability test proved that higher filler content increased the ignition time as the flame resistance increased with the increase of filler due to the silica content in raw RH that slows down combustion. In addition, the presence of RH in composites released a lot of black smoke and dripping during burning. To summarise, the mixing using the twin-screw extruder shows better mechanical properties of r-HDPE/RH composites compared to the heated two-roll mill. The result shows a slight difference between both melt blending methods. For the effect of different filler loadings, higher filler loading reduces the tensile strength and elongation at break, hence increases the stiffness of blends. This result is clearly supported by the increase of water uptake and longer ignition time as filler loading increases.

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