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Effect of Rice Husk Surface Modification by LENR the on Mechanical Properties of NR/HDPE Reinforced Rice Husk Composite

(Kesan Rawatan Permukaan Sekam Padi dengan LENR ke Atas Sifat Mekanik Komposit NR/HDPE diperkuat Sekam Padi)

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ABSTRACT

Surface modification of rice husk (RH) with alkali pre-treatment (NaOH solution 5% w/v) was carried out at the initial state to investigate the effect of surface treatment of fibre on the surface interaction between fibre and rubber. Further modification of RH surfaces after alkali treatment was using Liquid Epoxidized Natural Rubber (LENR) coating at three concentrations, 5%, 10%, and 20% wt LENR solution in toluene. Interfacial morphology and chemical reactions between RH fibre and rubber were analyzed by FTIR and Scanning Electron Microscope (SEM). It was found that 10% wt LENR solution gave the optimum interaction between fibre and rubber. Matrix and composite blends derived from 60% natural rubber (NR), 40% high density polyethylene (HDPE) reinforced with RH fibre were prepared using an internal mixer (Brabender Plasticoder). Result showed that pre-treatment of RH treated with 5% NaOH followed by treatment with 10% LENR solution given the maximum interaction between fibre and matrix that gave rise to better mechanical properties of the composites.

Keywords: High density polyethylene; liquid epoxidized natural rubber; mechanical properties; rice husks; sodium hydroxide

ABSTRAK

Modifikasi permukaan sekam padi (SP) dengan pra-rawatan alkali (larutan NaOH 5% w/v) telah dijalankan untuk mengkaji keberkesanan rawatan permukaan SP ke atas interaksi permukaan antara serabut dan getah. Modifikasi permukaan SP seterusnya adalah dengan penyalutan getah asli terepoksida cecair (LENR) pada tiga kepekatan yang berbeza iaitu 5%, 10% dan 20% w/w LENR dalam larutan toluena. Morfologi antaramuka dan reaksi kimia antara SP dan getah (LENR) dikaji melalui analisis FTIR dan mikroskop elektron imbasan (SEM). Didapati bahawa larutan LENR 10% memberikan interaksi yang optimum antara serabut dan getah (LENR). Adunan matriks dan komposit disediakan daripada 60% getah asli (NR), 40% polietilena berketumpatan tinggi (HDPE) diperkuat dengan pengisi SP di jalankan dengan menggunakan pengadun dalaman (Brabender Plasticoder). Keputusan kajian menunjukkan bahawa pra-rawatan SP dengan larutan NaOH 5% diikuti dengan rawatan LENR 10% memberikan interaksi maksimum antara pengisi dan matriks seterusnya meningkatkan sifat mekanik kommposit

Kata kunci: Getah asli terepoksida cecair; natrium hidroksida (NaOH); polietilena berketumpatan tinggi; sekam padi; sifat mekanik

INTRODUCTION

Biodegradable polymers are designed to be degraded by living organisms after the useful life. They offer an alternative to traditional non-degradable polymers when recycling is not economical or impractical, and they can be composted together with food, home and yard waste (Bastioli 1987). The increasing interest in the use of biodegradable materials has led to the use of natural fibres as reinforcing filler with the aim to improve the thermo-mechanical properties of composite materials. Rice husk, flax fibres, palm fibres and many more are the typical fibres which have been widely studied due to their suitability as filler in polymer composites.

Rice husk (RH) is a cellulosic-based fibre, which has been widely utilized in the manufacture of composite panels (Ndazi 2001; Vasishth 1971). The main advantages of using RH as biodegradable filler are their low cost, low density, high specific strength and modulus, and recyclability (Ismail et al. 2001; Ndazi 2001). It has been shown that the use of RH in natural rubber/ linear low-density polyethylene composite results in an increase in tensile modulus and hardness (Mwaikambo & Ansell 1999). The main disadvantage of using RH as reinforcing filler for composite material is the poor interaction between rice husk particle and polymer matrix (Avella et al. 1998). Compatibility of cellulosic fibres with non-polar matrices which are mostly hydrophobic is problematic due to high polarity and hydrophilicity in nature of cellulosic fibres. Therefore, modification of the surface of natural cellulosic fibres is critically important to increase the hydrophobicity of the fibres and enhances the adhesion with polymer matrix. Hence, modification of natural fibre surface represents the natural process used to increase the hydrophobic character or decrease the hydrophilicity of the fibres. The adhesion of RH to the matrix can be improved on minimizing the surface tension.

Surface modification of cellulosic fibres involves removal of impurities from the fibre surface to enhance its physical and chemical properties. Alkaline treatment of cellulosic fibres with sodium hydroxide (NaOH) is a well known method which has been employed to improve the fibre-matrix interface bonding (Mohanty et al. 2000). NaOH removes natural fats and waxes from cellulosic fibre surfaces and thus exposing chemically reactive functional groups such as hydroxyl (–OH) and other reactive functional groups. The removal of the surface impurities from the cellulose fibres also improves the surface roughness of the fibres (George et al. 2001). LENR is the preferred compatibilizer due to its co-polymeric in nature, example polyisoprene with regular epoxies groups at the internal. The polar epoxies group is compatible with the rice husk fibre and isoprene as matrix.

The main objective of this study was to investigate the effect of alkaline treatment of rice husk fibres and coating with LENR on the mechanical properties of NR/HDPE-RH composite. The coating method of the fibres with LENR is crucial as it may lead to agglomeration of fibres. Coating rice husk with LENR helps to enhance hydrophobic character and thus improve the interfacial compatibility between fibre and matrix, enhancing the mechanical properties of the composites.

Materials and Methods

MATERIALS

Natural rubber (NR) grade SMR-L with a density 0.92 g/cm^3, was supplied by Rubber Research Institute of Malaysia (RRIM) and high density polyethylene (HDPE) with a density 0.94 g/cm^3 was supplied by National Petrochemical Public Company Limited, Thailand. Rice husk obtained from Bernas, Selangor, Malaysia. LENR, was supplied by Guthrie Berhad Group, was prepared by photochemical degradation of ENR-50, supplied by Guthrie Berhad Group. Technique of photochemical rubber degradation was patented by UKM (Ibrahim 1996).

PREPARATION OF RICE HUSK FILLER

The rice husk obtained from a local rice milling plant was air-dried and then ground and sieved to fine particles using 150-200 μm siever. The rice husk particles were further modified with alkali treatment and LENR coating.

FIBRE SURFACE TREATMENTS

The nomenclature for the different rice husk (RH) surface treatments used is shown in Table 1. In order to improve the adhesion between the rice husk filler and matrix, the rice husk fibres were subjected to one or several surface treatments which will be described later.

TABLE 1. Nomenclature used for the different fibre surface treatments

Keyword	Description
Rhi	RH without treatment
RHiNa	RH treated with 5% NaOH aqueous solution
RhiNaLE 1	RH treated with 5% NaOH aqueous solution and then coated with 5% LENR in toluene solution
RhiNaLE 2	RH treated with 5% NaOH aqueous solution and then coated with 10% LENR in toluene solution
RhiNaLE 3	RH treated with 5% NaOH aqueous solution and then coated with 20% LENR in toluene solution

ALKALI TREATMENT

Treatment with NaOH was carried out by soaking and stirring the RH fibres in 5% wt/v NaOH aqueous solution for about 3 hour at room temperature, then RH was filtered out and washed several times with distilled water until all the sodium hydroxide was eliminated, that is, until the water no longer indicated any alkalinity reaction before being dried up in an oven at 60°C for 24 hours. Caustic soda treatment was chosen because it is inexpensive and effective.

LENR COATING

After drying, the alkali treated rice husk fibres was ground to break the agglomeration. RH was then stirred using mechanical stirrer in 5%, 10% and 20% LENR solution in toluene for about 1 h at 25°C. The mixture was then vibrated using an ultrasonic vibrator machine for 30 min. This surface modified procedure of RH using mechanical and ultrasonic vibrator was aimed to make the rice husk powder surface optimally coated by LENR. Finally, the mixture was filtered and dried in an oven at 60°C for about two weeks.

PREPARATION OF COMPOSITE BOARDS

Matrix NR/HDPE blend was prepared using an internal mixer (Brabender Plasticoder) with composition ratio 60:40 v/v at 135°C (for 12 min) and mixing rate at 50 rpm. NR/HDPE/RH composite was prepared with composition at 90% wt/v matrix NR/HDPE and 10% wt/v rice husk for 12 min at 135°C, by progressively increasing the mixing speed up to 45 rpm. Finally, the blends were compression molded at 135°C for 7 min to produce composite specimens (thickness 1 mm and 3 mm width) for further mechanical testing.

CHARACTERIZATIONS

Surface morphology of the rice husk before and after modification, also fractured surface of the composite specimens after tensile test were studied using a scanning electron microscope (SEM, Philip XL 30).

The tensile test was carried out using the Universal Testing Machine (INSTRON 5560) following the standard ASTM D412 98a with a load cell of 1kN. Sample was cut into 'dumbell' shape using Hollow Die Punch following BS6746 standard. Mechanical properties of composite like tensile strength and modulus were recorded. Izod impact strength was measured on a Zwick testing machine operating with a 7.5 J pendulum (ASTM D256). Hardness of the sample was tested using Shore D type Zwick materials testing 3100. A minimum of five samples was tested in each series.

RESULTS AND DISCUSSION

FTIR SPECTRA ANALYSIS

Figure 1(a) shows several peaks at 1251, 1063 and 875 cm^{-1} which are all attributable to the epoxide group (Dahlan & Ghani 1993). There is another broad peak at 3412 cm^{-1} due to the hydroxyl group. Depolymerisation of the rubber also shows the formation of hydroxyl group (Dahlan & Ghani 1993). The FTIR peaks are baseline corrected to 3429 cm^{-1} (see Figure1(b)). The CH stretch at 2923 cm^{-1} is present in all fibres. The carbonyl peak at 1728 cm^{-1} can be seen in all the fibres not treated with alkali represent to hemicelluloses and also C=C stretching peak at 1513 cm^{-1} from lignin was removed after alkali treatment. Disappearance of carbonyl peak represents hemicelluloses was further evidence that hemicelluloses were removed from fibre surface during the alkali treatment (Mohanty et al. 2005). FTIR spectra of the rice husk treated with alkali and LENR treatments, is indicated in the 4000-400 cm^{-1} as shown in Figure 1. It can be seen that there are no significant changes of peaks between IR spectrums of untreated rice husk (Figure 1(b)) and IR spectrum of rice husk treated with 5% NaOH (Figure 1(c)). The peak at 1644 cm^{-1} and 1641 cm^{-1} is attributed to water absorbed, showed decrease toward alkali treatment and LENR coating. IR spectrums of rice husk after treated with 5% NaOH followed by 10% LENR coating (Figure 1(d)) shows significant changes from peak 1053 cm^{-1} to 1021 cm^{-1}. Broaden peaks 1021 cm^{-1} suggest that there were physical interaction between cellulose groups with epoxy groups from LENR during the LENR coating.

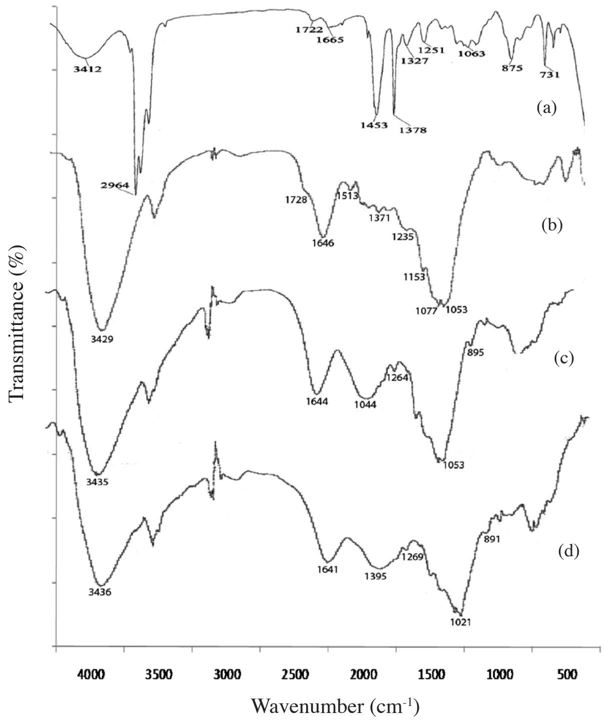

FIGURE 1. FTIR spectra of : (a) LENR, (b) RHi, (c) RHiNa, and (d) RH treated with 5% NaOH-10% LENR (RHiNaLE 2)

TABLE 2. IR data of LENR and RH

Wavenumber (cm^{-1})[a] LENR	Functional groups	Wavenumber (cm^{-1})[a] RH	Functional groups
3412 (m)	OH stretch	3700-3200 (m)	OH stretch.
2964 (s)	C-H stretch	3000-2850 (w)	C-H stretch
1722 (s)	C=O stretch. (from carbonyl groups)	1740-1720 (s)	C=O stretch.(ester & carboxylic group from hemicelluloses)
1665 (m)	C=C of cis-1,4	1680-1600 (m)	C=C (water absorbed)
1453 (m)	$-CH_2-$ deform. mode	1600-1475 (w)	C=C stretch (aromatic rings from lignin)
1378 (m)	$-CH_3$ deform. mode	1450-1375 (m)	C-O-H bending (ester)
1251 (s)	C-O stretch (from epoxy)	1300-1000 (s)	C-O stretch. (eter)
1063 (s)	C-O stretch (from cyclic eter)	900-690 (m)	C-H bending
875 (m)	C-O stretch (from epoxy)	700-400 (m)	C-C stretch

[a] s: strong, m: moderate, w: weak

SCANNING ELECTRON MICROGRAPHS

Figure 2(a) shows the SEM micrograph of untreated and treated rice husk. Untreated rice husk (Figure 2(a)), possesses smooth, flat and cloudy surface due to the presence of lignin, hemi-cellulose and wax (Mohanty et al. 2005). Figure 2(b) shows rice husk treated with 5% NaOH (RHiNa) exhibits rough surface. Cellulose micro fibrils of RH was exposed due to dissolution of lignin, hemicelluloses and other impurities dissolve in alkaline solution. The rough surface of alkaline treatment would make the mechanical interlocking between RH and rubber from LENR solution favourable. Figure 2(c) shows SEM micrograph of rice husk treated with 5% NaOH followed by 5% LENR coating (RHiNaLE 1). As shown in that figure, the grey and shining colour of rice husk surface was assumed to be the LENR that coated the surfaces of rice husk. The surface of rice husk treated with 5% NaOH followed by 10% LENR coating (RHiNaLE 2) as shown in Figure 2(d) were smoother than RHiNaLE 1 due to better dispersion of LENR on the surfaces of the rice husk. This indicated that LENR was able to fully penetrate into the roughness and fibrillation of RH fibres. Figure 2(e) shows SEM micrograph of rice husk treated with 5% NaOH followed by 20% LENR coating (RHiNaLE 3). The surface was smooth and flat because porous and holes on the rough surface of RH as result of alkali treatment was completely coated by LENR. Unfortunately, this rubber content seems too overload, which could probably lead to agglomeration of RH fibres when blending with polymer matrices.

MECHANICAL PROPERTIES

Figures 3 to 5 show the mechanical properties of the TPNR (60/40 NR/HDPE) and TPNR/NR (90/10) composites with different surface treatment of RH. Generally, addition of filler seems to decrease the tensile stress as well as impact strength of composites due to the incompatibility between filler particles and matrix, while the tensile modulus

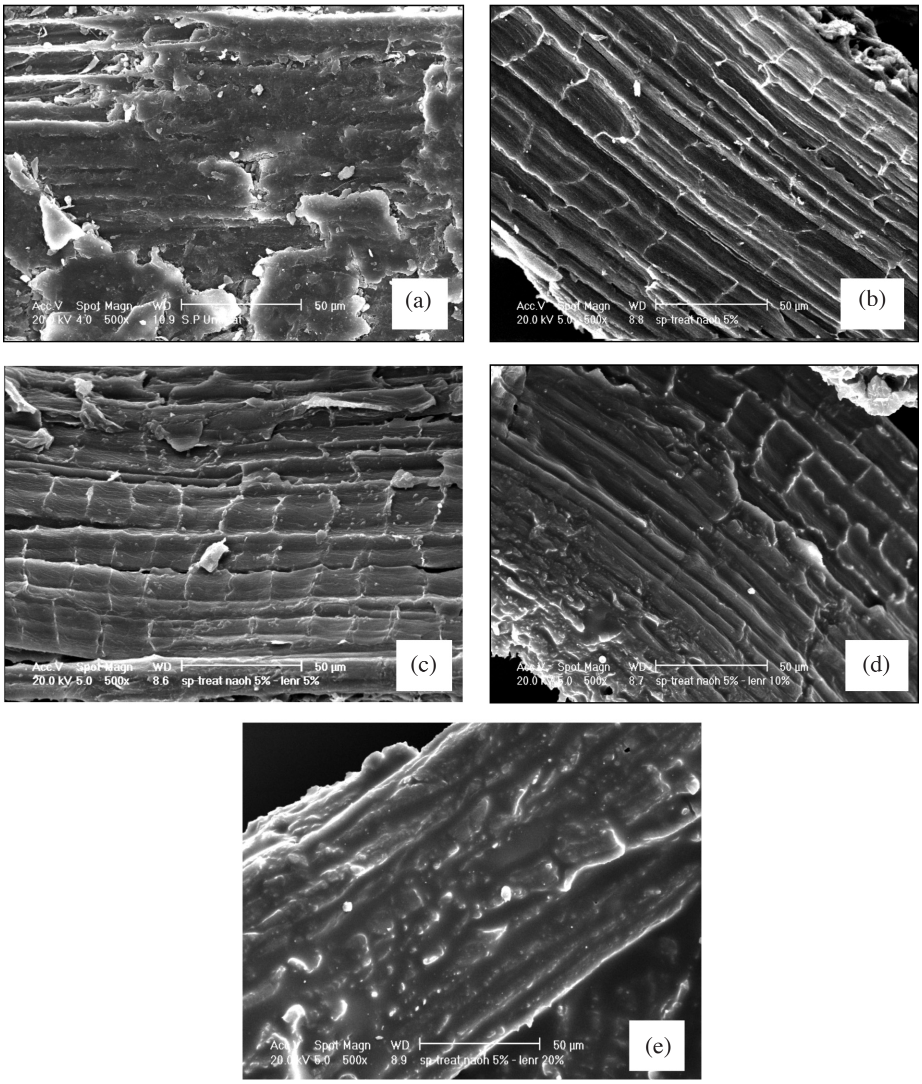

FIGURE 2. SEM micrographs of RH (a) RHi, (b) RHiNa, (c) RHiNaLE 1, (d) RHiNaLE 2 and (e) RHiNaLE 3

of composites increases with the addition of rigid filler particles.

Figure 3 shows the effect of addition of untreated rice husk filler with 10 wt% into NR/HDPE (60/40) matrix. The tensile strength decreased due to agglomeration of filler and hydrophylic properties that caused lack of homogeneity of composite. After treatment with 5% NaOH, tensile strength increased due to removal of impurities from the rice husk surface like lignin, hemi-cellulose, fats and waxes was removed thus revealing chemically reactive functional groups like –OH. Alkali treatment gave the rough surface on RH filler that can increase the interfacial interaction between filler and matrix, as can be seen in Figure 2(b). Further treatment with LENR compatibilizing agents was carried out to promote the adhesion or enhance the mechanical interlocking. However, TPNR/RHiNaLE 3 composite with LENR 20% wt/v content showed decrease in tensile strength, which was probably due to rubber coating on the RH surface was overload and led to agglomeration of RH fibre within the composite. From the graph plotted, it can be seen that TPNR/RHiNaLE 2 gives the highest tensile strength compared to untreated and other RH treatment. It can be concluded that the optimum LENR content for coating RH surface was 10% wt/v in toluene solution.

Figure 4 shows tensile modulus of composites with addition of RH at different surface modification. It can be seen that the composite filled with untreated rice husk increased the tensile modulus compared to composite NR/HDPE due to the increase of stiffness of composite as well. The RH fibres was treated with NaOH to remove lignin, pectin, waxy substances, and natural oils covering the surface of the fibre cell wall. This makes the surface of the fibre rough by revealing the fibrils (Valadez-Gonzales et al. 1999). Consequently, this change in fibre morphology may create more voids for water to absorb that can lead to agglomeration of RH filler. Another reason for the

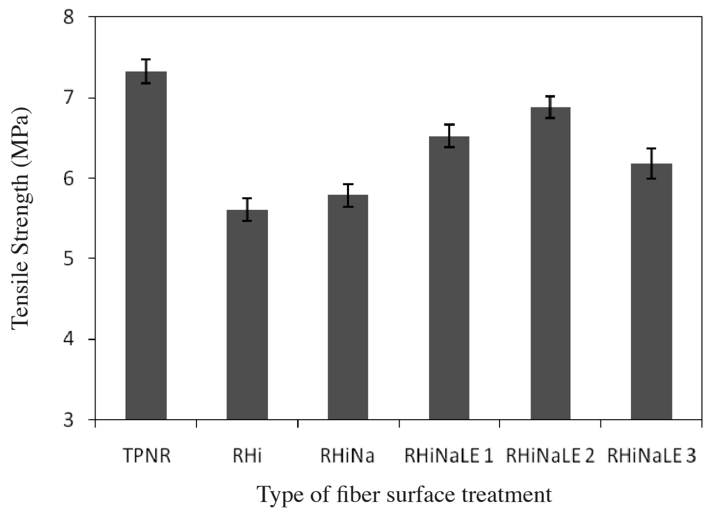

FIGURE 3. Tensile strength of TPNR and (NR/HDPE)/RH (90:10 v/v) composite, plotted as a function of RH surface treatment

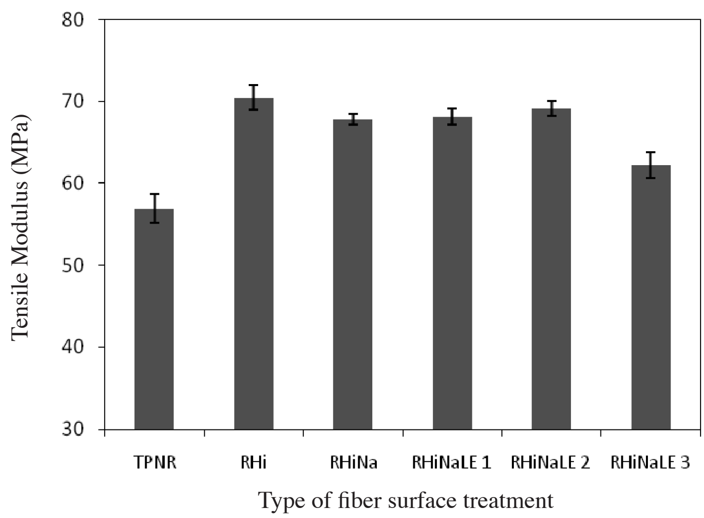

FIGURE 4. Tensile modulus of TPNR and (NR/HDPE)/RH (90:10 v/v) composite, plotted as a function of RH surface treatment

decrease of tensile modulus was due to the time taken for alkali treatment was too long that could possibly reduce the rigidity of RH fibre. It can be said that chemical and physical degradation that took place in NaOH treated rice husks and caused significant deterioration of particles stiffness (Ndazi et al. 2007). The presence of LENR as a surface modifier of rice husk filler decreased the tensile modulus compared to composite filled with untreated RH. Compared to rice husk treated with NaOH alone, rice husk treated with NaOH as pre-treatment followed by LENR coating (RHiNaLE) gave higher tensile modulus. LENR coating on the RH surface limits water absorption. It was due to hydrophobicity of rice husk filler that led to maximum interaction and compatibility between RH filler and matrix. This consequently increased the mechanical properties of the composite.

Figure 5 shows the impact strength of composite. The impact strengths of the composites decreased slightly as the RH filler was added into it (TPNR/RHi), due to the poor interfacial bonding between the filler and the matrix polymer and caused the Izod impact strength of the composite to be reduced. This poor interfacial bonding resulted in an increased in the number of micro voids, causing increased in water absorption. Because the number of micro voids caused by the larger amount of poor bonded area between the hydrophilic filler and the hydrophobic matrix polymer, water was easily absorbed by these voids. After treatment with NaOH, it showed a slight improvement in impact strength of composite (TPNR/RHiNa). The removal of surface impurities on RH filler surface seems beneficial in enhancing filler-matrix adhesion and increasing the toughness although it was not significant. With the addition of the compatibilizing agent (LENR), the interfacial bonding between the filler and the matrix polymer was greatly improved, resulting in the decreasing of water absorption. It showed that the highest impact strength for composite filled with LENR modified RH (TPNR/RHiNaLE 2) was obtained at 10% wt/v concentration of LENR. The strong interfacial bonding between the filler and the matrix polymer caused by the compatibilizing agents, limits the water absorption by the composites and increased the toughness of composite. It can be said that there exists a higher force transfer capability at the fibre-matrix interface in the composites with fibres treated by LENR as compatibilizing agent. It was due to hydrophobicity of rice husk filler was successfully obtained and led to maximum interaction and compatibility between RH filler and matrix. This subsequently increased the mechanical properties of the composite.

MORPHOLOGY OF COMPOSITE

The changes in the surface interaction between RH filler-polymer matrices were studied through morphology analysis. Figure 6 shows SEM micrograph of composite NR/HDPE/RH (90:10 v/v) with different RH surface treatments. As can be seen in Figure 6(a), the voids occur due to hydrophilic nature of RH fibre which reduce the compatibility with NR/HDPE matrix which is hydrophobic in nature. Moreover, surface impurities such as lignin, pectin, wax, natural oil and other surface impurities that cover the RH surface also weakened the adhesion and surface interaction between filler-matrix. After alkali treatment, the fibrillation of RH fibre was exposed and roughened the surface that lead to increase in the interfacial adhesion between RH filler and matrix TPNR as seen in Figure 6(b). Unfortunately, this change in morphology may create more voids for water to absorb thus, agglomeration of RH fibre occured. Figure 6(c) shows the morphology of TPNR/RH composite reinforced RH treated by NaOH then followed by 5% wt/v of LENR coating (TPNR/RHiNaLE 1). Treatment with NaOH removed some lignin, hemicelluloses and other impurities from the surface of fibre, thus exposing the fibre surface contact area. Therefore, the hydroxyl groups on the cellulose fibres were able to better interact with the LENR as compatibilizing agent because of the availability of a

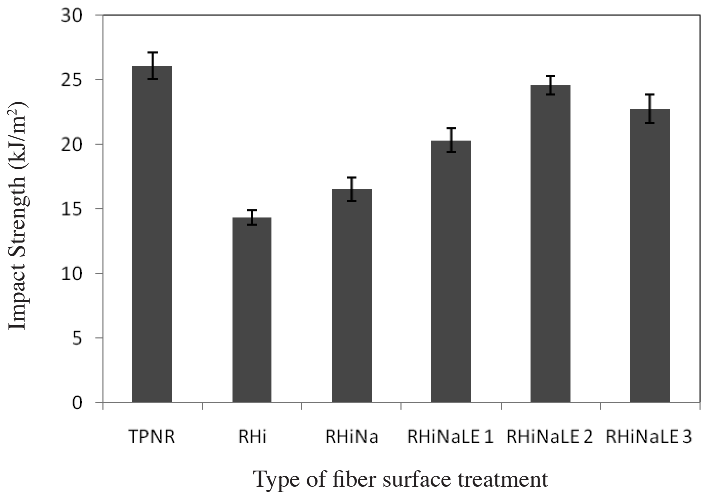

FIGURE 5. Impact strength of TPNR and (NR/HDPE)/RH (90:10 v/v) composite, plotted as a function of RH surface treatment

larger number of possible reaction sites. As can be seen on Figure 6(c), interaction between RH fibre and TPNR matrix occurred. It shows that the wetting process of matrix onto the RH fibre particle happen due to RH surface was already coated with LENR, which assumed improve the hydrophobicity of RH fibre. Even so, the wetting of matrix onto the RH particle was not fully achieved. Figure 6(d) shows morphology of TPNR composite reinforced RH treated by NaOH followed by 10% wt/v of LENR coating (TPNR/RHiNaLE 2). It shows that the wetting of matrix polymer onto the RH particle was perfectly achieved. Figure 6(e) shows morphology of TPNR composite reinforced RH treated by NaOH followed by 20% wt/v of LENR coating (TPNR/RHiNaLE 3). From the figure, it can be seen that LENR coating onto RH particle seems too much, or can be said the thickness of rubber which coat the RH surfaces was too high that could possibly lead to agglomeration between RH fibres within the composite. Consequently, it causes decreasing on mechanical properties of composite prepared as proven before.

CONCLUSION

This study investigated the effect of surface modification of rice husk with NaOH and LENR on mechanical properties of the NR/HDPE reinforced RH composites. The FTIR spectra showed the changes on peaks of LENR coated RH as rubber surface treatment in this investigation at 10% wt LENR in toluene solution. The SEM micrograph indicated that the surface of rice husk changed after treatment process. Modification of RH with NaOH 5% followed by 10% LENR showed the optimum tensile stress, modulus and impact strength as compared to untreated RH, NaOH treated and LENR treated RH composite. A significant increase of the strength was observed upon fibres addition when using LENR as compatibilizer. The changes were due to the increase of hydrophobicity and compatibility of the rice husk surface which improved the adhesion properties of rice husks and wettability of the fibre by the TPNR which led to imprased mechanical properties of the composite.

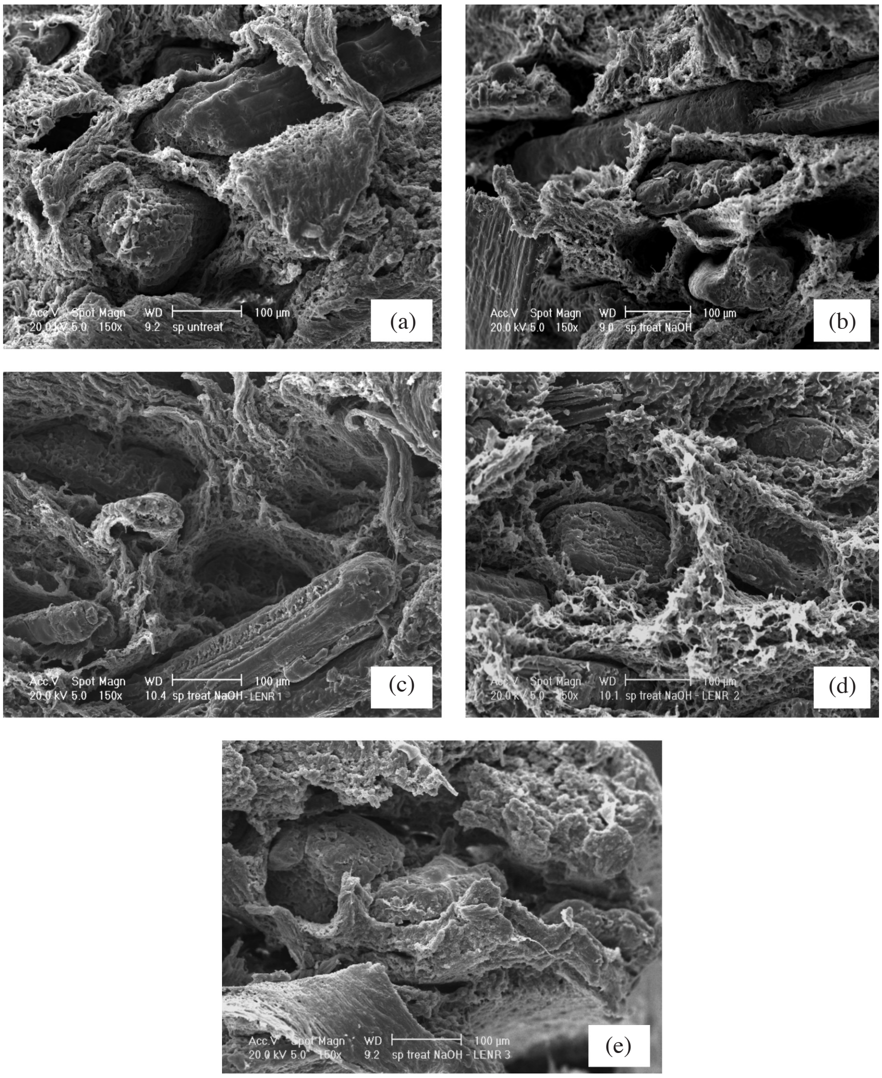

FIGURE 6. SEM micrographs of composites with magnification 150x: (a) TPNR/RHi, (b) TPNR/ RHiNa, (c) TPNR/RHiNaLE 1, (d) TPNR/RHiNaLE 2 and (e) TPNR/RHiNaLE 3

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