

EFFECT OF CHEMICAL TREATMENT ON RICE HUSK (RH) REINFORCED POLYETHYLENE (PE) COMPOSITES

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In this study rice husk reinforced polyethylene composites and their test specimens were manufactured using a single screw extruder and an injection molding machine, respectively. Raw rice husk was chemically treated with benzene diazonium salt in alkali, acidic, and neutral media, in order to improve in the mechanical properties. The mechanical properties of the composites prepared from alkaline media treated rice husk were found to increase substantially compared to those of acidic media, neutral media, and untreated ones. However, the values for the alkaline media treated rice husk-PE composites at all mixing ratios were found to be higher than those of treated acidic media, treated neutral media, and untreated rice husk composites respectively. The SEM micrographs reveal that interfacial bonding between the treated filler and the matrix has significantly improved, suggesting that better dispersion of the filler into the matrix was achieved upon treatment of rice husk. Based on filler loading, 35% filler reinforced composites had the optimum set of mechanical properties among all composites manufactured.

Keywords: Polymer matrix composites (PMCs); Mechanical properties; Scanning electron microscopy; Injection moulding

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INTRODUCTION

Recently there has been an increasing interest in the use of biodegradable polymers due to the serious environmental pollution arising from used and waste plastics, particularly polythene (PE). Biodegradable polymers can be obtained from renewable resources, microbially synthesized in the laboratory, or collected from petroleum-based chemicals (Premlal et al. 2002). Nowadays, synthetic polymers are combined with various biodegradable reinforcing fillers in order to improve the mechanical properties and obtain the bio-degradable and other characteristics demanded in actual applications (Yang et al. 2006, 2007; Choi et al. 2006). Research is being carried out to replace synthetic fibres with lignocellulosic fibres as reinforcing fillers (Thwe and Liao 2002; Park et al. 2003; Yang et al. 2004; Rana et al. 2003; Singleton et al. 2003). Compared to talc, silica, glass, carbon, and other synthetic fibres, the lignocellulosic fibres (corn stalk, rice husk, rice straw, wheat straw, grass, etc.) are lightweight, easily available, inexpensive, and contributing less wear of the machinery used for their production. Furthermore they are biodegradable and do not leave residues or resultant by-products that are toxic (Premlal et al. 2002; Vink et al. 2003). Besides, natural fibres as filler have

advantages over mineral fillers as they are non-abrasive and reduce the density of finished products. The cost of producing lignocellulosic reinforced polymeric composites is quite low. Hence, these composites have attracted much attention and are becoming increasingly important for the production of large variety of cheap, light-weight, environment friendly composites (Stokke et al. 2001).

Rice husk is one such widely available agro-waste byproduct, which contains cellulose (35%), hemicellulose (25%), lignin (20%), and ash (17% of which silica is 94%), by weight (Anon. 2008). Rice husk (RH) has been used as an alternative filler to commercial silica. In a similar study, maleic anhydride has been used to improve the properties of rice husk PP composites (Chand et al. 1987; Ismail 1999). This factor encouraged us to investigate the performance of rice husk as a filler for PE composites. One of the major drawbacks of using RH as a filling material is its hydrophilic nature, responsible for moisture absorption and consequent deformation of the product. To overcome this problem RH was chemically treated with benzene diazonium salt in different media at different pH, which in turn improved the mechanical properties of the composites. Thus the aim of this study is to prepare composite materials of bio-degradable nature and improved mechanical properties using chemically treated RH and polyethylene (PE). The effect of filler loading and chemical treatment at different pH on physico-mechanical properties and morphology of RH filled high density polyethylene (HDPE) composites are reported in this study.

MATERIALS AND METHODS

Materials

The high density polyethylene (HDPE), which was used as matrix material, was supplied by the Polyolefine Company, Private Limited, Singapore in the form of homopolymer pellets. The RH, used as reinforcing filler, was collected from Bangladesh Rice Research Institute (BRRI). The average particle diameter was 209 μm . Chemicals used to treat RH were HCl, NaNO_2 , $\text{C}_6\text{H}_5\text{NH}_2$ (Merck, Germany) and NaOH (Merck, India). Benzene diazonium salt was synthesized by the standard diazotization method using HCl, NaNO_2 and $\text{C}_6\text{H}_5\text{NH}_2$ (Kabir et al. 2006).

Treatment of Rice Husk

RH was dried at 105 °C for 24 hours and then kept in a sealed container. In order to have diazonium salt in acidic, neutral and alkali media (pH 6, 7, and 10.5 respectively), 80ml, 108ml, and 200ml of 5%NaOH was mixed with 300ml water in a beaker, respectively. 500g RH was submerged into the solutions mentioned above separately for 10 minutes at about 5 °C in an ice bath. A freshly prepared cooled solution of benzene diazonium salt was then poured slowly into the above mixtures with constant stirring for 10 minutes. RH was then taken out, washed with soap solution followed by water, and finally dried in open air.

Fabrication of Composites and Test Specimens

Dried raw and treated RH was initially mixed thoroughly with PE granules at 25, 30, 35, and 40 wt% each. The mixtures were passed through a single screw extruder machine at a constant temperature of $135 \pm 5^\circ\text{C}$. The extruded composites were cut into small pieces 15-20 cm long. All the pieces were then crushed into smaller granules using a grinding machine (Model FFC-23, Machinery Company Limited India). The granules were dried in a vacuum oven at 65°C for 1 hour and fed into an injection molding machine for making test specimens. The tensile and flexural test specimens were prepared at a molding temperature of 165°C .

Microstructural Analysis

Fourier transform infrared spectroscopy (FTIR)

The infrared spectra of the raw and treated RH were recorded on a Shimadzu FTIR-81001 Spectrophotometer.

Scanning electron microscopy (SEM)

The interfacial bonding between the polymer matrix and RH were investigated using a Scanning Electron Microscope (JSM-6701F) supplied by JEOL Company Limited, Japan. The micrographs were taken at a magnification of 300.

Mechanical Testing

Tensile, flexural, charpy impact, hardness and water absorption tests were carried out. In each case ten specimens were tested and the average values were reported.

Tensile test

Tensile tests of prepared composites were carried out with a Universal Testing Machine (Model: MSC-5/500, Agawn Seiki Company Limited, Japan) at a crosshead speed of 10 mm/min and the load cell used for the test was 250 KN. The tests were conducted according to ASTM D 638-01. Ten specimens were used for each evaluation and the dimension of the specimen used were 148mm x 10mm x 4mm. Tensile strength was calculated using the following equation,

$$\delta = F/A \quad (1)$$

where δ represents the tensile strength, F is the breaking load and A is the area of cross section.

Flexural test

The static flexural tests were carried out using the same Universal Testing Machine mentioned above. Tests were conducted following ASTM D 790-00. Ten specimens were used for each evaluation, and the dimensions of the specimen used were 79mm x 10mm x 4.1mm. The flexural strength and modulus were calculated using the following equations,

$$\text{Flexural strength, } \sigma_f = \frac{3PL}{2bd^2} \quad (2)$$

$$\text{Flexural modulus, } E = \frac{L^3m}{4bd^3} \quad (3)$$

where P is the maximum applied load, L is the length of support span, m is the slope of the tangent, and b and d are the width and thickness of the specimen respectively.

Charpy impact test

The dynamic Charpy impact tests of the composites were carried out according to ASTM D 6110-97 using a Universal Impact Testing Machine. The dimensions of the specimen used were 79mm x 10mm x 4.1mm.

Water absorption test

The water absorption tests on the composites were carried out following ASTM D 570-99. Specimens having dimension of 39mm × 10mm × 4.1mm were prepared. The specimens were dried in an oven at 105°C, cooled in a desiccator using silica gel, and immediately weighted. A Denver Instron balance was used for weight measurement. In order to allow the composites to absorb water, the weighted specimens were immersed in distilled water and kept for some specified time. The specimens were then taken out after 2 and 24 hours, for hot and cold water, respectively. The excess water on the surface of the specimens was removed using a soft cloth. The final weight of the specimens was then taken. The percentage of water absorption of the specimens was calculated using the following equation:

$$\text{Water absorption (\%)} = \frac{\text{Final Weight} - \text{Original Weight}}{\text{Original Weight}} \times 100 \quad (4)$$

Hardness test

The hardness tests of the composites were carried out using a Rockwell Hardness Testing Machine. The tests were conducted following ASTM D78.

RESULTS AND DISCUSSION

Tensile Properties

The tensile strength of raw and treated RH reinforced PE composites against different filler loading is presented in Fig. 1. It is observed from the figure that the tensile strength of raw RH reinforced PE composites decreased with increasing fibre loading. Similar results were also reported by other workers (Yang et al. 2006, 2007; Thwe and Liao 2002; Yang et al. 2004) who worked with different reinforcing agents. The decrease in tensile strength with the fibre loading may be due to the increase in weak interfacial area between the PE matrix and RH filler.

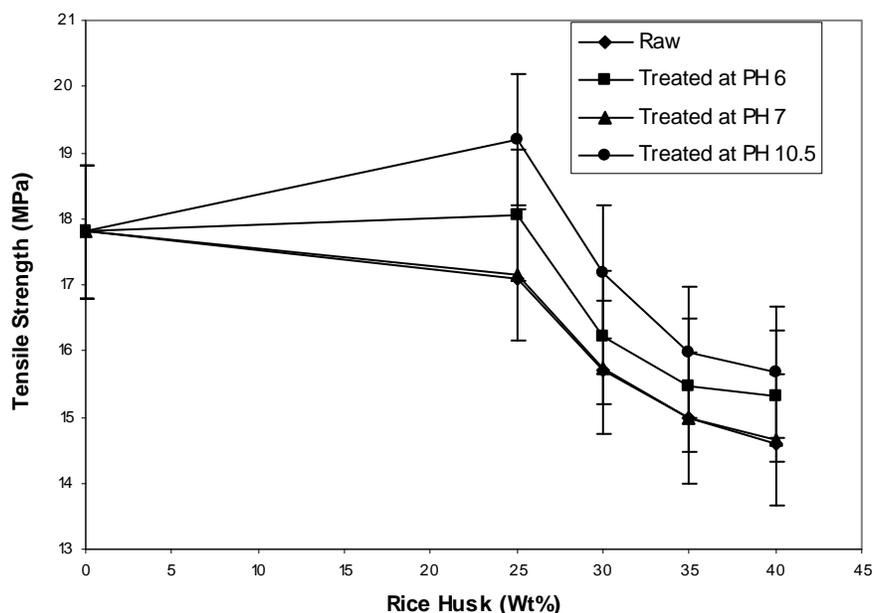


Figure 1. Variation of tensile strength at different filler loading

It is also evident from Fig. 1 that the tensile strength of PE composites reinforced with chemically treated RH changed differently with different treatment media and fibre loading. In alkaline media (pH 10.5) the tensile strength increased (approximately 10%) with the fibre loading up to 25%, then it decreased to a minimum value at 40% fibre loading. The decrease of tensile strength with the increase of fibre loading (from 25 to 40%) was also observed in other media. The change of tensile strength due to the change of media of chemical treatment may be due to the change in structure of cellulose anhydroglucose unit of RH. The chemical treatment of rice husk reduces the hydroxyl group content of cellulose anhydroglucose units by coupling with diazonium salts, as shown in Fig. 2. The FTIR spectroscopic analysis of the raw (Fig. 3) and treated RH (Fig. 4) confirms this phenomenon. The IR spectrum of treated RH clearly shows the presence of the characteristic band of the NO group in the region of $1600-1700\text{ cm}^{-1}$ and C-O stretching in the region of $1300-1000\text{ cm}^{-1}$. However the IR spectrum of the raw RH shows no such absorption bands but only in the region near 1716 cm^{-1} . This absorption band may be due to the carboxyl group of acetylene in hemicellulose and carboxyl aldehyde of lignin (Ismail et al. 2002).

The coupling reaction between hydroxyl groups and diazonium salts increases the interfacial bonding between the RH filler and PE matrix in the composites. However treatment with different media resulted in different extents of the coupling reaction. Treatment in alkaline media (pH 10.5) yielded the highest extent, followed by the acidic media (pH 6), and neutral media (pH 7), respectively. Thus the improvement of the interfacial bonding between the RH filler and PE matrix was highest in alkaline media, followed by acidic and neutral media. Consequently the tensile strength of the composites made of treated RH in alkali media had the highest tensile strength, followed by composites made of RH treated in acidic, neutral media, and raw RH, respectively.

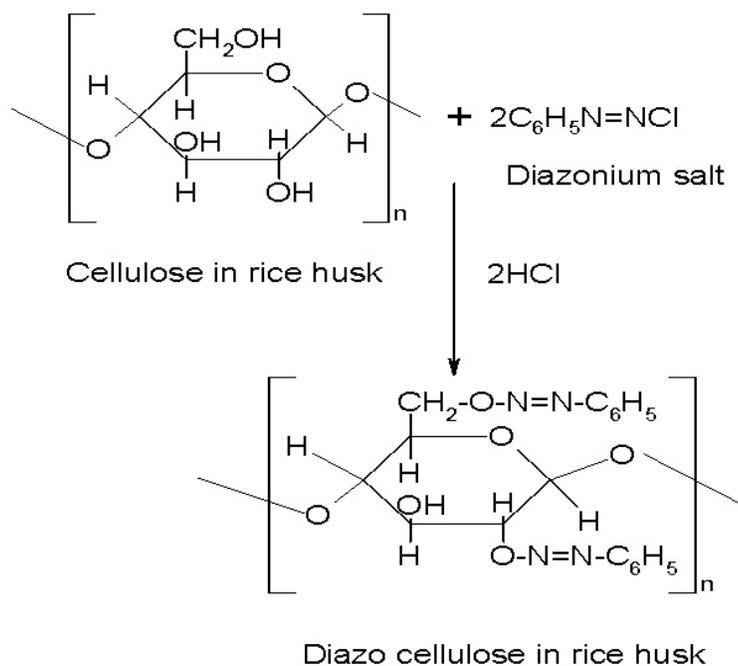


Figure 2. Treatment of cellulose in rice husk with diazonium salt

The tensile strength of the composites consisting of raw RH and neutral media treated RH were almost similar. This indicates that treatment in neutral media was not effective in increasing the tensile strength. The range of the tensile strength obtained in the current work, 14.58 to 19.2 MPa, was slightly lower than the range obtained in previous research (4-31 MPa) (Yang et al 2004). This could be due to the use of different matrix material in previous research.

Figure 4 shows the variation of the Young's modulus of composites made of treated and raw RH with PE at different filler loadings. The Young's modulus increase with filler loading was in accordance with other reported work (Thwe and Liao 2002; Yang et al. 2004; Rana et al. 2003; Joseph et al. 2002). During tensile loading, partially separated micro-spaces are created, which obstruct stress propagation between the filler and the matrix. As the fibre loading increases, the degree of obstruction increases, which in turns increases the stiffness.

The Young's modulus of composites made of treated RH in alkaline media were highest, followed by acidic and neutral media. The sample made from PE showed the lowest Young's modulus among all. The Young's modulus for rice husk filled polyethylene/rubber composites found in previous research was 0.1-0.16 GPa (Jamil et al 2006), whereas the range of the Young's modulus found in the current work was 1.78 to 2.32 GPa.

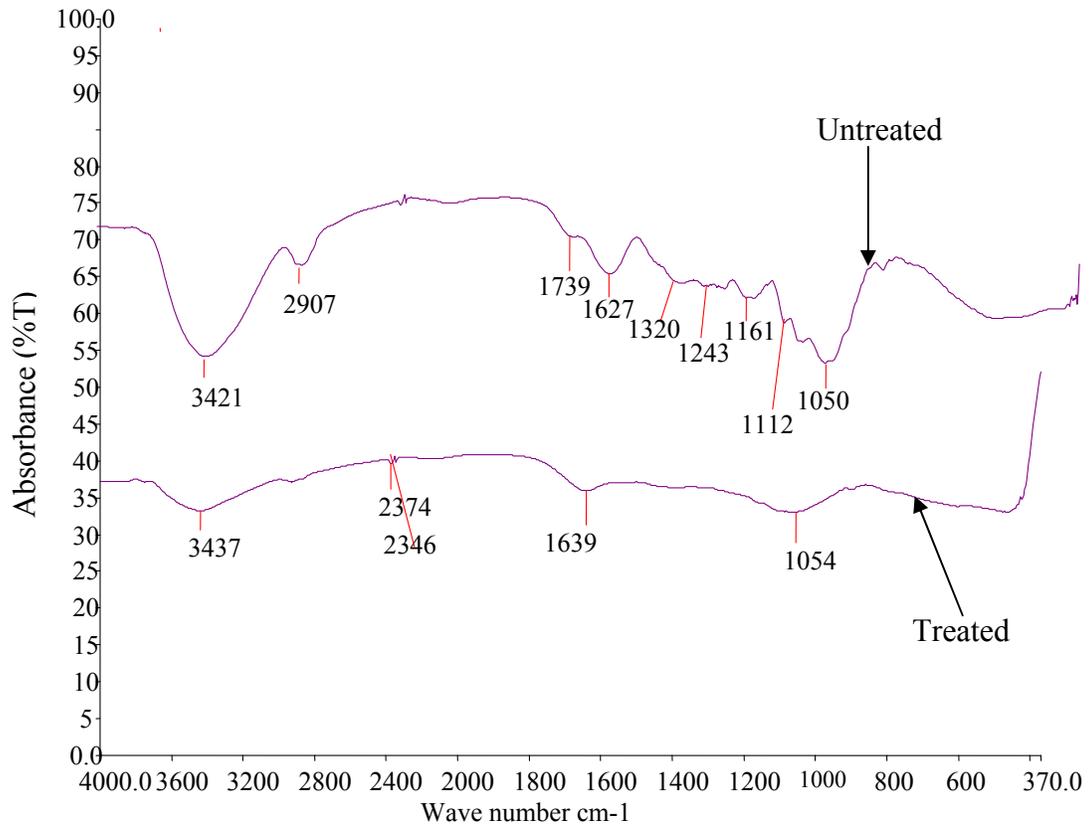


Figure 3. FT-IR spectra of untreated and treated rice husk

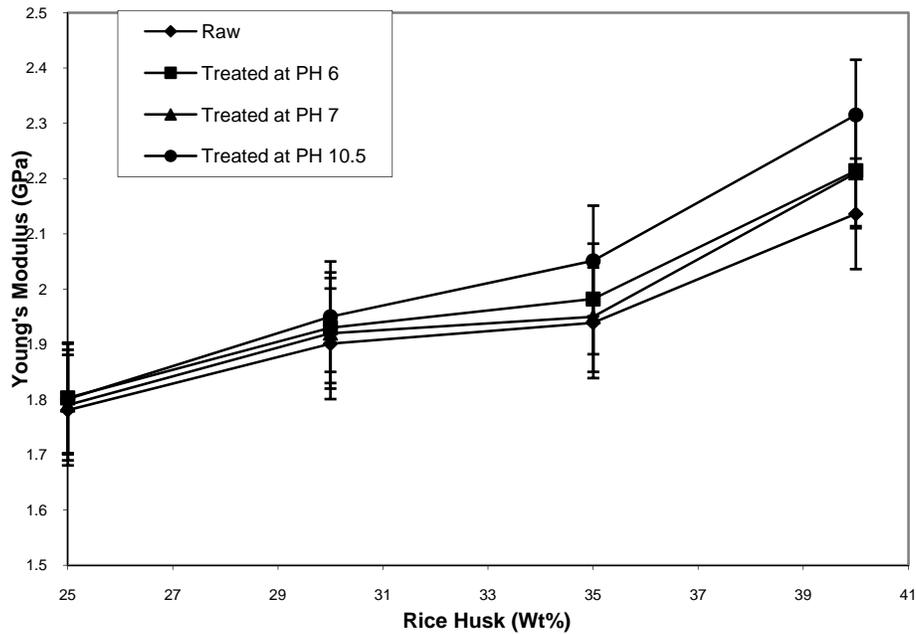


Figure 4. Variation of the Young's modulus at different filler loading

Flexural Properties

Flexural strength and modulus of both raw and treated RH reinforced PE composites at different filler loading are shown in Figs. 5 and 6 respectively. It is seen from Fig. 6 that the flexural strength increased with filler loading up to 35%; however at 40% the flexural strength decreased. Flexural strength of the alkaline and acidic media treated RH reinforced PE composites were almost similar but higher than those of raw and neutrally treated RH reinforced PE composites. Again the flexural strength of neutral media treated RH reinforced PE composites was slightly higher than those of raw RH reinforced PE composites.

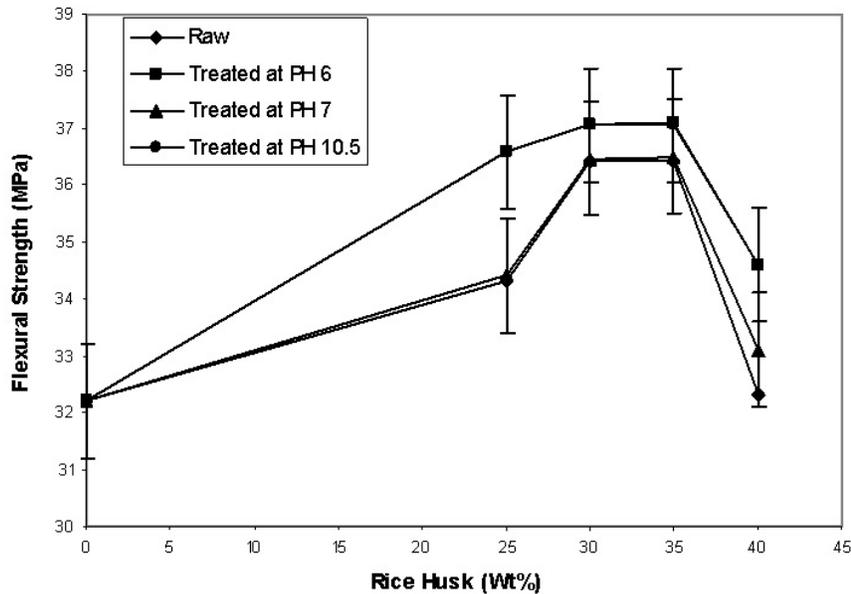


Figure 5. Variation of flexural strength at different filler loading

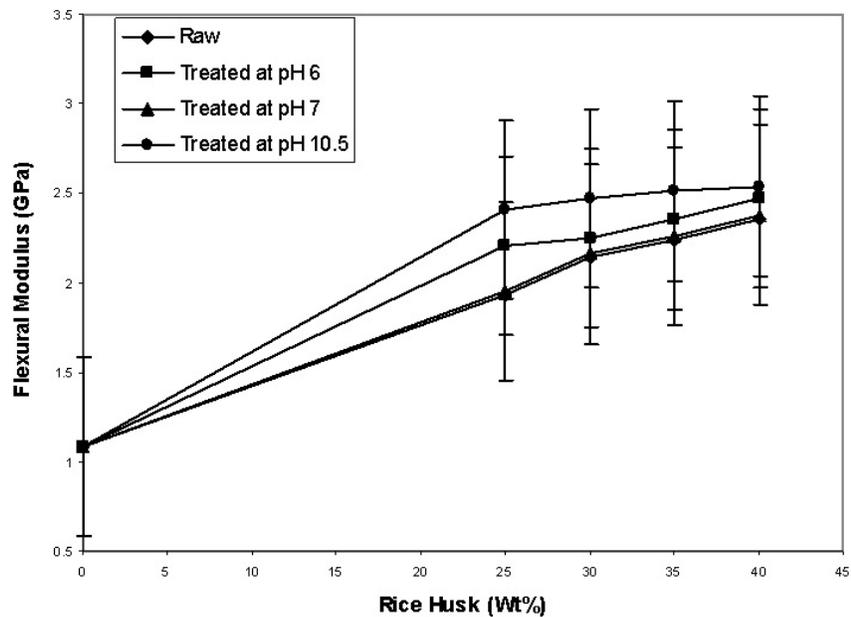


Figure 6. Variation of flexural modulus at different filler loading

Figure 6 shows that the flexural modulus of RH reinforced composites increased with the filler loading. Similar results have been also reported by other researchers with different reinforcement materials (Rana et al. 2003; Joseph et al. 2002; Lin et al. 2006). Flexural modulus of PE composites reinforced with alkaline media treated RH was highest, followed by the acidic media treated RH composites. Alkaline media treated RH exhibited better results because of the interfacial bonding between the filler and matrix. This is explained in SEM section. The flexural moduli of the PE composites reinforced with neutral media treated RH and raw RH were almost equal. This indicates that the composites reinforced with treated RH in neutral media were not so effective for the enhancement of flexural properties of the manufactured composites.

Impact Strength Results

Variation of the Charpy impact strength with filler loading for both raw and treated RH reinforced PE composites is shown in Fig. 7. The figure shows that the impact strength increased with filler loading. Similar results were also observed for different natural fibre reinforced composites (Joseph et al. 2002; Lou et al. 2007) up to 35% loading. At 40% filler loading the impact strength dropped. The impact strength of the fibre reinforced polymeric composites depends on the nature of the fibre, the polymer, and fibre-matrix interfacial bonding (Joseph et al. 2003). It has been reported that high fibre content increases the probability of fibre agglomeration, which results in regions of stress concentration requiring less energy for crack propagation (Karmakar et al. 2007).

As presented in Fig. 7, the impact strength of all composites increased with fibre loading. The impact strength increased gradually up to 30% due to the effect of the strengthening mechanism from the fibre (the rice husk). Upon addition loading after 30% the impact strength then becomes constant because the effect of the fibre become less significant due to the high ratio of fibre to the matrix. Eventually, the high loading of fiber above 35% weakened the bonding between fibre and matrix and eventually decreased the impact strength. This result suggests that the fibre was capable of absorbing energy because of strong interfacial bonding between the fibre and matrix. Another reason is that impact failure at higher fibre loading (%) may be due to fiber pull-out from the composites. The impact strengths of PE composites reinforced with RH in alkaline media were the highest, followed by treated in acidic media, neutral media, and raw RH respectively. The impact strength for rice husk filler/polypropylene composites found in previous research was 13-18 KJ/m² (Yang et al. 2004), whereas the range of the impact strength found in the current work was 31.74 to 41.45 J/m.

Hardness Results

Figure 8 shows the hardness of various manufactured composites at different filler loading. The figure shows that the average hardness increased with the increase of filler loading. This may be due to the increase in stiffness of the respective composites. The highest value was observed in the case of treatment in alkaline media. This could be attributed to both dispersion of the fibre into the matrix with minimization of voids and stronger interfacial bonding between the fibre and matrix.

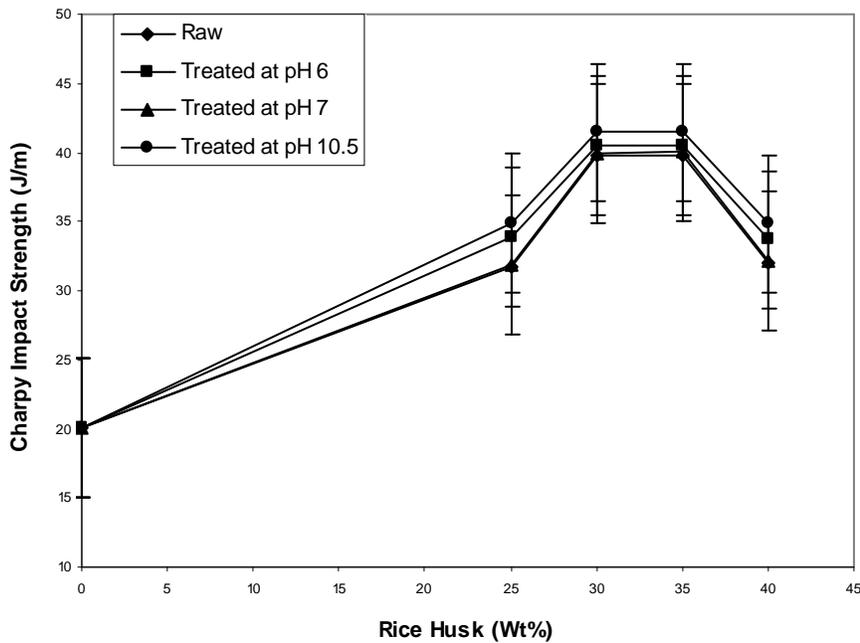


Figure 7. Variation of impact strength at different filler loading

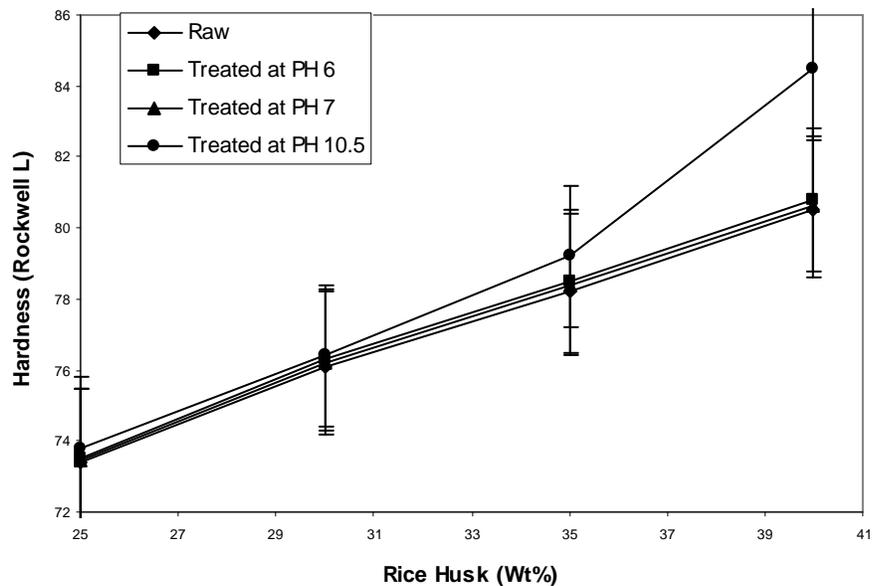


Figure 8. Variation of hardness at different filler loadings

Water Absorption Characteristics

Water absorption characteristics of the manufactured composites against the filler loadings in cold and hot water immersion are presented in Figs. 9 and 10, respectively. The water absorption (%) increased with the filler loading. As mentioned earlier, the

hydroxyl group of RH is responsible for the water absorption characteristics. With the increase in filler loading, the number of hydroxyl groups in the composites increases, which in turn increases the amount of water absorption. Composites reinforced with raw RH showed the highest water absorption, followed by neutral media treated RH composites, acidic media treated RH composites, and alkaline media treated RH composites, respectively.

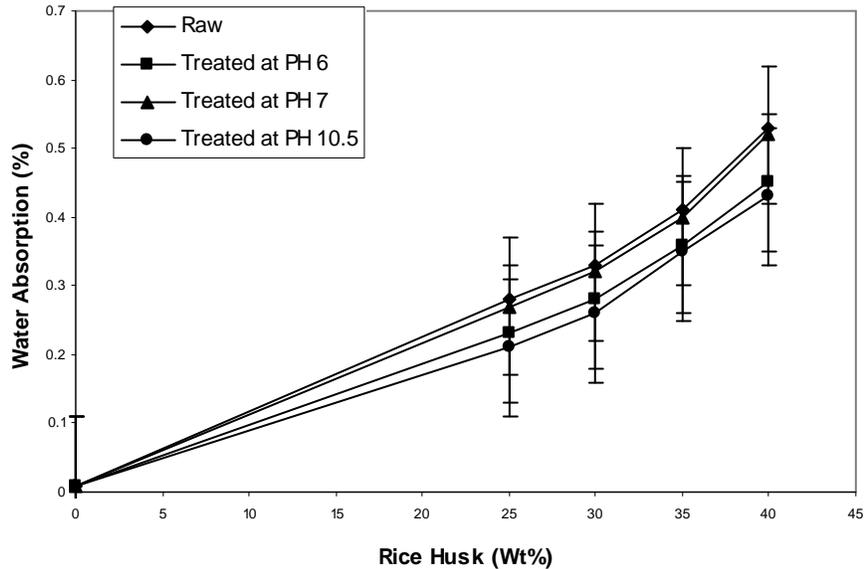


Figure 9. Variation of water absorption at different filler loadings immersed in cold water

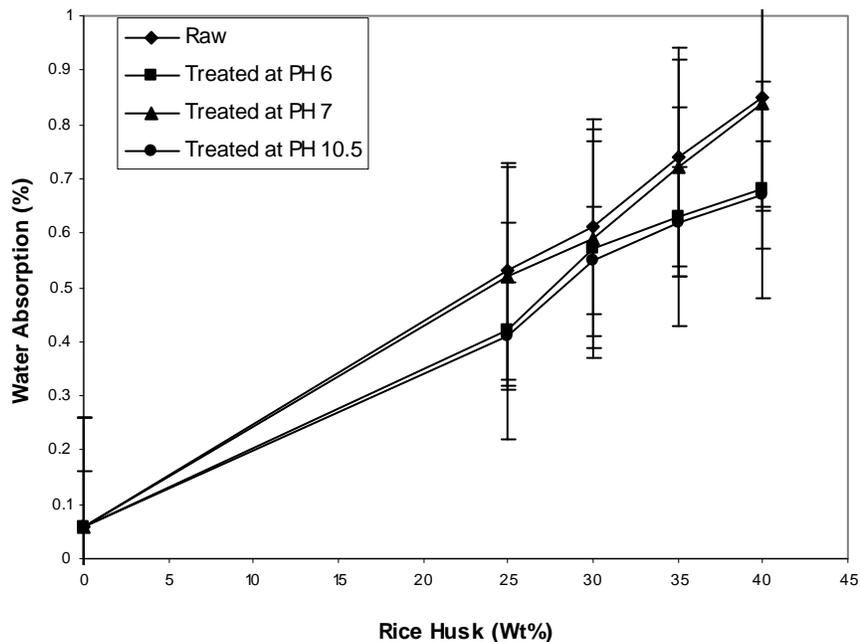


Figure 10. Variation of water absorption at different filler loadings immersed in hot water

SEM Morphology

Scanning electron micrographs of the raw, neutral, acidic, and alkaline media treated 35% RH reinforced PE composites are shown in Figs. 11 to 14, respectively. The raw RH fillers can be clearly seen in the composite micrograph due to the weak interfacial bonding between the filler and matrix (Fig. 11).

The alkali media treated system (Fig. 14) shows that the filler and matrix were not clearly differentiable due to the most improved interfacial bonding between them. From the SEM it is clear that the alkali media treated RH filler composite had a smooth surface texture. This smooth surface texture improves the fibre matrix interface bonding, as proven by other workers (Mohanty et al. 2000). The strong fibre matrix interface bonding was reflected in the tensile strength (Fig 1), Young's modulus (Fig 4), flexural modulus (Fig 6), and impact strength (Fig 7).

The results showed that the flexural strength (Fig 5) of both alkali and acidic media treated were similar. This finding needs further clarification in the future work.

The acidic and neutral media treated RH reinforced PE composite structures (Figs. 12 and 13) were in between the two mentioned above, due to medium improvement in the interfacial bonding between the filler and matrix.

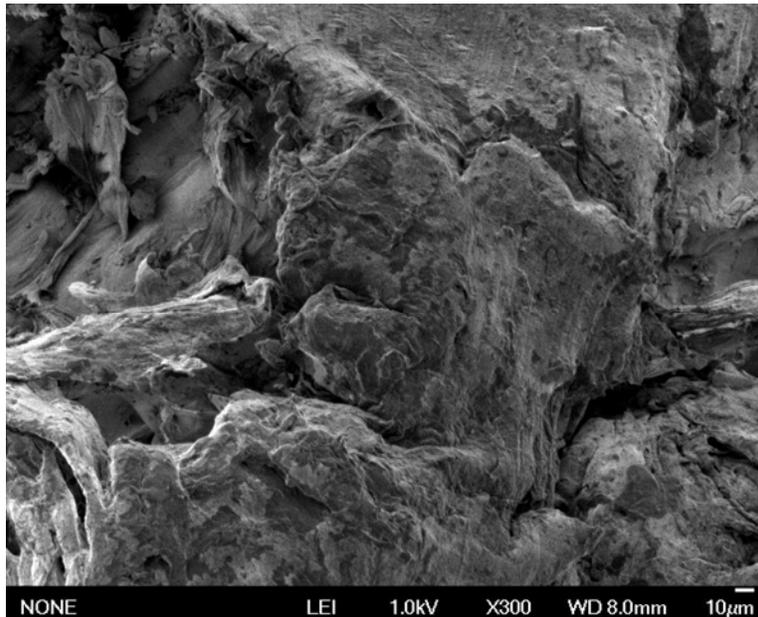


Figure 11. SEM micrograph of the 35% raw RH filler composite

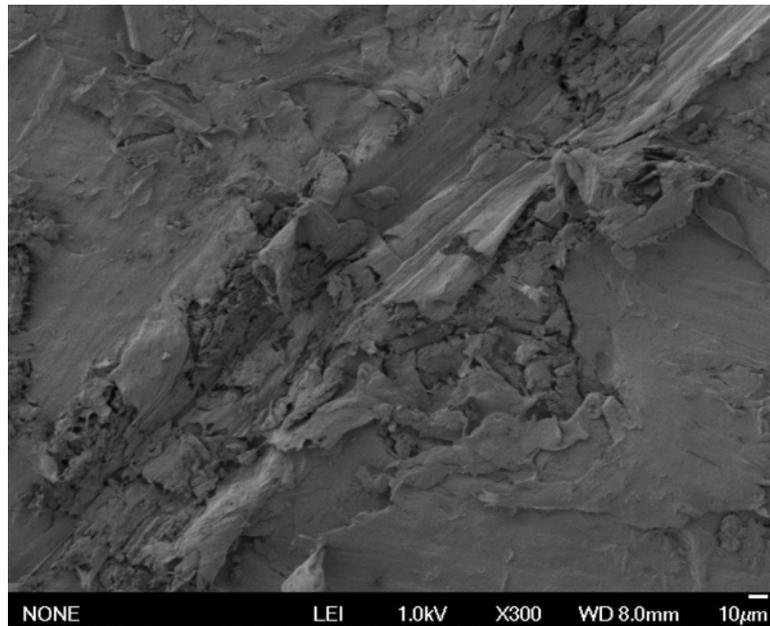


Figure 12. SEM micrograph of the 35% neutral media treated RH filler composite

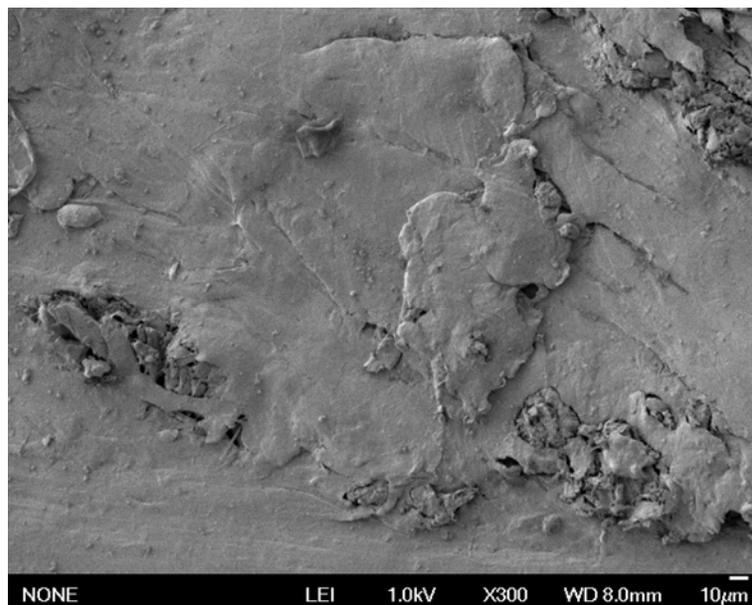


Figure 13. SEM micrograph of the 35% acidic media treated RH filler composite

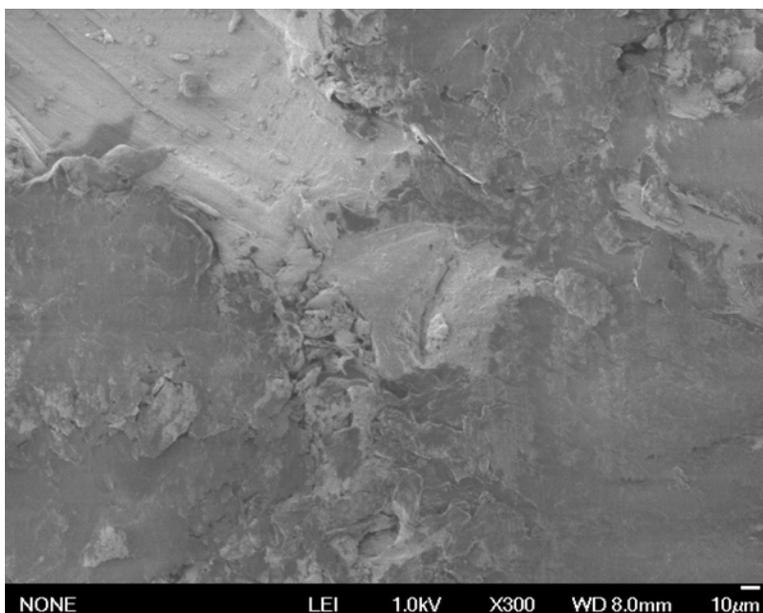


Figure 14. SEM micrograph of the 35% alkali media treated RH filler composite

CONCLUSIONS

Raw rice husk (RH) was chemically treated with benzene diazonium salt in alkaline, acidic, and neutral media separately in order to improve the compatibility between the hydrophilic RH and hydrophobic PE. Chemical treatment of the raw RH decreased the water absorption capacity of the resultant composites and improved the interfacial bonding between the filler and matrix. The tensile strength of the composites decreased with the RH filler loading. However, there was an increase in the tensile strength of the alkaline and acidic media treated 25% RH reinforced composite compared to PE alone.

The Young's flexural strength, flexural modulus, and Charpy impact strength increased with the filler loading. However the 40% filler loaded composites had lower flexural strength, modulus, and impact strength values compared to the 35% ones. Rockwell hardness results showed an increase in average hardness values with filler loading. The tensile strength, Young's modulus, flexural strength, flexural modulus, impact strength, and hardness values of the alkaline media treated RH reinforced PE composites were the highest, followed the acidic media treated RH, neutral media treated RH, and raw RH reinforced PE composites, respectively. The water absorption (%) increased with the filler loading, whereas the alkali media treated RH composites yielded lower water absorption capacity compared to the other ones.

The authors conclude that the alkaline media treated RH composites yielded the best mechanical properties, while the 35% RH filler reinforced PE composites had the optimum set of mechanical properties in comparison to other manufactured composites.

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