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Minh Hai Nguyen [a] , Byung Sun Kim [b] , Jong Rok Ha [c] & Jung Il Song [d]

[a] Composite Materials Lab, Korea Institute of Materials Science, Changwon, Korea

[b] Composite Materials Lab, Korea Institute of Materials Science, Changwon, Korea;, Email: kbs@kims.re.kr

[c] Composite Materials Lab, Korea Institute of Materials Science, Changwon, Korea

[d] Department of Mechanical Engineering, Changwon National University, Korea

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Effect of Plasma and NaOH Treatment for Rice Husk/PP Composites

Minh Hai Nguyen [a], Byung Sun Kim [a,*], Jong Rok Ha [a] and Jung Il Song [b]

[a] Composite Materials Lab, Korea Institute of Materials Science, Changwon, Korea
[b] Department of Mechanical Engineering, Changwon National University, Korea

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Abstract
Changes occurring in rice husk (RH) polypropylene (PP) composites with 20% concentration of a NaOH treatment for 24 h and plasma treatment, respectively, were compared with raw RH/PP for the void contents, mechanical properties and water absorptions. In all cases, 2% MAPP was evenly mixed with PP and atmospheric glow discharge (AGD) was used in plasma coating of RH. A study of the mechanical properties and SEM observations for plasma- and NaOH-treated, respectively, for RH/PP indicated improved adhesion of RH with PP. When RH was alkali-treated with 20% concentration for 24 h, RH/PP's tensile strength and modulus were improved by 100 and 229%, respectively, compared with raw RH/PP. However, plasma treatment on RH showed better improvement in tensile strengths by 140% and modulus by 247%, respectively. Moisture absorption on plasma-treated RH/PP was lowered by more than 56% compared with raw RH/PP.

Keywords
Rice husk, NaOH, plasma treatment, RH/PP composites

1. Introduction

Rice husk is a cellulose-based fibre, which can be utilized in the manufacture of composite panels. Poor interaction between rice husks and the binding materials results in a weak particle–matrix adhesion and therefore rice husks have not been successfully exploited like other cellulose fibres [1]. The outer surface of rice husk is relatively rougher than the inner surface that houses the rice grain.

Alkali treatment is one of the well known processes to increase mechanical properties. The process alters the chemical content in crude fiber by removing lignin, hemi-cellulose, pectin and changing the state of the materials from hydrophilic to hydrophobic. The large amount of hemi-cellulose lost made the fibers lose their ce-

* To whom correspondence should be addressed. E-mail: kbs@kims.re.kr
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menting capacity and caused them to separate out from each other, making them finer [2–8].

The plasma state, recognized as the fourth state of matter, is broadly defined as a gaseous environment composed of charged and neutral species with a net zero electric charge. This manifestation of matter can be generated by increasing the energy content of atoms and molecules regardless of the nature of the energy [9]. Plasma surface treatment can be used to modify the chemical and physical structure of the fibre surface layer and tailor the fibre–matrix source. In addition to the bonding strength, to positively influence the bulk mechanical properties, its advantages are that it is clean and dry, the material integrity is maintained and there are no environmental problems created by the processing [9].

In this study, atmospheric glow discharge (AGD) is developed with industrial plasma sources by using high voltage radio frequency (RF) excitation at kHz frequency ranges. It is possible to produce a steady-state uniform glow discharge at atmospheric pressure with various gases, thus eliminating the requirement of a vacuum system to expose the materials to plasma. The RF frequency should be in the right rage: if it too low, the discharge will not initiate and if it too high, the plasma will form filamentary discharge or will be transformed to arc discharge. Therefore only in the limited range of frequency band can the AGD be produced. The advantage of the use of AGD is that it will modify or coat the materials with good uniformity and will produce modifications in the coated materials continuously.

For RH/PP, the effect of plasma and NaOH treatment on RH, respectively, are studied for the void contents, mechanical properties and water absorptions with 2% MAPP addition to all samples.

2. Experimental

2.1. Materials

2.1.1. Polypropylene Fibres

PP fibre was supplied by Honam Petrochemical Corporation, specific weight ~0.95 g/cm^3; diameter = 20–200 μm; melting point = 170°C; molecular weight >10 000 g/mol.

2.1.2. Rise Husk (RH)

RH was collected in Busan, Korea: density, 0.99 g/cm^3; length, 2.0 mm; moisture content, about 6% before drying and 2% after drying without vacuum. RH was vacuum dried at RT to 0.0% before plasma coating.

2.2. Processing

2.2.1. Rice Husk Treatments

The concentration of 20% in NaOH treatment on RH for 24 h was the most appropriate NaOH concentration. The rice husk was then washed with fresh water several times to remove any traces of NaOH on the fibre surfaces, neutralized with

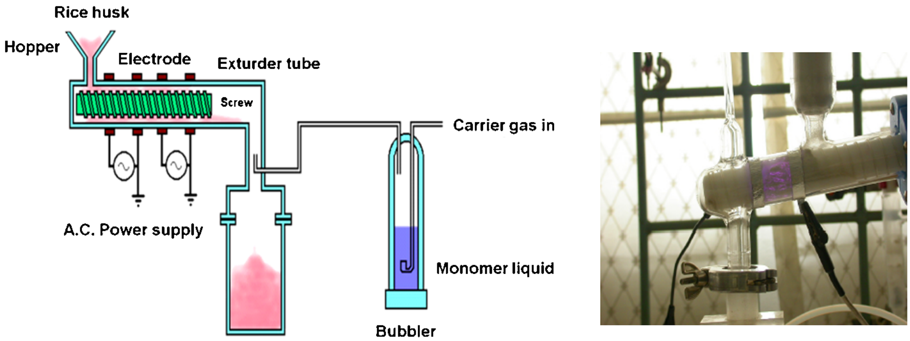


Figure 1. Schematics of the plasma surface coating process. This figure is published in color in the online version.

dilute acetic acid, and again washed with distilled water. The final pH of 7 was maintained.

2.2.2. Rice Husk Plasma Treatments

For rice husk plasma treatments, helium was used as carrier gas and a monomer such as hexamethyl-disiloxane (HMDSO) was used to modify the surface property by plasma polymerization. The monomer molecules undergo selective chemical bond breaking and recombination processes to form macromolecules. The new structures, consisting of Si–C and Si–O–Si functionalities, are found to be chemically inert and cross-linked in nature. Coatings obtained from HMDSO plasmas demonstrated reduced moisture and oxygen permeability, both of which are related to the density and structure of the plasma 'polymer' films [10].

As shown in Fig. 1, the electrodes were inductively connected around the tube and the frequency and the voltage input were 20 kHz and 3 kV, respectively. Rice husk is added to the hopper, which is advanced by the screw located within the reactor. This is where the rice husk is plasma coated in a continuous process with HMDSO.

2.2.3. Preparation of Composite Blends

The PP and plasma coated RH were mechanically mixed and then pellets were produced by PRISM TSC 16 TC (Thermo Electron Corporation) twin-screw extruder whose screw speed was 160 rpm and extruder temperatures at the inlet, middle and outlet were 135, 200 and 160°C, respectively. The pellets were vacuum dried at 75°C for 48 h. The vacuum dried pellets were molded in a hot press to form a mechanical test specimen for 6 min at 190°C and 300 kgf/cm^2 pressure.

2.3. Voids Content Test

The void contents of a composite may significantly affect some of its mechanical properties (ASTM D-2734). Higher void contents usually mean lower fatigue resistance, greater susceptibility to water penetration and weathering, and increased variation or scatter in strength properties. Calculation of the void content is desirable to estimate the quality of a composite. This can be calculated with the following equation:

$$V = 100(T_\mathrm{d} - M_\mathrm{d})T_\mathrm{d},$$

where V is the void content, volume (%), T_d is the theoretical composite density, and M_d is the measured composite density (D792).

2.4. Mechanical Properties

Tensile strength, tensile modulus and elongation were measured using ASTM D-638 Type-I test specimens. Instron 5882 was used for the mechanical properties tests. For each mechanical test, at least 7 specimens were tested.

2.5. Water Absorption of Composites

Specimens of $76.2 \times 25.5 \times 3.2\ \mathrm{mm}^3$ were immersed in distilled water at room temperature (ASTM D-570). They were removed from the bath and weighed before and after drying. Weight change measurements were made in quadruplicate using a micro-balance. Percent weight change during water sorption was determined as follows:

$$WA\ (\%) = (M_\mathrm{c} - M_\mathrm{o})/M_\mathrm{o} \times 100,$$

where M_o is the mass of the dried specimen and M_c is the mass of the specimen as a function of immersion time.

3. Results and Discussion

3.1. Void Contents

With raw RH, the voids content for RH/PP was 4.72%. After plasma and NaOH treatment, respectively, the voids contents of the composite were 1.26 and 1.42%, respectively (Table 1).

3.2. Mechanical Properties

3.2.1. Tensile Strengths and Tensile Modulus

Figure 2 shows the tensile strengths of RH/PP composites. The tensile strength of untreated RH/PP composites is about 9 MPa, while the tensile strength of NaOH-treated is about 18 MPa — an improvement of about 100% compared with raw

Table 1.
Void content in RH/PP composites

Samples	RH, PP and MAPP (wt%) in composites	Voids content (%)	Density ($\mathrm{g/cm^3}$)
Raw	50% Raw RH, 50% PP, 2% MAPP	4.72	0.95
NaOH-treated	50% NaOH-treated RH, 50% PP, 2% MAPP	1.42	1.02
Plasma-treated	50% Plasma-treated RH, 50% PP, 2% MAPP	1.26	1.04

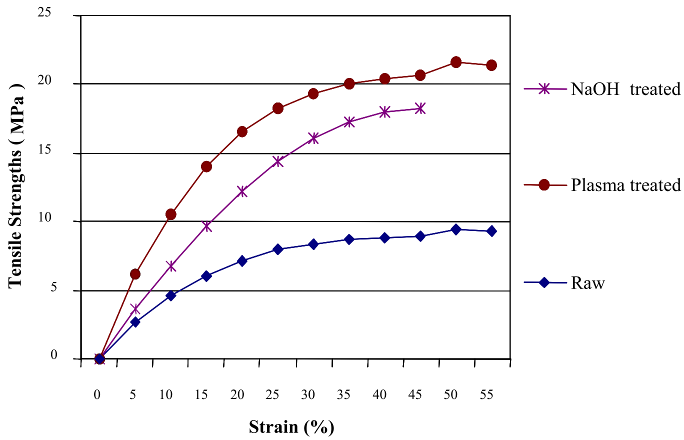


Figure 2. Tensile strengths of RH/PP composites. This figure is published in color in the online version.

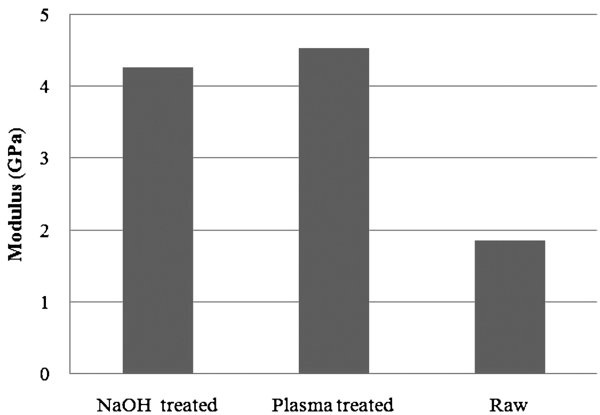


Figure 3. Tensile modulus of RH/PP composites.

rice husk. The highest tensile strength for plasma-treated RH/PP composites was 22 MPa, which represents a 140% improvement. This huge increase in tensile strength due to the plasma treatment of RH may have resulted from the improved compatibility of the RH with the matrix.

Figure 3 shows the tensile modulus of RH/PP composites. The tensile modulus of raw RH composites is about 1.86 GPa, while the tensile modulus of NaOH-treated is about 4.26 GPa — an improvement of 247% compared to the raw RH. For plasma-treated RH/PP composites, the highest tensile modulus obtained was 4.6 GPa.

3.2.2. Elongation at Break

Figure 4 shows the elongations at break for RH/PP composites. The lowest elongation at break was 3.7% obtained with raw RH/PP. For plasma- and NaOH-treated,

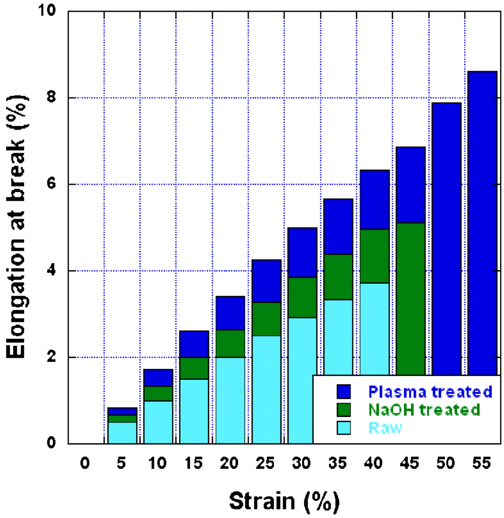


Figure 4. Elongation at break of RH/PP composites. This figure is published in color in the online version.

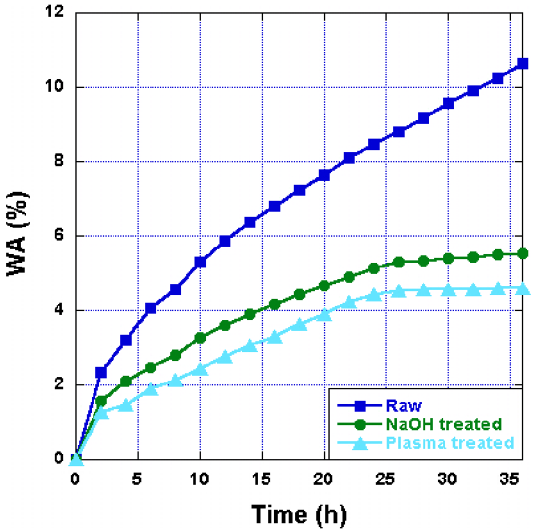
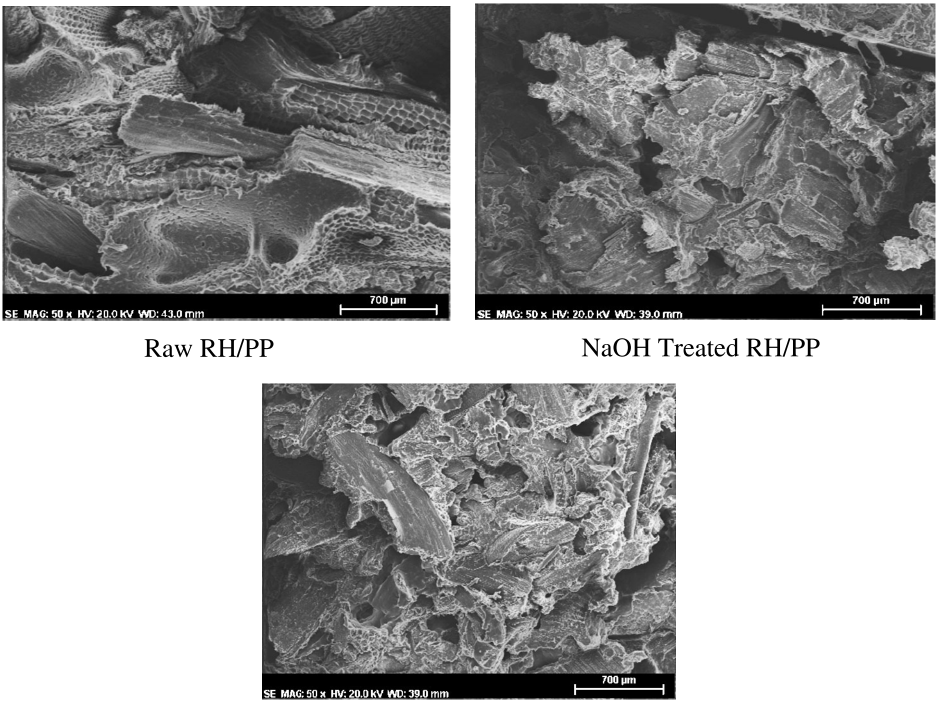


Figure 5. Water absorption of RH/PP composites. This figure is published in color in the online version.

respectively, RH/PP composites the values were 8.7 and 5.1%, respectively. The highest elongations at break were obtained with plasma treatment.

3.3. Water Absorption

Figure 5 shows water absorptions of RH/PP composites. Water absorptions for plasma-treated RH/PP was lowest with a maximum of 4.5%. Maximum water absorption for NaOH-treated RH/PP was about 5.5% while the maximum water



Raw RH/PP NaOH Treated RH/PP

Plasma treated RH/PP

Figure 6. SEM on the fracture surface of RH/PP composites.

absorbed for raw RH/PP were more than 10.5% for 36 h immersion at room temperature. The results indicate that the plasma coatings have most effectively resisted the water absorption.

3.4. SEM of RH/PP Composites

In Fig. 6, the bonding between RH and PP is observed to be improved from left to the right figure indicating that the plasma treatment on RH indeed provided best interfacial bonding between RH and PP.

4. Conclusions

(a) The lowest void content, highest tensile strength and modulus for RH composites were achieved from the plasma treatments on RH.

(b) The visual inspection and mechanical test results indicate that the RH were quite evenly and strongly adhesive within the PP matrix. This may be due to the enhancement of the compatibility of the RH with the matrix.

(c) Lowest water absorption on RH/PP was also obtained from the result of plasma treatments on RH. Moistured absorption was lowered by 55% compared with raw RH/PP.

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