

## **MECHANICAL PROPERTIES, WATER ABSORPTION, AND SWELLING BEHAVIOUR OF RICE HUSK POWDER FILLED POLYPROPYLENE/ RECYCLED ACRYLONITRILE BUTADIENE RUBBER (PP/NBRr/RHP) BIOCOMPOSITES USING SILANE AS A COUPLING AGENT**

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The performance of rice husk powder (RHP) filled polypropylene (PP)/ recycled acrylonitrile butadiene rubber (NBRr) biocomposites with and without coupling agent,  $\gamma$ -aminopropyltrimethoxysilane (APS), were investigated. The composites with different RHP filler loading (0 to 30 phr) were prepared in a Haake internal mixer. Mechanical properties, swelling behavior, and water absorption of PP/NBRr/RHP were studied. Increasing RHP loading in PP/NBRr/RHP biocomposites increased processing torque, tensile modulus, water absorption, and swelling in oil but decreased the tensile strength and elongation at break of the biocomposites. The  $\gamma$ -APS treated RHP composites exhibited higher processing torque, tensile strength, and tensile modulus but lower elongation at break when compared to untreated RHP composites. This is due to strong bonding between  $\gamma$ -APS treated RHP filler and PP/NBRr matrices. These findings were well supported by micrographs from the morphology studies. The  $\gamma$ -APS treatment on RHP improved the adhesion between RHP fiber and PP/NBRr polymer matrices, which led to less water and oil absorption into PP/NBRr/RHP/ biocomposites.

*Keywords: Rice husk powder; Biocomposites; Mechanical properties; Water absorption; Swelling; Coupling agent; Polypropylene*

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### **INTRODUCTION**

The use of natural fibers as reinforcements or fillers in polymer composite systems has gained extra attention in recent years. Numerous studies have been carried out on the utilisation of natural fillers such as sago, sisal, short silk fibre, oil palm, empty fruit bunch, rice husk ash, jute fibre, rubber wood powder, jute, hemp, sisal, cotton stalk, kenaf, sugarcane, banana fibers, and cellulose fibres as reinforcement materials (Satyanarayana et al. 2009; Nabi and Jog 1999; Tajvidi et al. 2006; Sgriccia et al. 2008). Compared with traditional reinforcement materials such as inorganic fiber, natural fibers such as rice husk have advantages: low density, low cost, harmlessness, abundance, being renewable, being less abrasive, having high toughness and comparable specific strength, and being easily biodegradable (Premalal et al. 2002).

Such advantages of natural fibers over man-made fibers favor the use of natural fibers a potential replacement for traditional reinforcement materials such as glass fiber in composite material systems (Rozman et al. 2001). Its suitability as a filler or additive in plastic materials, however, is not well defined because these composites are found to be incompatible or immiscible due to poor physical and chemical interaction between the phases. The incompatibility may be due to the fact that the polyolefin is non-polar and hydrophobic, whereas lignocellulosic material is polar due to the presence of hydroxyl groups in cellulose (Holden et al. 1996).

In order to solve this problem, studies have been performed on surface modification for the purpose of rendering the hydrophilic surface of rice husk powder (RHP) more compatible with hydrophobic polyolefins by using coupling agents (Holden et al. 1996; John et al. 2008; Ismail and Mega 2001). Chemical treatment on fiber surface and use of coupling agent are excellent methods to improve the compatibility between hydrophobic matrix's and hydrophilic fillers (Paul et al. 2008; Ismail et al. 2002). Many investigations have been carried out on this subject by numerous researchers (Kazayawoko et al. 1999; Li and Matuana 2003).

Coupling agents based on alkoxy silanes are frequently employed for cellulosic fibers. Hydrolysis of the alkoxy groups with a subsequent reaction with hydroxyl groups from cellulose can provide chemical bonding with the fibers (Bledzki et al. 1996). Ismail et al. (2001) utilized silane coupling agent on white rice ash-polypropylene/natural rubber composites, and found that silane coupling agent improved the mechanical properties of the composite.

Liu et al. 2009 also reported that silane modification of jute fibers improved the mechanical properties of jute fiber reinforced poly(butylenes succinate) bio-composites. However, so far no work has been reported on the effect of  $\gamma$ -aminopropyltrimethoxysilane ( $\gamma$ -APS) on the properties of PP/NBRr/RHP biocomposites.

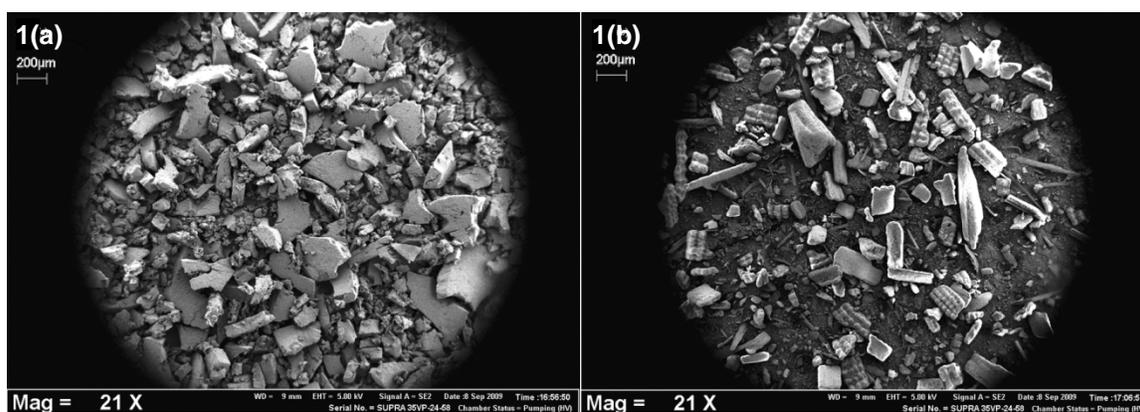
Acrylonitrile-butadiene rubber (NBR) is commonly considered the workhorse of the industrial and automotive rubber products industries. By selecting an elastomer with the appropriate acrylonitrile content in balance with other properties, NBR can be use in a wide variety of application areas requiring oil, fuel, and chemical resistance. On the industrial side, NBR finds uses in conveyor belting, hydraulic hose, roll covers, oil field packers, and seals for all kinds of plumbing and appliance applications. In Malaysia, the output of nitrile rubber gloves is large. Most of this material originates from medical, industrial, as well as research activities. After a certain period of time these polymeric materials are not serviceable and mostly discarded. To solve this environmental issue, we have used a NBRr gloves (waste) obtained from industrial floor in effort to create a value added instead of being scrapped.

In this study, rice husk powder was used as a filler to produce biocomposites based on polypropylene/recycled acrylonitrile butadiene rubber. The effect of filler content and a coupling agent,  $\gamma$ -aminopropyltrimethoxysilane (APS), on tensile properties, swelling behavior, and water absorption of PP/NBRr/RHP biocomposites was investigated.

## EXPERIMENTAL

### Materials

PP Grade 6331 was obtained from Titan Pro Polymers (M) Sdn. Bhd. Johor, Malaysia with a melt flow index of 14g/10 min at 230° C and a density of 0.9g/cm<sup>3</sup>. Recycled Acrylonitrile Butadiene Rubber (NBRr) with 33% acrylonitril content was obtained from Juara One Resources Sdn. Bhd. Penang, Malaysia. Coupling agent  $\gamma$ -aminopropyltrimethoxysilane (APS) was supplied by Alfa Aesar (M) Sdn Bhd, Malaysia. Rice husk powder was produced by grinding rice husk supplied by Thye Heng Chan Enterprise Sdn. Bhd, Malaysia in a pulverizing machine (Rong Tsong Precision Technology Co. Product Id: RT-34) at a speed of 2850 rpm. The powder was then sieved at a particle size of 150 to 300  $\mu$ m and dried at 110° C for 24 h in a vacuum oven to produce rice husk powder of homogeneous fractions. An homogenous particle size of RHP and NBRr was maintained with mean particle size of 200  $\mu$ m at magnification of 21X [Fig. 1(a-b)].



**Fig. 1(a-b).** SEM micrographs of (a) recycle acrylonitrile butadiene rubber and (b) rice husk powder at magnification of 21X

### APS Treatment

The APS treatment reaction for RHP was carried out in a mixture of water and ethanol (40/60 volume, respectively). 3g of  $\gamma$ -aminopropyltrimethoxysilane was first introduced into 1000 mL of the water/ethanol mixture and was allowed to stand for 1 hour. The pH of the solution was maintained at 4 by the addition of acetic acid. Then, 10 g of RHP was added into the solutions, which were continuously stirred for 1.5 hours. The treated RHP was filtered, dried by air and then by vacuum oven at 80°C for 24 hours.

### Processing

Formulations of RHP filled PP/NBRr biocomposites are given in Table 1. Prior to compounding, PP granule, NBRr powder, and RHP powder were dried using a vacuum oven at 80° C for 24 h. The melt mixing technique using Haake Internal mixer at 180° C was employed. PP and NBRr matrices are allowed to melt prior to adding of RHP filler for the fabrication of this biocomposite. PP was first charged into the internal mixer and

melted for 4 minutes prior to NBRr addition. RHP was then added into the mixture after 6 minutes of mixing. The mixture was allowed to further mix for another 3 minutes to obtain the stabilization torque. The total mixing time was 9 minutes for all samples. The composites were then compression-molded into a 1 mm sheet for preparing test samples using Go-Tech compression moulding machine type GT-7014-A30C, manufactured by Go-Tech Testing Machines Inc, Taichung, Taiwan. For test sample fabrication, the composites were preheated for 7 minutes at 180°C, compressed at 1000 psi for 2 minutes, and then cooled for 2 minutes. Moulded samples were then cut into dumbbell shapes with a Wallace die cutter model: S6/1/6.A, from Wallace Instruments Inc., England.

### Tensile Measurement

The tensile properties were measured using an Instron machine Model: 3366, manufactured by Mecom Sdn Bhd, Malaysia at a cross-head speed of 5 mm/min at  $25 \pm 3^\circ\text{C}$ , according to ASTM D 638-08. Tensile strength, tensile modulus, and elongation at break of the each sample were obtained from the average of five specimens with their corresponding standard deviations.

### Water Uptake

The water uptake test was carried out by immersing the samples in distilled water at room temperature ( $25^\circ\text{C}$ ). The specimens were dried for 4 hours in a vacuum oven at  $100^\circ\text{C}$  until a constant weight was attained prior to immersion in water in thermostated vessels at ambient temperature. Weight gains after exposure were recorded by weighing them periodically on a Sartorius balance Model: BS224S, with a precision of 1 mg, manufactured by Sartorius AG, Germany. The moisture content at any time  $t$ ,  $M_t$  (%) as a result of moisture absorption, was calculated by using Equation 1.

$$M_t = 100 \times (w_t - w_o) / w_o \quad (1)$$

where  $w_o$  and  $w_t$  are original dry weight and weight after exposure, respectively.

### Swelling

Swelling test was determined by immersing samples in ASTM oil No. 3, in accordance with ASTM D 471-98. The test pieces of dimension 30 mm x 5 mm x 1 mm were weighed and immersed in ASTM oil No. 3 at room temperature for 70 hours. The swelling percentage of the samples was calculated by using Equation 22 with 5 replicates,

$$S = 100 \times (w_s - w_o) / w_o \quad (2)$$

where  $w_o$  and  $w_s$  are original dry weight and weight after swelling exposure, respectively.

### Morphology Study

The tensile 1 mm dumbbell fracture surface at failure mode was examined using Field Emission Scanning Electron Microscope (FESEM ZEISS SUPRA36VP-24-58). SEM micrographs were taken at various magnifications for morphological evaluation. Prior to the SEM observations, the fractured ends of the specimens were mounted on

aluminium stubs and sputter coated with a thin layer of gold to avoid electrical charging during examinations.

**Table 1.** Formation of RHP filled PP/NBRr Biocomposites\*

Materials	Composition (phr)
Polypropylene(PP)	70
Recycled Acrylonitrile Butadiene Rubber (NBRr)	30
Rice Husk Powder (RHP)	0, 5, 10, 15, 30

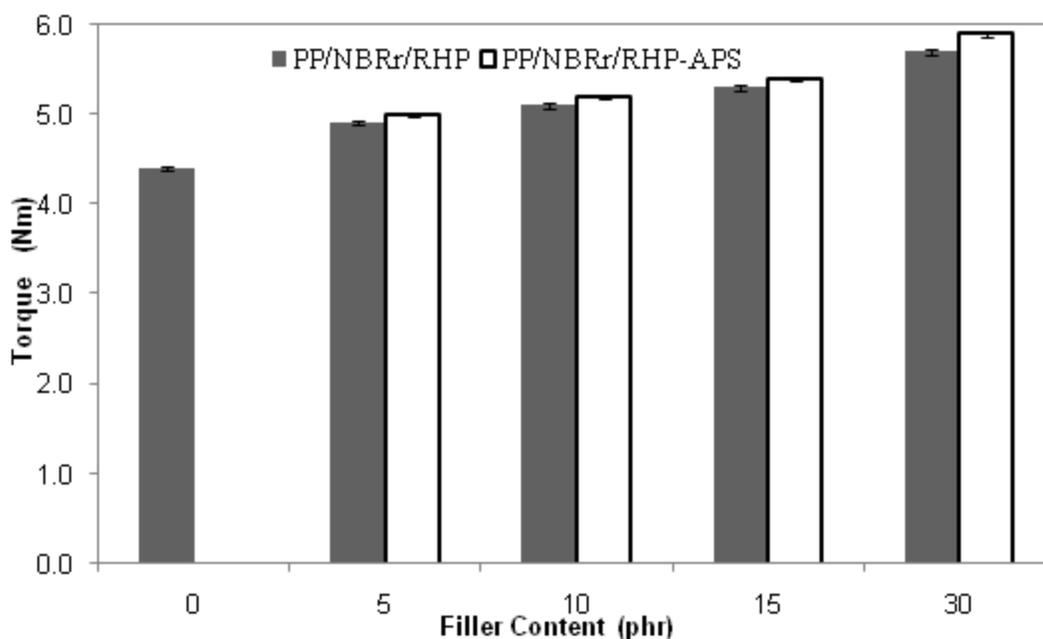
Note: (phr)-part per hundred resin

\* Similar biocomposites but without  $\gamma$ -APS treatment were also prepared.

## RESULTS AND DISCUSSION

### Mixing and Processing Torque

Results for processing stabilization torque of PP/NBRr/RHP biocomposites after 9 minutes are shown in Fig. 2. An initial torque value of 4.4 Nm was recorded in the absence of RHP filler in the mixtures. However the stabilization torque increased gradually as filler loading increased. Increasing RHP filler loading further enhances interactions between RHP filler and the PP/NBRr matrix, thus reducing the polymer chain's mobility (Premalal et al. 2002; Ismail and Mega 2001).



**Fig. 2.** Processing stabilization torque of PP/NBRr/RHP biocomposites after 9 minutes

At similar filler loading,  $\gamma$ -APS treated RHP showed a higher stabilization torque, which indicates an increase in the viscosity of the molten materials during mixing. These findings were due to enhanced interactions between filler and matrix (as shown later in the SEM study). We have reported a similar observation in our previous study involving the PP/NBRr/RHP with PPMAH (Polypropylene Maleic Anyhdride) compatibilization (Ismail et al. 2010a), whereby higher stabilization torque resulted from composites with good filler-matrix interfacial bonding between the RHP and PP/NBRr matrices.

### Tensile Properties

Figure 3 shows tensile strength of PP/NBRr/RHP biocomposites with and without  $\gamma$ -APS treated RHP. Tensile strength was found to be reduced with the incorporation of RHP. The reduction of tensile strength may due to poor adhesion between polar RHP and non polar PP matrix's.

This also could also be due to the incapability of the irregular RHP filler shape to support stress and transmit it to polymer matrix. The reducing trend in tensile strength could be also explained by the agglomeration of RHP filler particles and poor compatibility of the polymer at the interphase (Ismail et al. 2001a).

However,  $\gamma$ -APS treated RHP composite exhibited comparatively higher tensile strength when compared to the untreated RHP. The presence of  $\gamma$ -APS coupling agent significantly improved the adhesion at the fiber-matrix interface, as shown later in the morphological study. The strong interfacial bonding provides better stress distribution which results in good tensile strength of PP/NBRr/RHP biocomposites.

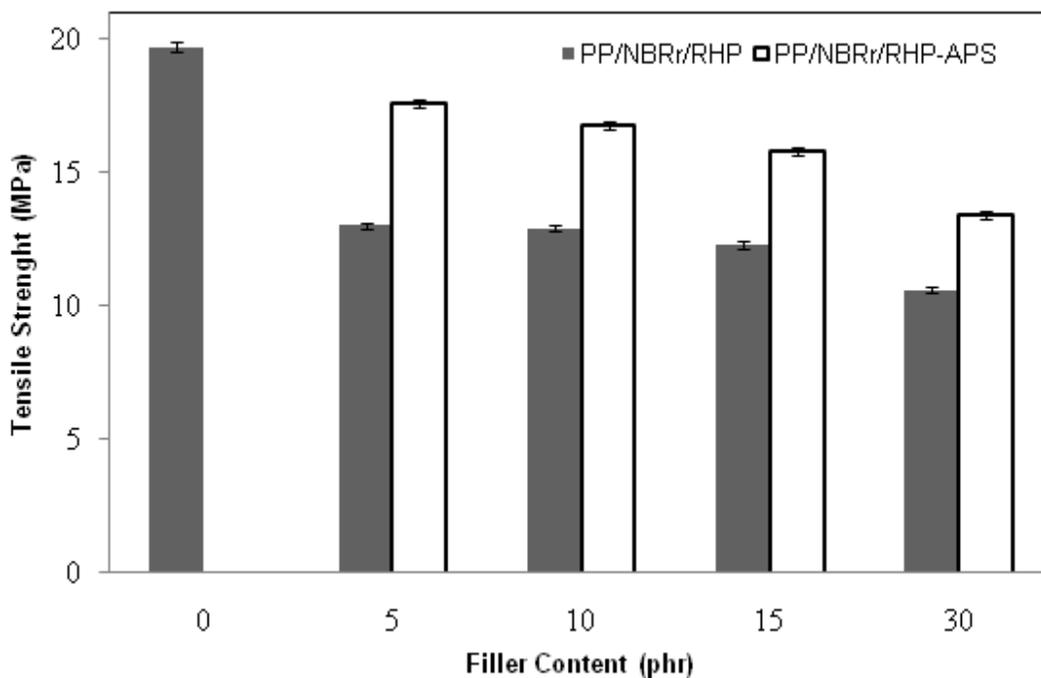


Fig. 3. Tensile strength of PP/NBRr/RHP biocomposites with and without  $\gamma$ -APS treated RHP

The tensile modulus of PP/NBRr/RHP biocomposites with and without  $\gamma$ -APS treated RHP is shown in Fig. 4. The addition of RHP increased the tensile modulus of composites with and without  $\gamma$ -APS treatment. The incorporation of RHP filler into the matrix improved the stiffness of the composites. The addition of RHP filler into the PP/NBRr matrices reduces the PP chains mobility, consequently producing more rigid composites. At a similar filler loading, tensile modulus for  $\gamma$ -APS treated RHP composites exhibited much higher tensile modulus. Similar findings were reported by Liu et al. (2009), whereby higher rigidity of biocomposites exhibit higher tensile strength in surface modified jute fibers with silane coupling agent. According to Bledzki et al. (1996), an increase of 50% in tensile modulus was reported in natural vegetable fiber due to better interactions between the matrix and filler with the presence of a silane coupling agent.

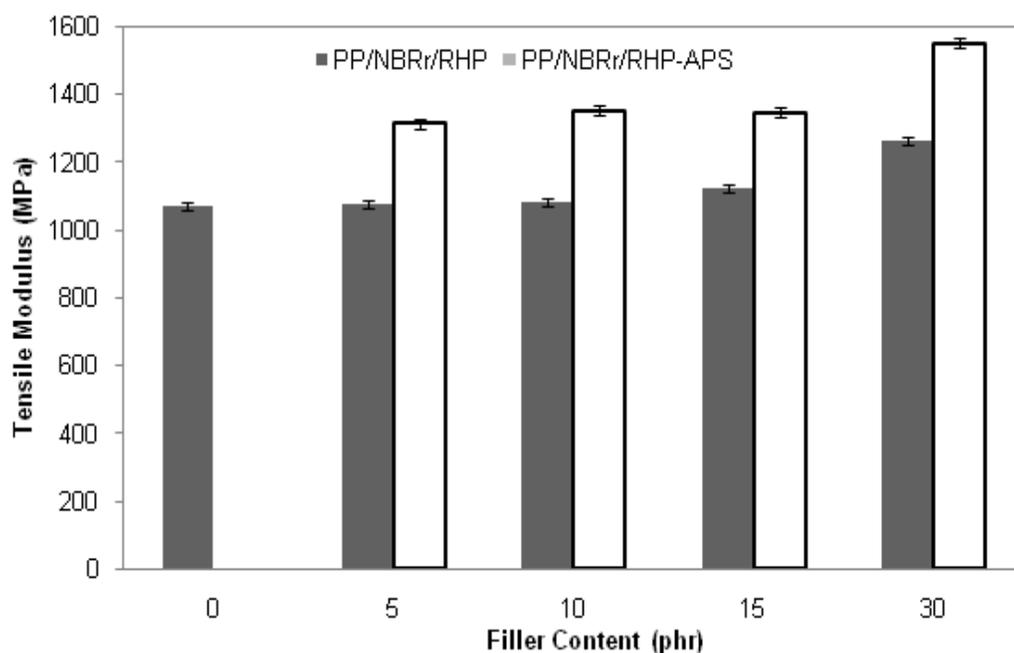


Fig. 4. Tensile modulus of PP/NBRr/RHP biocomposites with and without  $\gamma$ -APS treated RHP

Figure 5 shows elongation at break of PP/NBRr/RHP biocomposites with and without  $\gamma$ -APS-treated RHP. It can be seen that the incorporation of RHP into the PP/NBRr composites resulted in a reduction of elongation at break. A decreased elongation at break at a higher RHP fiber loading may be due to the increase of stiffness and brittleness of the biocomposites. However, at a similar filler loading, the elongation at break for PP/NBRr/RHP composites with  $\gamma$ -APS treated RHP was lower than the composites with untreated filler. As explained earlier, this may be due to the presence of coupling agent, which enhances the matrix and filler interactions resulting in lower elongation at break. Hong et al. (2008) reported that silane treatment of natural fiber yielded a stronger interfacial adhesion, resulting in low elongation at break in composites. Decreased polymer chain mobility resulted from good adhesion of RHP filler and PP/NBRr matrix in the presence of  $\gamma$ -APS coupling agent. The overall, tensile properties

of the PP/NBRr/RHP composite were found to be decreasing with increasing RHP content. RHP content resulted in increase composite stiffness and lower composite elongation due to the brittle nature of RHP. However in  $\gamma$ -APS treated RHP composites, enhance tensile strength was observed due to good adhesion of RHP and PP/NBRr matrices.

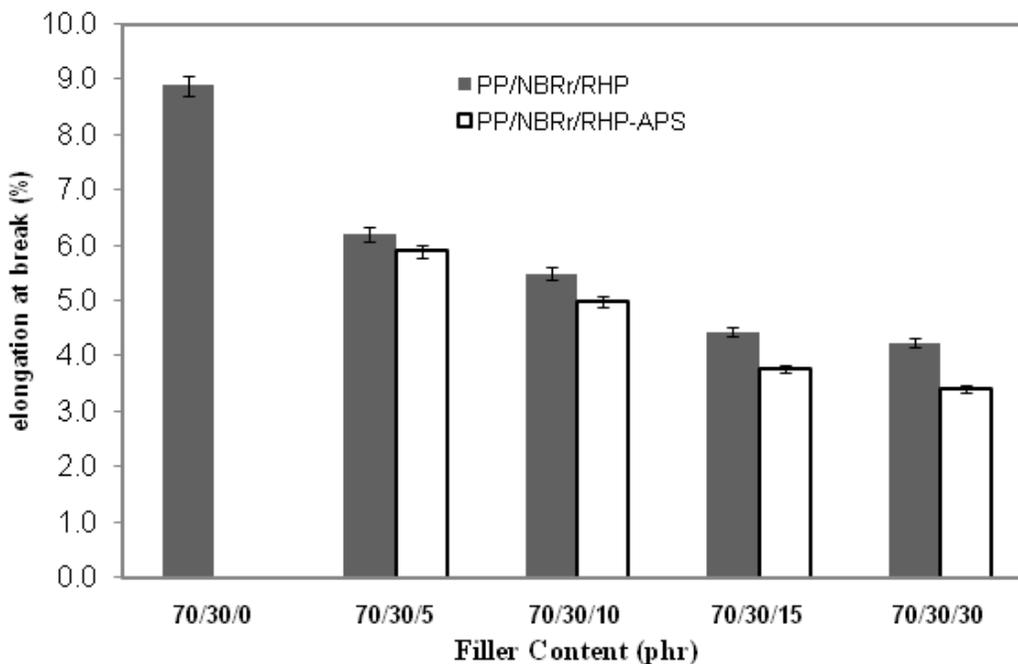


Fig. 5. Elongation at break of PP/NBRr/RHP biocomposites with and without  $\gamma$ -APS treated RHP

### Water Uptake

Figure 6 shows the plot of PP/NBRr/RHP biocomposites with and without  $\gamma$ -APS-treated RHP. The results show that water uptake increased as RHP filler content increased for average five sample replicates. Natural fibers are highly hydrophilic due to hydroxyl group of polysaccharides found in cellulose, which are able to form hydrogen bonds between water and the RHP fiber. As filler content increased, the number of hydrogen bonds between organic components and water molecules also increased. The increasing amount of voids between RHP and the PP/NBRr matrices also allows more water uptake. Similar findings have been reported by other researchers (Bledzki et al. 1996; Ismail et al. 2001; Ishak et al. 2001; Razavi et al. 2006; Ismail et al. 2010b). However, in our previous work, PP/NBRr/RHP was found to exhibit 2-stage uptake behavior (Ismail et al. 2010b). The RHP in PP/NBRr matrices uptakes water much easier compared to the RHP particles encapsulated by NBRr. It can be seen in Fig. 6 that filler content had a significant effect on water uptake properties of the composites. It indicates that, the higher the filler content, the higher the percentage of equilibrium water uptake (Razavi et al. 2006). This is due to the fact that as the filler content in composite increases, the number of free OH groups on the lignocellulosic fibers increases. Free OH groups come in contact with water through hydrogen bonding, which results in water

uptake and weight gain in the composites. At a similar filler loading, it can be seen that the composites with  $\gamma$ -APS treated filler showed a lower water uptake when compared to the composites with untreated filler. This result provides a clear indication that the RHP treatment with  $\gamma$ -APS enhances the filler-matrix interactions at the interface, thus decreasing the amount of equilibrium water uptake by the composites. Ismail et al. (2001) reported similar findings whereby lower absorption in PP/NR/RHP ash was attributed to the ability of the chemical to form a protective layer at the interfacial zone and consequently prevent the direct diffusion of water molecules into silane-treated filler composites.

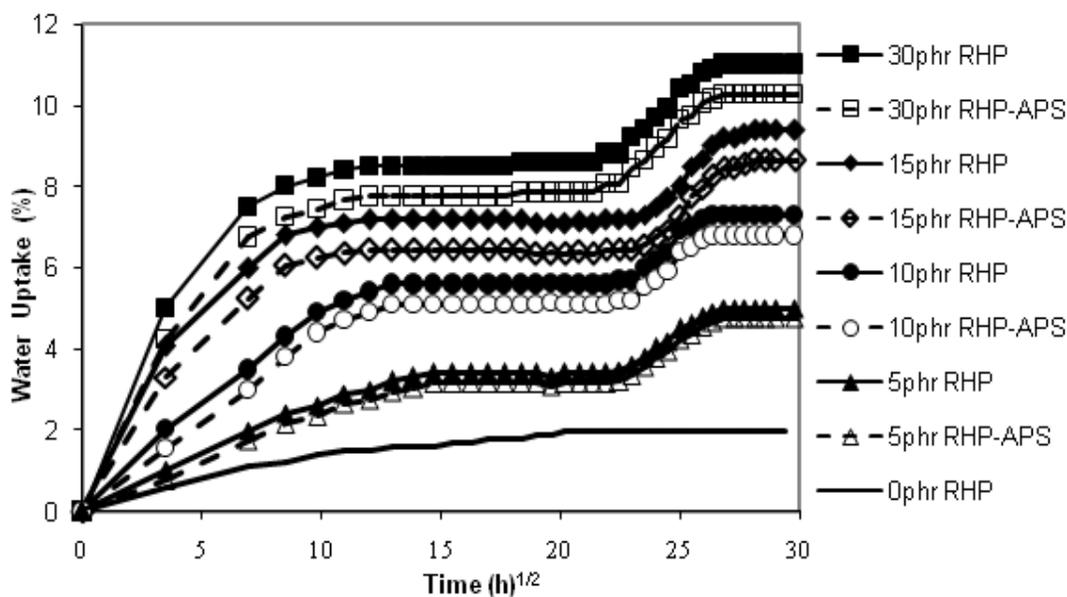
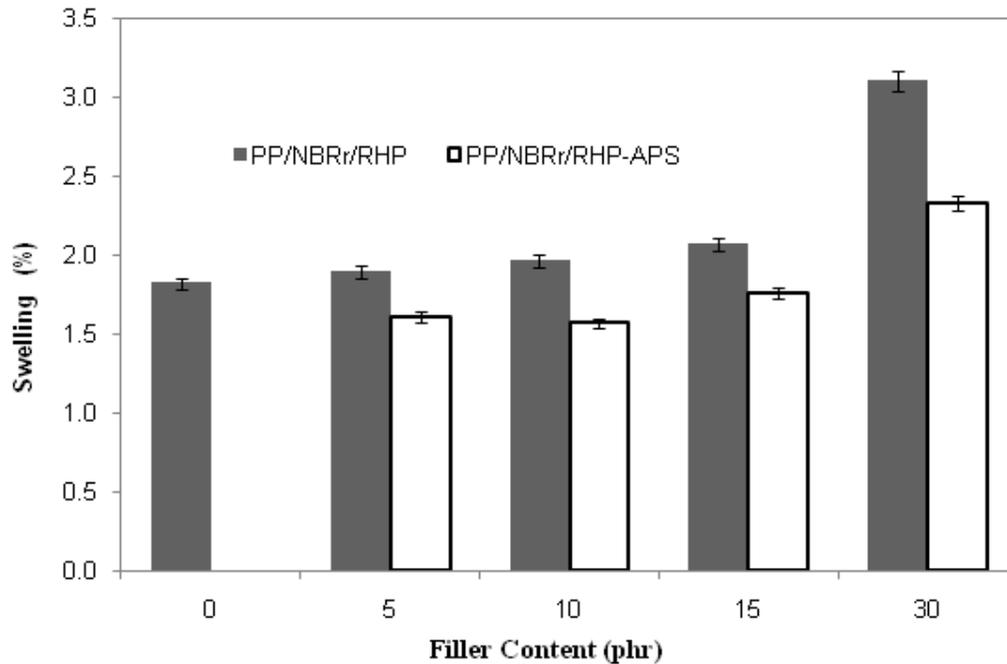


Fig. 6. Plot of PP/NBRr/RHP biocomposites with and without  $\gamma$ -APS treated RHP

### Swelling Test

A swelling test in oil was done to evaluate the capacity of oil uptake in the composites. Inhibition of oil uptake is primarily associated with level of filler-matrix adhesion. Figure 7 shows the swelling percentage (%) of PP/NBRr/RHP biocomposites with and without  $\gamma$ -APS treated RHP in ASTM oil No. 3 for 70 hours. It was found that the swelling percentage increased at higher RHP content in PP/NBRr/RHP biocomposites. This was due to the properties of the lignocellulosic fiber (RHP), which absorbs oil on its surface (Satyanarayana et al. 2009; Nabi et al. 1999; Premalal et al. 2002). However, for similar composites with the same composition, the  $\gamma$ -APS treated RHP composites exhibited lower swelling percentage due to better interaction of RHP particles in the continuous PP/NBRr matrix. The interaction limits the penetration of oil into the treated composites matrix. This might be due to the ability of  $\gamma$ -APS coupling agent to cause better interaction and form a protective layer at the interfacial zone, which consequently prevents the direct diffusion of oil molecules into the composites. A similar observation was reported using maleic-anhydride grafted polypropylene (PPMAH) as a compatibilizer in PP/NBRr/RHP composites (Ismail et al. 2010a). Hence, this composite

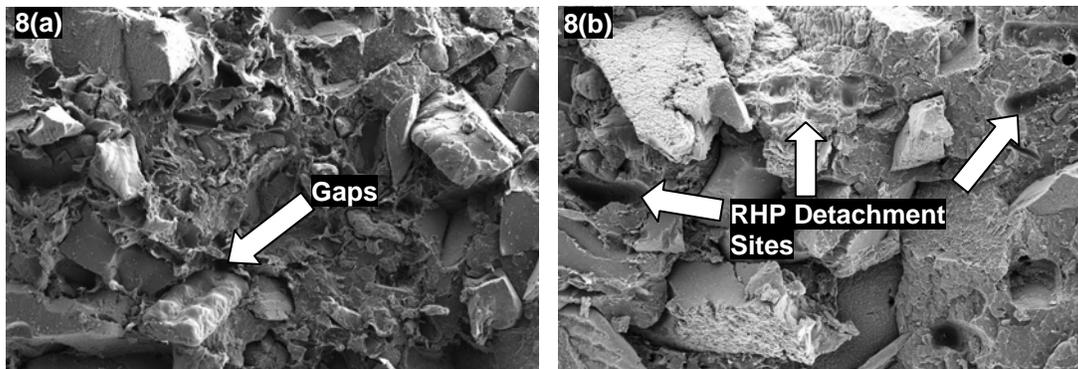
may be well utilized for commercial fabrication of partition boards and photo frames due to its comparable tensile properties and non-oil uptake characteristics.



**Fig. 7.** Swelling (%) of PP/NBRr/RHP biocomposites with and without  $\gamma$ -APS treated RHP in ASTM oil No.3 for 70 hours

### Morphology

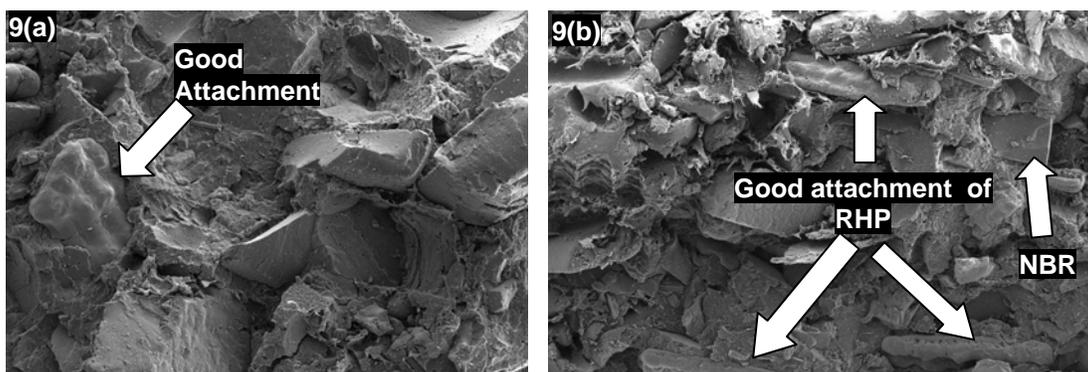
Figure 8(a-b) shows SEM micrographs of PP/NBRr/RHP biocomposites without  $\gamma$ -APS treated RHP (100X) at filler content of 10 phr and 30 phr respectively. The SEM micrographs show that poor adhesion of RHP to the PP/NBRr matrix is the main factor for the reduction of the tensile strength with an increase of filler content in the composites. The incorporation of RHP filler into the PP/NBRr matrix increases the rigidity of the material thus reducing the ductility of the composites.



**Fig. 8(a-b).** SEM micrographs of PP/NBRr/RHP biocomposites without  $\gamma$ -APS treated RHP (100X) at filler content: 10 phr and 30 phr respectively

Figure 8a shows some noticeable gaps between the filler and matrix, which is evidence of poor adhesion between the filler and matrix. Figure 8b shows that the RHP fillers were pulled out from the matrix or remained loosely in the matrix, which resulted in more detachments of RHP filler from the matrix. This is due to the poor dispersion and poor wettability of the RHP filler with the PP/NBRr matrices.

Figure 9(a-b) shows SEM micrographs of PP/NBRr/RHP biocomposites (100X) with  $\gamma$ -APS treated RHP at filler content 10 phr and 30 phr, respectively. The adhesion between filler and matrix was enhanced. The morphology showed more fractures and higher toughening effect when compared to the uncompatibilized composites. The better fiber-matrix adhesion can be seen by the fact that fewer fibers were pulled out. The adhesion between  $\gamma$ -APS treated RHP filler and PP/NBRr matrices was strong enough that the fiber was rather broken under stress. This explained the improvement in tensile properties of the PP/NBRr/RHP-APS biocomposites. This proved that RHP filler treatment with  $\gamma$ -APS coupling agent was effective at enhancing the properties for PP/NBRr/RHP biocomposites.



**Fig. 9(a-b).** SEM micrographs of PP/NBRr/RHP biocomposites (100X) with  $\gamma$ -APS treated RHP at filler content 10 phr and 30 phr respectively

## CONCLUSIONS

1. The addition of rice husk powder into PP/NBRr/RHP biocomposites increased the processing torque, tensile modulus, water absorption, and swelling in oil, but it decreased the tensile strength and elongation at break of the biocomposites.
2. The  $\gamma$ -APS treated RHP composites exhibited higher processing torque, tensile strength, tensile modulus, and lower elongation at break when compared to untreated RHP composites due to the strong bonding between  $\gamma$ -APS treated RHP filler and PP/NBRr matrices.
3. The  $\gamma$ -APS treatment on RHP improved the adhesion between hydrophilic RHP fiber and the hydrophobic PP/NBRr polymer matrices, which led to reduction of water and oil absorption into PP/NBRr/RHP/ biocomposites.

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