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Physical Properties of Rice Husk Fiber/Natural Rubber Composites

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Abstract. In this study, rice husk fiber (RHF) was used as a reinforcing filler for natural rubber (NR). NR composites were prepared at various RHF contents, *i.e.,* 10, 20, 30, 40 and 50 phr. Sulfur conventional vulcanization was used. Effect of RHF content on cure characteristics, mechanical properties and morphological properties of NR composites were investigated. The results showed that scorch and cure times of RHF/NR composites were not affected by increasing RHF content. Crosslink density, tensile strength, elongation at break and tear strength of NR composites slightly decreased with increasing RHF content whereas M100 and M300 of the composites slightly increased with increasing RHF content.

Introduction

Rice husk (RH) is abundantly available in Thailand as a byproduct from the rice milling process. Only a minor portion of the rice husk is reserved as animal feed, bedding material or household fuel while the huge quantities are burned in fields leading to environmental pollution. Various researches attempted to expand the utilization of RH in industries, e.g. as pozzolanic material to enhance the lime treatment of degraded soil, as filler to increase compressive strength in cement, particleboard material and as a source for activated carbon [1]. In addition, RH can be used as reinforcing filler for natural rubber (NR). RH contains 45% cellulose, 19% hemicelluloses, 19.5% lignin and 15% silica by weight [2,3]. Besides cellulose, RH has high silica content as compared with other natural fibers [3]. Both cellulose and silica in RH can provide strength to the fiber as well as improve mechanical properties of NR. In addition, RH has many advantages similar to other natural fibers such as low density, low price, high strength, stiffness and non abrasiveness [4]. The utilization of RH as reinforcing filler for NR is an approach to obtain value added products from an agricultural waste.

Materials and Methods

Materials

Natural rubber (STR 5L) was purchased from Thai Hoa Rubber Public Co., Ltd. Rice husk (RH) was purchased from a local rice mill in Nakhon Ratchasima, Thailand. N-cyclohexyl-2-benzothiazole-2-sulphenamide (CBS), stearic acid, zinc oxide (ZnO), and sulfur (S) were supplied by Channel Chemicals Co., Ltd.

Preparation of rice husk fiber (RHF)

RH was washed thoroughly with tap water to remove the adhered soil and dust, and then dried in open air. The dried rice husk was ground using a grinding machine (RETSCH/SR200) and sieved with a sieve shaker (RETSCH/AS200). The rice husk fiber (RHF) retained in sieve size ranging between 150-300 μm was used. The RHF was dried in an air oven at 80ºC overnight to discharge the moisture before compounding.

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Preparation and characterization of RHF/NR composites

To prepare NR composites, NR was masticated for 5 min on a two-roll mill (CHAICHAREON) to reduce its viscosity. Then, stearic acid, zinc oxide, CBS, RHF, and sulfur were added, respectively. Total mixing time was 20 min.

Cure characteristics of RHF/NR composites were determined using a moving die rheometer (MDR) (GOTECH/GT-M200F) at a temperature of 150 ºC. Scroch time (T_s) and cure time (T_{c90}) of the RHF/NR composites were determined.

RHF/NR composites were vulcanized using a compression molding machine (LAB TECH/L320) at 150 ºC. The vulcanized time was based on the cure time obtained from MDR. The composite sheet was cut into dumbbell shaped specimens with a die cutter (Type C).Tensile and tear properties of RHF/NR composites were determined according to ASTM D 412–06a and ASTM D 624, respectively.

Crosslink density of RHF/NR composites was measured according to ASTM D 6814. The vulcanized NR composites were swollen in toluene at 27˚ C for 72 h to obtain equilibrium swelling stage. Crosslink density was calculated by the Flory-Rhener equation [5].

Dispersion of RHF in RHF/NR composites and surface morphologies of tensile fracture surfaces of the composites were characterized using a scanning electron microscope (SEM) (JEOL/JSM-6400) at 10 kV. Samples were coated with gold before analysis.

Result and discussions

Cure characteristics and crosslink density

Cure characteristics and crosslink density of NR and RHF/NR composites are shown in Table 1. NR showed longer scorch and cure times than NR composites. The incorporation of RHF in NR matrix slightly decreased scorch and cure times of NR composites. The similar results were observed by Jacob *et al* [6], Ismail and Khalil [7] and Geetham *et al* [8]. They suggested that the addition of fiber in rubber increased the mixing time of rubber compounds leading to the high heat buildup during compounding and the formation of premature crosslinking in rubber composites. However, increasing RHF content in the NR composites had no effect on their scorch and cure times.

As shown in Table 1, gum NR had the highest crosslink density. With increasing RHF content, crosslink density of NR composites slightly decreased. Similar observation was reported by Zeng *et al* [9]. The decrement of crosslink density of NR composites was attributed to the adsorption of the accelerator by hydroxyl group of the fiber. RHF content had slightly effect on crosslink density of the NR composites. The NR composites containing RHF of 20 phr provided the lowest crosslink density.

Table 1 Cure characteristics of NR and RHF/NR composite

Samples	T_s (min)	T_{c90} (min)	Crosslink density (*10^4 mol/cm^3)
NR	5.24	7.54	0.85
10RHF/NR	4.06	7.24	0.76
20RHF/NR	3.53	7.48	0.66
30RHF/NR	4.06	7.17	0.73
40RHF/NR	4.11	7.16	0.71
50RHF/NR	4.17	7.25	0.79

Mechanical properties

Properties of natural fiber reinforced rubber composites depend on fiber dispersion, fiber-rubber adhesion, fiber orientation, fiber aspect ratio and fiber content, *etc*. The incorporation of natural fiber in rubber improved modulus of the rubber composites while the composites maintained elasticity of the rubber matrix [10]. Tensile strength and elongation at break of NR and RHF/NR composites are shown in Fig. 1. Gum NR represented the highest tensile strength and elongation at break. The high strength of NR was because of the high crosslink density of the vulcanized NR as well as its strain-induced crystallization behavior [6]. For RHF/NR composites, tensile strength and elongation at break of NR composites slightly decreased with increasing RHF content. This was because RHF hindered the arrangement of rubber molecules leading to the reduction of strain-induced crystallization of NR. In addition, the reduction of both tensile strength and elongation at break of the composites was attributed to the incompatibility between hydrophilic RHF and hydrophobic NR.

Modulus at 100 %strain (M100) and modulus at 300% strain (M300) of NR and RHF/NR composites are shown in Fig. 2. Modulus of gum NR was lower than those of NR composites. In addition, M100 and M300 of RHF/NR composites slightly increased by adding RHF. This was because of the high stiffness of RHF.

Tear strength of NR and RHF/NR composites are shown in Fig. 3. NR represented the highest tear strength. The incorporation of RHF in NR reduced tear strength of RHF/NR composites. In addition, tear strength of the composites decreased as RHF content increased. This was because RHF obstructed strain-induced crystallization of NR leading to the decrement of tear properties of NR composites.

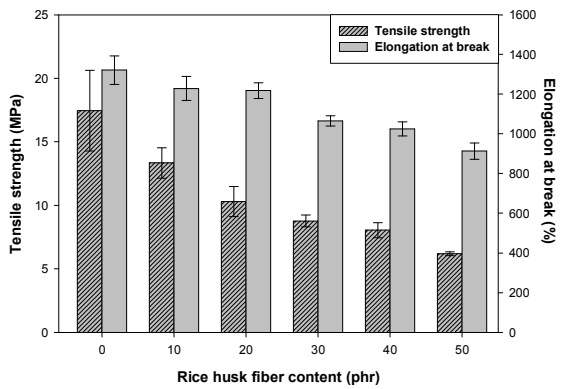

Figure 1 Tensile strength and elongation at break of NR and RHF/NR composites.

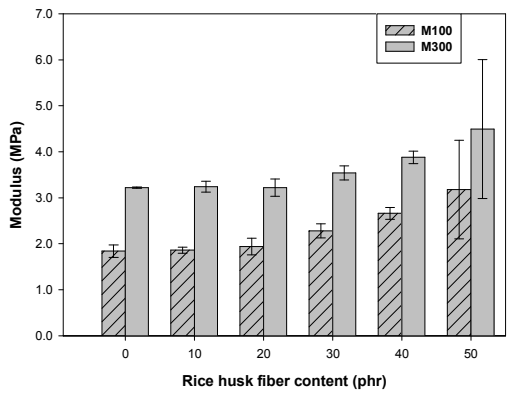

Figure 2 M100 and M300 of NR and RHF/NR composites.

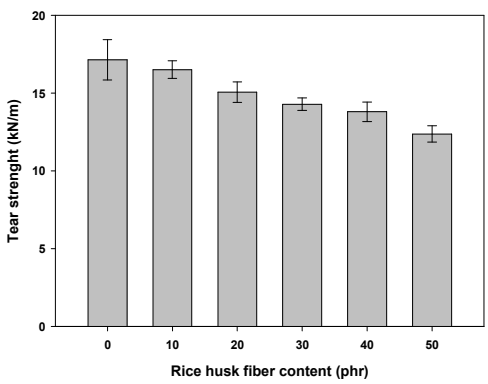

Figure 3 Tear strength of NR and RHF/NR composites.

Morphological properties

SEM micrograph of tensile fracture surface of NR and RHF/NR composites are shown in Fig. 4. Gum NR showed the smooth surface. NR composites exhibited a rough surface because of the presence of fiber. NR composites containing 10 phr RHF showed uniform dispersion of RHF while the composites containing 50 phr RHF illustrated agglomerated RHF in rubber matrix. In addition, all NR composites exhibited numerous holes due to the fiber pullout and large gap between RHF and NR matrix. These implied the poor interfacial adhesion between RHF and NR matrix. The SEM micrographs of NR composites were in good agreement with the reduction in tensile properties of NR composites.

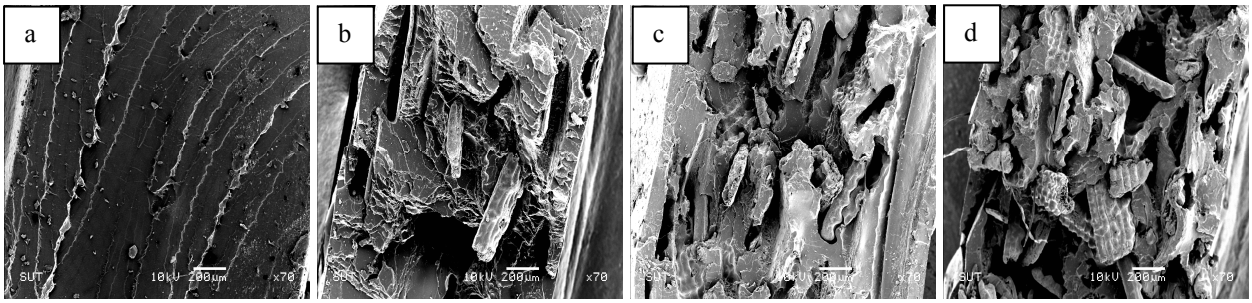

Figure 4 SEM micrographs of tensile fracture surface of (a) NR and (b) RHF/NR composites containing RHF of 10 phr, (c) 30 phr and (d) 50 phr.

Summary

Effect of RHF content on cure characteristics, mechanical properties and morphological of RHF/NR composites were studied. Gum NR showed the highest crosslink, tensile strength, elongation at break and tear strength. Tensile strength, elongation at break and tear strength of RHF/NR composites slightly decreased with increasing RHF content. RHF/NR composites with 50 phr RHF represented the highest M100 and M300. SEM micrographs showed that RHF and NR had poor interfacial adhesion which caused the decrement of mechanical properties of NR composites.

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