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Physical and Mechanical Properties of Natural Rubber Composites Filled with Rice Husk and Rice Straw

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Abstract

In this work, the natural rubber (NR) composites filled with two wastes from rice cultivation, such as rice husk (RH) and rice straw (RS) were studied. Different sizes of each waste were prepared which were RH-S, RH-L, RS-S, and RS-L and used as fillers in composites. The effects of filler types and contents (15, 30 and 45 phr) on the physical and mechanical properties of NR composites were studied. The results showed that NR composites reinforced with RS and RH had lower densities than that of neat NR. The types, sizes, and contents of fillers showed minor effects on the composite's density. Interestingly, due to the difference in the chemical composition of RS and RH, the trend of water absorption and an oil absorption of NR composites filled with RSs and RHs were different. RSs/NR composites showed a higher percentage of water absorption but RHs/NR systems showed higher oil uptake. Using larger size of fillers (RS-L and RH-L) in composites displayed higher tendency of both water and oil absorptions than that using smaller fillers. The percentage of water absorption of all types of composites was higher than that of NR sheet. This value increased with increasing filler content. In contrast, the addition of filler loading resulted in decreasing the percentage of oil absorption of all types of composites. Comparing with neat NR, Young's moduli of all types of composite were found improved with an increase of filler loading. All RSs/NR composites were dramatically higher in effectiveness. However, a decrement of tensile strength of all composites was observed. SEM micrographs clearly revealed the lack of interfacial interaction between fillers and NR that showed the gap and hole. These evidences supported the composite properties that mentioned above.

Keywords: Natural rubber composites; Natural latex, Rice straw; Rice husk; Biocomposite

1. Introduction

Like other agricultural countries, Thailand has a large amount of agricultural wastes which are not used economically and mostly left in the field such as rice straw, rice husk, sugarcane leaves, pineapple leaves, and corn husk. Due to the uselessness of these wastes and some needed quickly clean before turnaround crop, farmers prefer to burn these residues after plant harvest resulting in the occurrence of several environmental problems like air pollution, PM 2.5 emission, and soil moisture loss [1 – 2]. To solve those problems and make efficient use of agricultural wastes, an approach with optimization of farmers and industries should be considered. In recent years, owing to the plastic waste crisis, the trend of green technology as biocomposites has much attention to developing for replacing petrochemical-based materials [3 – 5]. Natural resource materials like agricultural wastes were attractive to make an ecofriendly composite material and it could be the most suitable way to solve environmental problems related to agricultural and plastic waste and also increase the income of farmers.

Thailand is one of the world's largest rice exporters [6]. For rice production, it is normally providing two major types of lignocellulosic biomass resources which are rice straw (RS) and rice husk (RH). RS is produced when harvesting paddy while RH is generated during the first stage of rice milling. Because of the abundant rice production wastes, they have attractive potential in terms of natural fiber sources and both are mainly composed of cellulose, hemicellulose, and lignin [7 – 8]. Many reviews [9 – 12] reported that RS and RH can be used as fillers in composite materials in various petroleum-based polymer matrices such as polyester, polypropylene, polyethylene that provided the modulus enhancement and light weight, and probably obtained greener materials. To get more attention on the use of renewable resources and environmental problem awareness, the approach to use the natural origin matrix replacing the petrochemical-based matrix is interesting. Natural rubber latex (NR latex) is one of the natural polymers extracted from a rubber tree by tapping. Thailand, Indonesia, and Vietnam are the largest producers of natural rubber in the world [13]. NR latex can be used in various applications such as tubing, rubber nipple, surgical glove, wood binder and asphalt modification [14]. However, NR is normally low modulus at low strain region and high flexible. To deal with the versatile application of NR and its end-of-life consideration, it is interestingly to know the improvement of the NR mechanical properties by using biodegradable fillers.

As mention above, Thailand has abundant rice production residues and is one of the largest NR latex producers. Thus, the idea to develop biocomposites between rice wastes and NR latex is the appropriate way that may be beneficial, environmentally friendly, and economically practical for farmers and industries. In this work, the biocomposites based on NR latex with two wastes from rice production (RS and RH) were prepared, and also, all composites were evaluated based on their effects of filler types, filler sizes, and filler contents on the physical and mechanical properties of composites, and the relationship between these properties and morphology in composites.

2. Materials and Methods

Materials

RS was collected from a paddy field in Ban krang District, Amphor Muang, Phitsanulok Province, Thailand. RH was obtained from the PLC-RMUTL rice mill, Phitsanulok Province, Thailand. High ammonia NR latex with 60% of dry rubber content was supplied by Viroonkit Industry CO., LTD, Nakhonratchasima Province, Thailand.

Filler preparation

As-prepared RS and RH were washed with tap water to remove sand and dirt and then dry in a hot air oven 80 °C for 24 h. For RS fillers, RS was chopped about 2.50 cm and grounded with high-speed grinding for 1 min. Grounded RS was the sieve on screen 30 mesh. RS particles that pass through a 30 mesh screen were called RS-S and the other one that remains on screen was coded RS-L. The average diameter and average length of RS-S were 0.03 cm and 0.19 cm, respectively, while that of RS-L were 0.10 cm and 0.79 cm, respectively. Two types of RH filler were also prepared. RH-S was prepared similarly to RS-S preparation while RH-L was named the as-prepared RH. The average dimension of RH-S was 0.05 cm diameter and 0.12 cm length, and RH-L having an average diameter and length of about 0.20 cm and 0.85 cm, respectively.

Chemical and thermal analysis of rice straw and rice husk

The cellulose, hemicellulose and lignin of RS, and RH was determined using forage fiber analysis [15] which was performed in the Laboratory of Nakhonratchasima Animal Nutrition Research and Development Center, Nakhon Ratchasima, Thailand. Thermogravimetric analyzer (TGA) was carried out with Mettler Toledo TGA instrument (Model SDTA851, Switzerland) for rice straw and rice husk to evaluate their decomposition temperature characteristics which was analyzed in Rubber Technology Research Center, Nakhon Pathom, Thailand.

Composite preparation

Elastomer mixture between high ammonia NR latex with different types of fillers was mixed in a mixing a bowl. Three filler contents which were 15, 30 and 45 parts per hundred rubber (phr) were applied. Each mixture was stirred at low speed for 3 min to get a homogeneous compound. Each composite compound was poured into a stainless tray and then dried in a hot air oven at 60 °C for 6 h. The 30 g of each mixture was pressed at 150 °C with 1500 psi for 15 min to form a sheet of 3 mm thickness.

Composite characterization

Composite density

The composite densities were performed with a density determination kit of the analytical balance (XS105, Mettler Toledo, Greifensee, Switzerland). The specimens were used in the size of 10 × 20 × 3 mm. Five replicates were tested and the densities of composite samples were calculated. The mass of the specimen was weighed in air and distilled water at 25 °C and the composite density was calculated according to the equation (1):

$$D=\frac{M_1}{M_1-M_2}\times D_W \tag{1}$$

where D and D_w are the densities of the composite sample and the water at the measured temperature and M_1 and M_2 are the weights of the sample in air and in distilled water, respectively.

Water absorption and oil absorption

Water absorption of NR composites was evaluated by immersing the composite samples in distilled water at room temperature. Weight gain measurement was evaluated after 1, 2, 4 and 7 days, respectively. The percentage of water absorption was calculated using the following equation (2):

$$\%\text{Water absorption}=\frac{M_f - M_i}{M_i}\times 100 \tag{2}$$

where M_i and M_f are the initial weight of the sample before immersion and the final weight of the sample after immersion in water for a determined time, respectively.

The oil absorption of NR composites was investigated by immersion of specimens in cooking oil at room temperature. Weight gain determination was collected after 1, 2, 4 and 7 days, respectively. The percentage of oil absorption was calculated according to the equation (3):

$$\%\text{Oil absorption}=\frac{M_f - M_i}{M_i}\times 100 \tag{3}$$

where M_i and M_f are the initial weight of the specimen before immersion and the final weight of the specimen after immersion in cooking oil for a determined time, respectively.

Mechanical testing

Tensile testing was carried out on a tensile testing machine (M350-10CT, Testometric, UK) with a 1 kN load cell. The specimens were 3 mm thick, 10 mm wide and 90 mm long. The gauge length was 50 mm and the extension rate was 50 mm min^{-1}. For each composite, five specimens were tested and the average Young's modulus and tensile strengths were estimated.

Scanning electron microscopy (SEM) analysis

The morphology of the tensile fracture surfaces of the specimen was analyzed by using SEM (TM-1000, Hitachi, Japan). Each tensile fracture specimen was observed at 50x and 100x magnifications, respectively.

3. Results and Discussion

Like other natural fibers, RS and RH contain a combination of cellulose, hemicellulose, and lignin [7]. The chemical composition of RH and RS are different in each content as summarized in Table 1. These contents of RH and RS are similar to those reported by other work [16]. Cellulose and lignin contents of RH display higher than that of RS whereas hemicellulose shows higher in RS. Normally, cellulose and hemicellulose are highly hydrophilic while lignin is more hydrophobic. Thus, the different proportion of chemical composition may be resulting in the difference in the hydrophilic nature of natural fiber [17].

The thermogravimetric analysis curves (TGA) and derivative thermogravimetric (DTG) curves of fillers are shown in Fig.1 which reveal the decomposition temperature characteristics of RH and RS. The initial decomposition temperature (T_{in}) and the decomposition temperature at maximum loss rate (T_{max}) could be used to evaluate the thermal stability of materials. Comparing with the thermal decomposition of dried natural latex [18], RS and RH showed lower thermal stability. T_{in} of RS and RH were 200 °C and 250 °C, respectively which are assigned to the degradation of hemicellulose and lignin [8, 19]. T_{max} of RS and RH were 330 °C and 366 °C, respectively which is demonstrated in the degradation of cellulose [20]. Thus, the composite preparation temperature should use lower than 200 °C.

In fact, natural fiber has attracted in using as reinforcement or filler in composite due to its lower densities compared with commercial fillers. The density of natural fiber is usually between 0.60 – 1.60 g cm^{-3} [7]. For RS and RH, it has been reported that their densities were 1.60 g cm^{-3} [9] and 1.30 g cm^{-3} [10], respectively. The densities of NR composites with RS and RH are shown in Table 2. All composite samples had lower densities comparing with neat NR and did not follow the rule of mixture. This may be due to the gap between the hydrophobic matrix and hydrophilic filler [10, 21]. The filler contents slightly affected the density of NR composites that showed an increase with filler content increased. Composites with fibrous fillers (RS-S and RS-L) showed insignificantly different in densities of both systems. In the case of composites with particulate fillers, RH-S composites displayed higher density than that of RH-L. This may be due to smaller particulate filler leading to easier natural latex penetrated and wetted with fine filler providing the occurrence of smaller gaps between RH-S filler with latex.

Table 1 Chemical compositions of Rice husk and Rice straw.

Fillers	Dry matter (%)	Cellulose (%)	Hemicellulose (%)	Lignin (%)
Rice husk (RH)	93.25 ± 0.23	43.41 ± 0.06	24.14 ± 0.08	15.43 ± 0.13
Rice straw (RS)	89.97 ± 0.12	35.45 ± 0.14	33.56 ± 0.08	4.12 ± 0.04

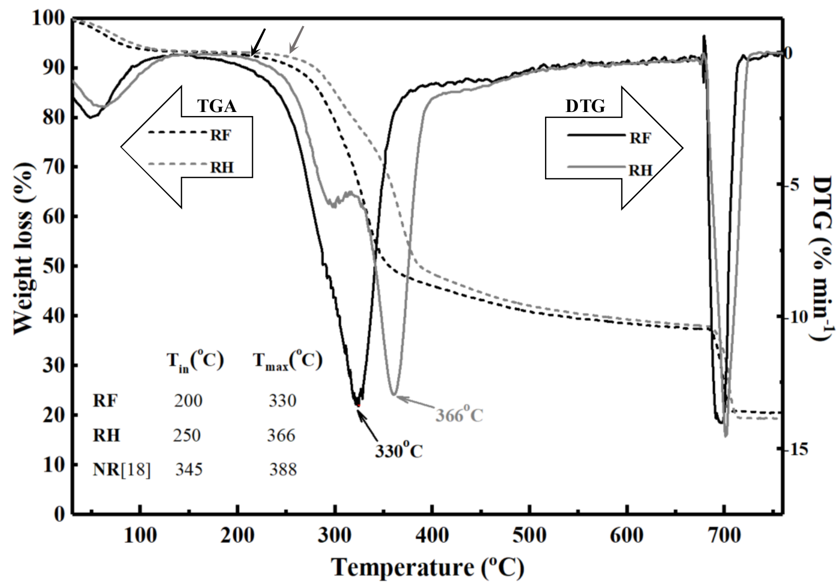

Fig. 1 Thermogravimetric analysis (TGA) curves (dashed line) and derivative thermogravimetric (DTG) curves (solid line) of RH (gray) and RS (black).

Table 2 Density of various types of NR composite sheets.

Sample/ Filler content	Density (g cm^{-3})			
	0 phr	**15 phr**	**30 phr**	**45 phr**
Neat NR	0.93 ± 0.01	-	-	-
RH-S	-	0.78 ± 0.03	0.86 ± 0.07	0.95 ± 0.06
RH-L	-	0.77 ± 0.04	0.77 ± 0.02	0.81 ± 0.02
RS-S	-	0.75 ± 0.01	0.84 ± 0.02	0.88 ± 0.02
RS-L	-	0.76 ± 0.02	0.83 ± 0.04	0.86 ± 0.04

The percentage of water absorption of NR composites filled with various filler types is shown in Fig. 2(a). In general, the hydrophobicity matrix hardly absorbs water [22], so neat NR showed an insignificant increase in water absorption. On the contrary, composite filled with RHs and RSs displayed a higher percentage of water absorption with immersion time due to the hydrophilic nature of natural fiber [10, 23, 24]. After immersing in water for 7 days, the percentage of water absorption of all types of NR composite was 1.92 – 6.97 times higher than that of NR. The composite with higher filler contents showed higher water absorption ability that similar to many works [10, 12, 23]. The shape of fillers also affected the water permeability of composites. The fibrous (RS-S and RS-L) and large (RH-L) structure filler displayed higher water absorption because of the larger gap size between fillers and matrix while using finer filler (RH-S) in composite provided smaller gap resulting in lower water uptake.

Fig. 2(b) displays the percentage of oil absorption of neat NR and NR composites. In contrast with water uptake results, the oil absorption of NR was dramatically increased with increasing oil contact time due to the hydrophobicity of NR. Therefore, the oil uptake of neat NR after immersing in cooking oil for 7 days was up to 59%. In the case of NR composites filled with RHs and RSs, the oil uptake characteristics of composites were affected by the hydrophilic nature of components of each filler. Generally, cellulose and hemicellulose are highly hydrophilic and prefer water absorption, whereas lignin is more hydrophobic so it prefers to absorb oil [25, 26]. Thus, NR composite-filled RHs (higher lignin content) displayed higher oil uptake while filled RSs (higher hemicellulose content) showed lower oil absorption. From Fig 2(b), the percentage of oil absorption of all RHs composites was 0.08 – 0.47

times higher while that of all RSs composites was 0.04 – 0.29 times lower when comparing with neat NR after immersing in oil for 7 days. The effects of filler contents and sizes on oil uptake were also observed. NR composites showed a decrease in oil uptake with increasing hydrophilic filler content and composites using larger filler displayed the higher oil uptake.

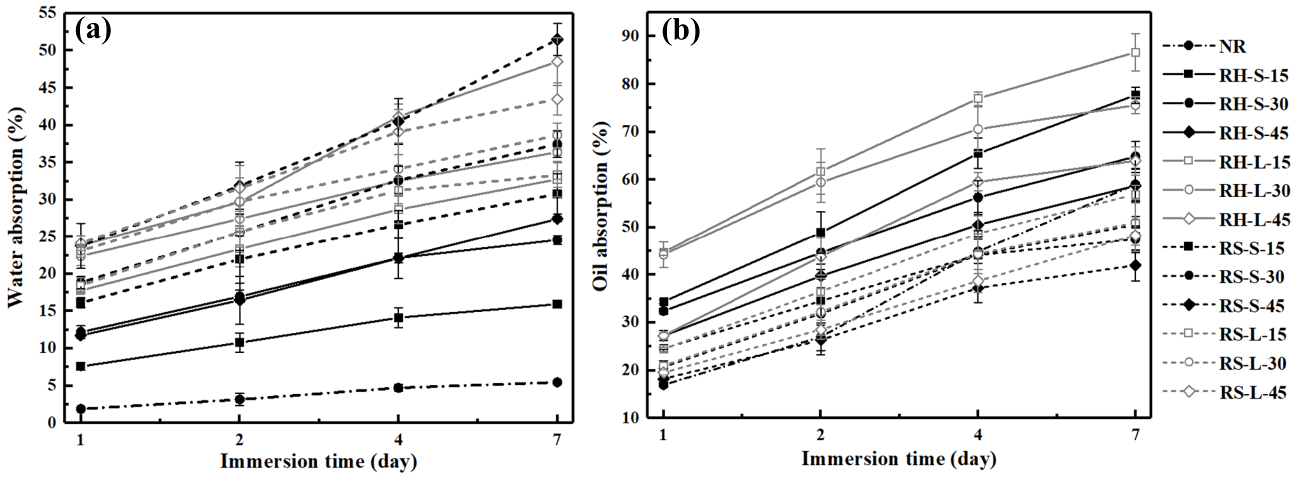

Fig. 2 Percentage of water absorption (a) and oil absorption (b) of NR composites filled with various sizes and contents of RH (solid line) and RS (dashed line).

Young's modulus and tensile strengths of NR and NR composites filled with various sizes and contents of RHs and RSs are shown in Fig. 3(a) and 3(b), respectively. As many works of NR composite system have noted that the incorporation of stiffness filler in soft matrix resulting to the resistance of elastic deformation, thus it markedly displayed stiffness improvement of the composite [11, 27]. The addition of RHs and RSs in NR systems displayed a significant increase in their moduli with increasing filler contents as shown in Fig. 3(a). Compared to the Young's modulus of NR (inserted graph in Fig. 3(a), the Young's modulus of all RHs composites was 4 – 114 times higher than that of NR. While composite using fibrous structure fillers (RS-S and RS-L) revealed the stronger improvement of moduli which was 117 – 549 times higher than that of neat NR. NR composite with 45 phr RS-L displayed the greatest in reinforcing effectiveness. It has been explained that using higher aspect ratio filler in composite provided higher stress transfer between filler and matrix [11 – 12] resulting in the modulus improvement. On the other hand, composite with particulate structure fillers (RH-S and RH-L) did not observe any effects of particulate size, but all types of RH/NR composites showed a slight improvement in moduli compared to NR.

Tensile strengths of NR composites with various filler types and contents are shown in Fig. 3(b). It is clearly observed that tensile strengths of all composites revealed lower than that of neat NR similar to many reviews [11, 24, 27]. This may be due to the presence of fillers in NR composites providing the void occurrence between hydrophilic filler and hydrophobic matrix leading to the internal cracks and a reduction in tensile strength of all NR composites. Moreover, RH-S/NR, RS-S/NR, and RS-L/NR composites showed a decrease of tensile strength with increasing filler contents. However, in the case of RH-L/NR composite (the large filler system) displayed the worsen tensile strength and did not show the effect of RH-L content.

SEM micrographs of the tensile fracture surface of RHs/NR composites and RSs/NR composites are shown in Fig. 4 and 5, respectively. SEM images of all composites at low magnification showed a rough surface and revealed the fillers (solid arrow) on tensile fracture surfaces. Furthermore, SEM micrographs of all composite systems at high magnification observed the hole (dashed arrow) of filler pullout and the gap around the fillers (circled) which clearly indicates the poor interfacial adhesion between the filler and the NR. These SEM micrograph evidences agreed with the decrement of tensile strength of NR composites with RSs and RHs.

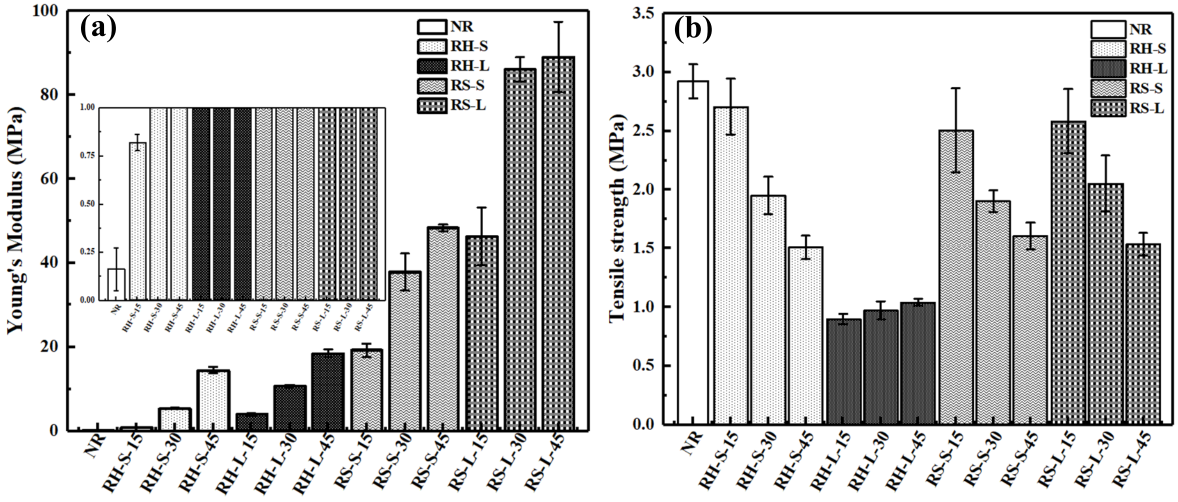

Fig. 3 Young's moduli (a) and tensile strengths (b) of NR composites filled with various sizes and contents of RH and RS.

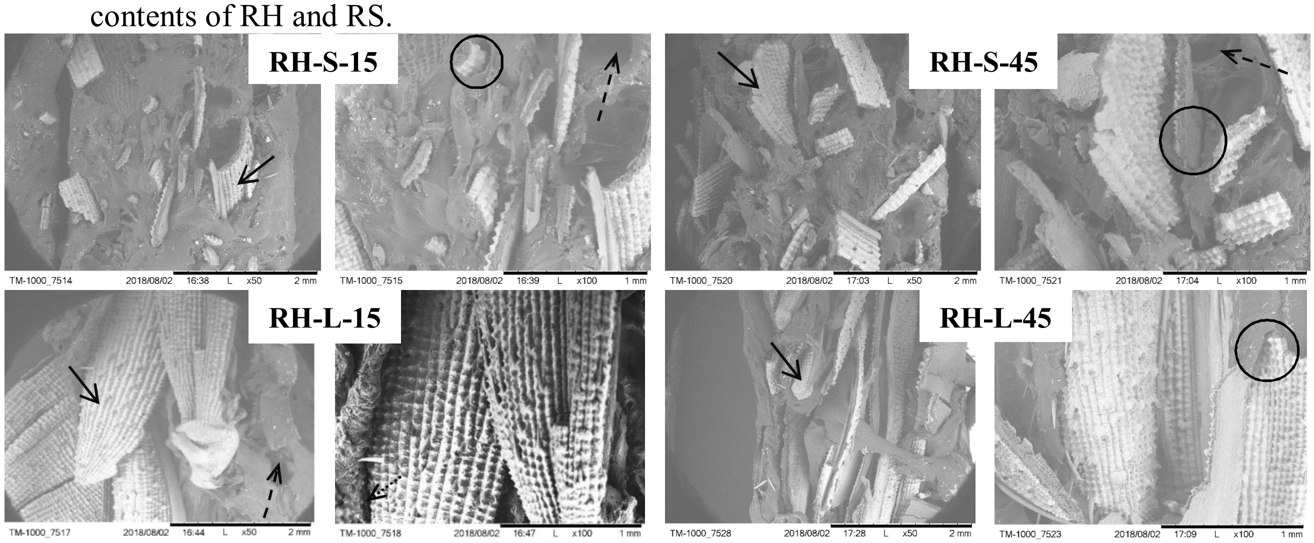

Fig. 4 SEM micrographs of tensile fracture surfaces of RHs/NR composites.

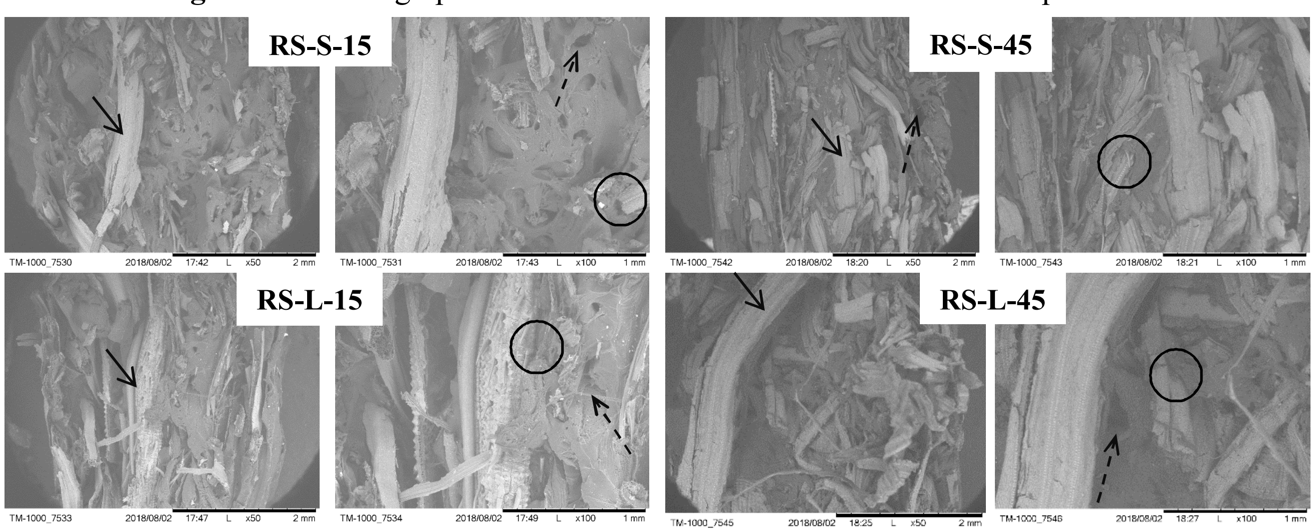

Fig. 5 SEM micrographs of tensile fracture surfaces of RSs/NR composites.

4. Conclusion

NR composites filled with RH and RS were prepared. The effects of filler sizes (RH-S, RH-L, RS-S, and RS-L) and filler contents (15, 30 and 45 phr) on the physical and mechanical properties of NR composites were investigated. The results showed the filler types, filler sizes, and amount of fillers insignificantly affected on the composite's density of all composite types. However, these above effects strongly affected the water and oil absorption of NR systems. Comparing with NR, natural fillers had a more hydrophilic nature. Thus, the percentage of water absorption of all types of composites increased with an increase in the filler content garnering an increase up to 1.92 – 6.97 times of NR. However, the value of percentage of oil absorption of all type composites decreased with an increase in the filler content. NR composites filled RSs had decreased the oil uptake up to 0.04 – 0.29 times of NR. The water and oil absorption characteristics of the NR composite were different due to the difference in the chemical composition of RS and RH. RS has higher hemicellulose content, thus RSs/NR composites showed a higher percentage of water absorption but a lower percentage of oil absorption. On the contrary, RH has higher lignin content, so RHs/NR composites showed higher oil uptake. Composites with long shape fillers like RS-L and RH-L displayed a higher tendency of water and oil absorptions, respectively. The tensile property result reveals that Young's modulus of composite with all rigid fillers displayed an improvement that increased with increasing filler contents. Composites using RSs showed better modulus improvement tendency. The RS-L/NR composite with 45 phr filler content provided the greatest in Young's modulus enhancement which was 549 times higher than that of NR. However, strengths of all composites decreased with the increase of the filler content. This is resulting from the fillers acting as a hindrance in NR and the lack of interfacial interaction between fillers and NR, and these evidences could be seen by SEM micrographs.

5. Suggestions

This work is focused on the evaluation of the physical and mechanical properties of NR reinforced with RS and RH. In order to get more attention on environmental awareness, these biocomposites should be investigated for their biodegradability in further work.

6. Acknowledgement

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