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ISSN: 2394-5311

Development of Agro-Industrial Waste Reinforced Natural Rubber Composite: A Potential Formulation for Rubber Flooring Product

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ARTICLE DETAILS

Article history:
Received 19 August 2018
Accepted 08 September 2018
Available online 19 September 2018

Keywords:
Flexible Composites
Polymer-Matrix Composites
Particle Reinforced Composites

ABSTRACT

Agro-industrial waste has become a major environmental issue in most parts of the world. Rice husk is one of the major agricultural wastes especially in Asian countries. It is currently thrown away into landfills or rarely use as an alternative energy source, which in turn produce another waste, rice husk ash (RHA). Silica is the major component in well-burnt RHA. This work was aimed to evaluate the feasibility of utilizing silica extracted from RHA and another industrial waste, used tyre treads, with natural rubber to develop composite with enhanced mechanical properties especially for flooring products such as rugs, pavement blocks, door mats, etc. In this work extracted silica was characterized using Fourier-transform infrared spectroscopy, X-ray diffractometry (XRD), X-ray fluorescence spectroscopy (XRF) and scanning electron microscopy (SEM). The SEM images confirmed that the extracted silica is in the nanometer to sub-micrometer length scale in size. Further our results confirmed that extracted silica has comparable chemical composition and amorphous nature as commercially used silica in rubber compounding. Composites were first formulated using crumb rubber to identify the optimum crumb rubber loading. It is found that 25 phr of crumb rubber provides the optimal mechanical properties. Finally, extracted silica was incorporated to 25 phr crumb rubber loaded composite to further reinforcement. SEM images confirm that 10 phr of silica have enhanced matrix-filler interactions to produce continuous structure, which was not observed in crumb rubber loaded rubber composite.

1. Introduction

One of the most frightening matters facing most part of the world is the pilling up of wastes, which spoils many areas but not limited to public health and environmental pollution [1]. This work introduces a method to utilize two major wastes, i.e. used-automobile tyres and rice husk in useful way. This will introduce not only a partial solution for waste management but also a value addition to the existing products.

Disposal of waste tyre has become a major environmental impact since every year millions of tyres are discarded in various methods like land filling, burning, use as fuel, pyrolysis to produce carbon black etc., [2, 3]. Stockpiled tires present many types of health, environmental and economic risks through air, water and soil pollution [4-6]. At present most of the countries do not use this waste in useful ways rather they move directly to the final step of the waste management hierarchy which is the landfilling. One of our aims in this work is to identify the ways that one can utilize this valuable resource. One of the easiest and economical methods to manage tyre wastes is to produce granular form known as crumb rubber and incorporate them as a filler to some low-grade natural rubber-based products such as shoe soles, vehicle parts, doormats, water containers, pots, dustbins, bicycles pedals and so on. Such attempts could be environmental friendly as it helps to dispose the waste tyres and prevent environmental pollution. It also helps to reduce the carbon dioxide emission by the pyrolysis of waste tyres.

Rice is the second most consumed food item worldwide [7]. Approximately 20 wt.% of the paddy grain is the husk, which is the most representative by-product of processing of rice. The destiny of this waste is the landfill or burn to get the energy. Burning of rice husk yields approximately 20 wt.% of ash which has high residual carbon and silica [8]. The percentage of silica depends on the temperature at which the rice husk burns. Thus, this could be an excellent source for extracting silica for various purposes such as adsorbent to remove coloring agents from waste water [9], fillers, reinforcement agent to strengthening the polymers [10], binder materials in civil construction [11] and as a raw material in the production of bio based silica [12].

The products made from pristine rubber show retarded mechanical properties, for instance: poor hardness, low abrasion resistance, and high elasticity. The problem will overcome through by introducing sophisticated series of processes such as mastication, mixing, shaping, moulding and vulcanisation as well as adding fillers and other ingredients into the rubber matrix [13]. The filler must be cheaper and must be readily available. In that sense, the filler produced from agro-industrial waste could be a good option to reinforce the rubber matrices. Such reinforced material can be used to manufacture mats or tiles for all purposes. In order to obtained the best mechanical properties in such products it is important to have dense packing of the ingredients while maintaining good interfacial contact among the particulate matter. This could be easily achieved by having particles of two different sizes with good adhesive; the fine particles fill the voids between the larger particles and adhesive muddle everything together under simple contact mixing or compression molding. Such a dense structures will produce robust properties to the end product. This hypothesis is analogues to dense structure in concrete mixtures in which fine aggregates of sand fill the gaps between coarse-gravel and cement binds those particles each other.

In this study, we used micrometer length scale crumb rubber particles with sub micrometer length scale silica extracted from rice husk ash as the fillers to make rubber composites. The automobile tyre treads were mechanically grinded and the fine particles having particle size less than 125 μm were selected. The precipitated amorphous silica in the range of sub micrometer length scale extracted from rice husk ash was used to fill the fine voids in the matrix. The test samples were prepared using two-roll mill. The vulcanization was done at the later stage to further reinforce the material. The best formulation was selected by evaluating the mechanical properties.

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https://doi.org/10.30799/jacs.190.18040302

Cite this Article as: T. Kirushanthi, Thusitha N. Etampawala, Dilhara Edirisinghe, Jagath Pitawala, D.R. Ratnaweera, Development of agro-industrial waste reinforced natural rubber composite: A potential formulation for rubber flooring product, J. Adv. Chem. Sci. 4(3) (2018) 571–575.

2. Experimental Methods

2.1 Materials

Used automobile tyre treads were obtained from local agents. Rice husk ash was collected from a rice mill in Jaffna district, Sri Lanka. Industrial grade sodium hydroxide, sulfuric acid, hydrochloric acid, zinc oxide, stearic acid, N-tert-butyl-2-benzthiozole sulfenamide (TBBS), tetramethyl thiuram disulfide (TMTD), N-isopropyl-N'-phenyl-p-phenylenediamine (IPPD) and sulfur were used. The ribbed smoke sheet (RSS) was obtained from the local market.

2.2 Preparation of Crumb Rubber

Waste pneumatic tyre treads were shredded using mechanical grinder under ambient conditions. Resulted crumb rubber were sieved and particle size below 125 µm were selected.

2.3 Extraction of Silica

Silica was extracted from rice husk ash using the method as described by Palanivelu et.al with slight modifications [14]. In brief, rice husk ash was sieved and the husk particles having size smaller than 125 µm was selected. Approximately 100 g of sieved ash was digested at 100 °C in 800 ml of 3 moldm^{-3} NaOH solution for 3 hours. After digestion the sodium silicate solution was filtered. Then, 2.5 moldm^{-3} of H_2SO_4 was added until silica precipitates. Then this mixture was allowed to set for overnight. The resulted silica gel was filtered and washed with deionized water. Resulted silica was refluxed with 2 moldm^{-3} hydrochloric acid solution for 4 hours to remove the metallic impurities. Finally, the silica sample was washed thoroughly with deionized water and dried at 50 °C in a vacuum oven for overnight. The extracted silica was characterized by FTIR, SEM coupled with EDX, XRF and XRD techniques.

2.4 Preparation of Crumb Rubber Filled Composites

Seven samples labeling from S_0 to S_6 with and without adding crumb rubber fillers were prepared according to the formulation given in the Table 1. The ingredients were compounded using two rolls mill with the mixing cycle of 9 minutes.

Table 1 Composition of crumb rubber filled rubber composites

Ingredients	Loading (phr)						
	S_0	S_1	S_2	S_3	S_4	S_5	S_6
RSS	100	100	100	100	100	100	100
Crumb rubber	0	5	10	25	50	75	125
Zinc oxide	5	5	5	5	5	5	5
Stearic acid	2	2	2	2	2	2	2
TBBS	2	2	2	2	2	2	2
TMTD	0.1	0.1	0.1	0.1	0.1	0.1	0.1
IPPD	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Sulfur	2.5	2.5	2.5	2.5	2.5	2.5	2.5

2.5 Preparation of Crumb Rubber and Silica Filled Composites

Five different samples labeling from S_7 to S_{11} by varying the amount of silica filler were prepared. All other ingredients except silica were held constant for all the samples. The compositions of the samples were given in the Table 2.

Table 2 Composition of crumb rubber and silica filler rubber composites

Ingredients	Loading (phr)				
	S_7	S_8	S_9	S_{10}	S_{11}
RSS	100	100	100	100	100
Crumb rubber	25	25	25	25	25
Silica	10	25	75	150	200
ZnO	5.0	5.0	5.0	5.0	5.0
Stearic acid	2.0	2.0	2.0	2.0	2.0
TBBS	2	2	2	2	2
TMTD	0.1	0.1	0.1	0.1	0.1
IPPD	0.5	0.5	0.5	0.5	0.5
Sulfur	2.0	2.0	2.0	2.0	2.0

2.6 Characterization

2.6.1 Fourier Transformation Spectroscopic Analysis

FTIR spectrum of extracted silica was obtained using KBr pellet method using Bruker ALPHA FTIR analysis system.

2.6.2 X-Ray Diffraction Analysis

X-ray diffraction pattern of extracted silica was obtained from 2-theta from 2° to 60° using scan speed of 2° min^{-1} with step size of 0.02° using Rigaku Ultima IV XRD analyzer equipped with copper target. The XRD instrument was operated at tube voltage of 40 kV and tube current of 30 mA.

2.6.3 Analysis of Mechanical Properties

Each composite samples were analyzed to obtain percentage of elongation at break, Modulus at 100 percent elongation, hardness, rebound resilience, compression set and volume loss according to the ISO 37: 2005, ISO 48: 1994, ISO 4662-1986, ISO 815-1991 and DIN 53516 (1987) methods respectively.

2.6.4 Scanning Electron Microscopic Analysis

Cross sectional morphology of composites were observed using energy dispersive X-ray coupled scanning electron microscopy (ZEISS, model EVO LS15, Germany). The samples were mounted on aluminium stub with the help of double-sided adhesive carbon tapes. The mounted samples were sputtered with gold for about 3 minutes to make the samples electrically conductive.

3. Results and Discussion

3.1 Characterization of Extracted Silica

The FTIR spectrum of purified silica extracted from rice husk ash is presented in Fig. 1.

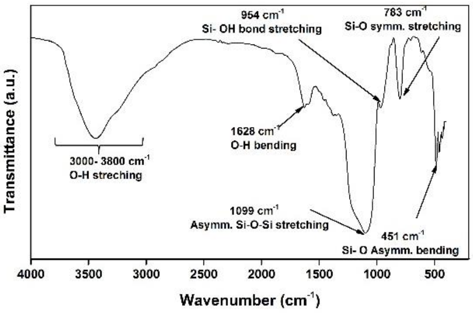

Fig. 1 FTIR spectrum of purified silica extracted from rice husk ash

The broad band observed at 3000 cm^{-1} to 3800 cm^{-1} is attributed to silanol OH groups and adsorbed water molecules [15, 16]. The OH bending vibrational modes of the silica gel network is confirmed from the bands at 1628 cm^{-1} [15]. The strong band observed at 1099 cm^{-1} corresponds to the asymmetric stretching vibration of Si–O–Si bonds [17]. The symmetric stretching vibration of Si–O–Si bond is evident from the band at 783 cm^{-1}. The band observed at 451 cm^{-1} confirms the existence of asymmetric bending vibration of Si–O–Si bonds [17]. These bands confirm the identity of extracted silica. Further, the absence of unknown bands confirms the purity of the sample.

Fig. 2 present a comparison of extracted silica before and after purification with a commercially used silica in local tyre tread industry. It is clearly shown that some bands in FTIR pattern disappeared after purification. The pattern of the commercially used silica matches well with the pattern of extracted silica after purification. However the band observed at 3000 to 3800 cm^{-1} is more prominent in extracted silica than the commercially used silica. This could be attributed to the absorbed water or presence of silanol OH groups in the extracted sample than commercially available sample.

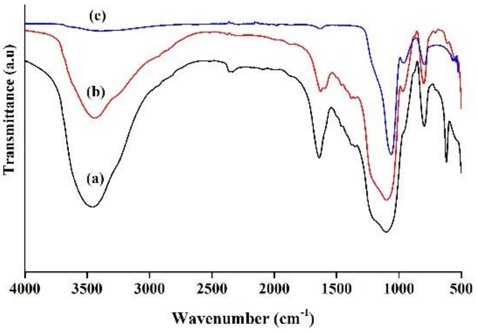

Fig. 2 A comparison of FTIR spectrums of (a) unpurified silica (b) purified silica and (c) commercially used silica in local tyre tread industry

https://doi.org/10.30799/jacs.190.18040302

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In general, rice husk consists of many organic and inorganic impurities. Apart from silicon there are metals such as Na, K, Mg, Ca, Fe, Cu and organic materials such as cellulose, hemicellulose, and lignin [18]. The elemental analysis of RHA and purified silica was done by energy dispersive X-ray diffraction technique couple to scanning electron microscopy. The Table 3 presents the analyzed elemental composition in RHA and purified silica. The presence of silicon and other metals such as Na, K, Mg, Al, and Mn can easily be detected in the RHA. Further, the results show that the SiO_2 was the major component present in the purified silica sample. The results confirm the purification process that was followed is sufficient to eliminate the trace metals present in the RHA.

Table 3 Elemental composition of rice husk ash and purified silica determined by Energy dispersive X-ray diffraction technique

Component	Weight % in RHA	Weight % in extracted silica
SiO_2	94.8	99.1
Al_2O_3	0.74	0.269
SO_3	0.32	0.161
CaO	1.89	0.122
Na_2O	0.28	0.117
K_2O	1.12	0.0647
Fe_2O_3	0.30	0.0329
MnO	0.37	0.0054
TiO_2	0.10	0.0038

The metals can be commonly exists as their oxides. The different phases and the crystallinity of silica were determined by X-ray diffraction technique. The Fig. 3a shows the X-ray diffraction pattern of unpurified silica extracted from rice husk ash. The sharp peaks with broad halo confirm the semi-crystalline nature of the unpurified silica. Once it is purified by acid digestion the sharp peaks disappeared as shown in Fig. 3b and a broad halo at about 2θ value of 22 became prominent. This is a characteristic to the amorphous silica. The nature of the extracted silica was compared with that of the commercially used silica in local tyre tread industry. Fig. 3c presents the comparison. The patterns are almost same which confirms the purity and amorphous nature of the extracted silica.

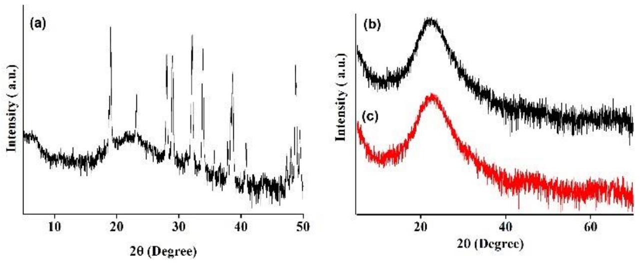

Fig. 3 X-ray diffraction pattern of (a) unpurified silica (b) purified silica using acid digestion and (c) commercially used silica in local tyre tread industry

3.2 Morphology of Extracted Silica

Fig. 4 presents the SEM image of extracted silica after purification. The samples were dried in drying oven overnight at about 60 °C prior to taking SEM images. This image confirms that the silica particles are agglomerated in to larger clusters. However, one can clearly observed that these large chunks were built up by coalescence of fine particles. It is generally accepted that silica particles are rich with silanol groups at the surfaces that cause for strong interaction of the polar groups such as other silica particles or moisture in the atmosphere. Presence of silanol OH groups was further confirmed by the presence of a broad band at 3000 to 3800 cm^{-1}. These polar groups may held the fine silica particles together to form larger chunks as observed in the Fig. 4.

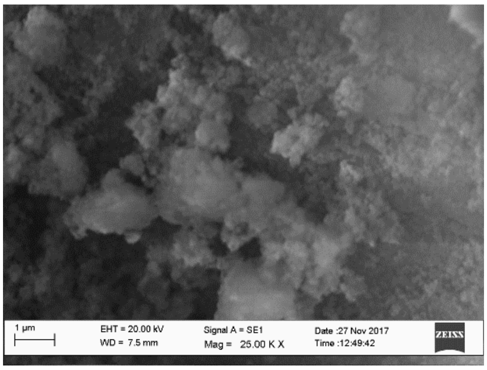

Fig. 4. Scanning electron microscopic image of purified silica

3.3 Characterization of Composites

The composites were prepared by incorporating the crumb rubber and other additives with natural rubber as shown in Table 1. The mechanical properties including percentage of elongation at break, modulus at 100% elongation, hardness, rebound resilience, compression set and volume loss were measured to evaluate the sample having optimum mechanical properties. After selecting the optimal crumb rubber loading extracted silica was added as shown in Table 2 in different ratios from 10 to 200 phr for further reinforcing the samples. The above-mentioned mechanical properties were measured to evaluate the reinforcement.

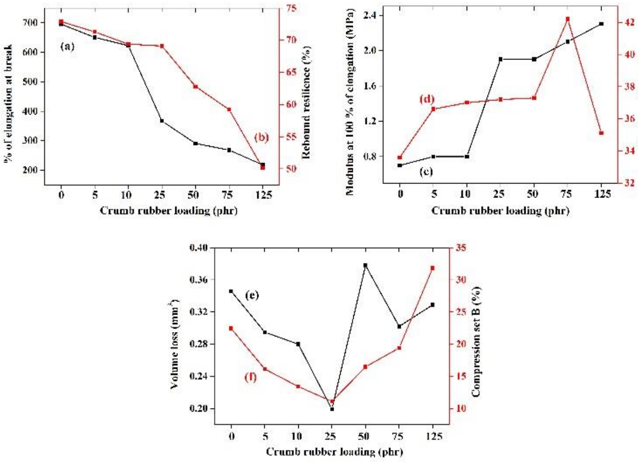

Fig. 5 Mechanical properties of crumb rubber reinforced natural rubber composites. (a) Percentage of elongation at break (b) Rebound resilience % (c) Modulus at 100 % elongation (d) hardness (e) volume loss and (f) compression set%.

Fig. 5a clearly shows the addition of crumb rubber into natural rubber matrix result in decreasing the percent elongation. However, large values of the percentage elongation at break suggests that natural rubber composites with the crumb rubber are more elastic. A dramatic change of percent elongation happens between the 10 to 25 phr of crumb rubber loading. Percent elongation of natural rubber matrix decreases about 46% with respect to pristine rubber samples just by adding 25 phr. All the samples at least elongate 100% before the break. Thus, the modulus at 100% of elongation of all the samples was measured for comparison. The modulus increases as crumb rubber loading increases as shown in Fig. 5c. Approximately 137% increment in modulus at 100% elongation can be seen when crumb rubber loading increases from 10 to 25 phr.

Resilience of a rubber-based product is an important parameter since it represents a measure of how elastic it is when exposed to various stresses. Fig. 5b present the rebound resilience of the crumb rubber loaded composites. Resilience decreases gradually up to 50 phr of crumb rubber loading. After 50 phr loading it decreases dramatically. The results are agreeing with the percent elongation at break, modulus at 100% elongation and hardness. When crumb rubber loading increases the probability to make interaction with the matrix chains also increases which in turn decreases the elasticity and the mobility of the matrix material.

Crumb rubber were obtained from already vulcanized tyre treads. Thus, once it is mixed with natural rubber one can expect to increase the hardness of the composite. As presented in Fig. 5d the shore A hardness of the crumb rubber loaded composites were increased gradually up to 10 phr loading and rapid jump of about 14% was observed between 10 to 25 phr. With further increment of crumb rubber hardness was hardly changed. However, rapid decrement of hardness was observed when crumb rubber loading is 125 phr. At low loading rubber matrix could be sufficient to entangle around the crumb rubber and held them tightly together with the matrix. However at higher loading, the matrix material may not be sufficient to bind all the crumb rubber together. The chemical linkages between rubber matrix and already vulcanized crumb rubber also possible since these composites were vulcanized at the later stages. Since hardness implies the resistance to introduce foreign bodies into its surface layer, the resistance to foreign bodies can be dramatically increased just by adding 10 to 25 phr of crumb rubber particles.

The volume loss of the composites presents in Fig. 5e. The behavior of volume loss is comparable with the behavior of resilience. The volume loss decreases up to 25 phr crumb rubber loaded sample. The decrement is approximately 43% with respect to the pristine rubber sample. However with further increment of crumb rubber higher amount of material was lost. Rubber chains are not tightly interact with added particles at higher loading. Thus, they can easily peel off. The results indicates that wear

https://doi.org/10.30799/jacs.190.18040302

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resistance of rubber matrices can be improved significantly by incorporating about 25 phr of crumb rubber.

A compression set data can be used to interpret the capability of a material to permanently deform in presence of external stresses. Lower percentage of compression set represents low resilient and thus low deformation in presence of external forces. As shown in Fig. 5f the compression percentage decreases gradually with increasing the crumb rubber loading up to 25 phr followed by it increases gradually up to 75 phr crumb rubber loading. However, compared to 75 phr loading the compression percentage increases dramatically when 125 phr of crumb rubber was loaded. The observations are well tally with our previous interpretation. At low loading, the matrix chains interact well with the added crumb rubber particles. Crumb rubber are harder materials. With increasing its volume fraction in the matrix the hardness increases and percent compression decreases. With further increasing its volume fraction it is possible to have aggregate them and have clusters inside the matrix with minimal interactions with the rubber chains. Thus, in presence of compression force the particles can be packed well.

Optimal mechanical properties were obtained when 25 phr of crumb rubber was loaded. The stress was effectively transferred to crumb rubber when the loading is optimum. With further loading of crumb rubber the degree of reinforcement decreases. Thus, 25 phr loaded formulations were selected and check whether incorporating silica extracted from rice husk ash can further reinforces the composites. Here, 10, 25, 75, 150 and 200 phr of extracted silica was incorporated with 25 phr crumb rubber loaded rubber composites. It is important to emphasize that the physical appearance of the composites having 75, 150 and 200 phr was not good. Thus, the mechanical properties of those three composites were not evaluated. One can expect that small silica particles fill into the voids spaces between large crumb rubber particles and form a continuous phase. However excess of silica enhances the incompatibility owing to its hydrophilic nature in hydrophobic rubber matrix.

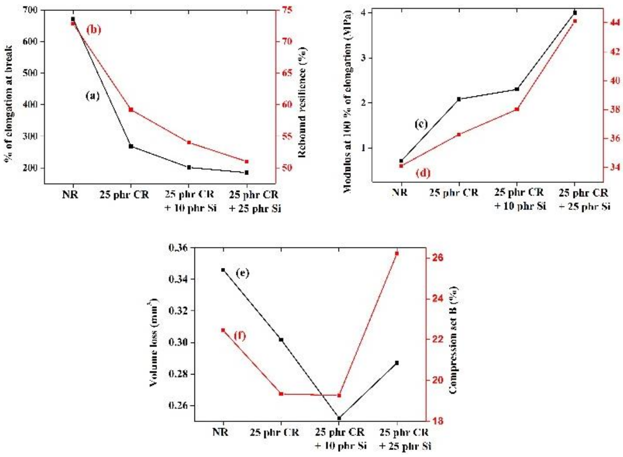

Fig. 6 Comparison of mechanical properties of pristine rubber, crumb rubber loaded rubber composite, 10 phr silica and 25 phr crumb rubber loaded rubber composite and 25 phr silica and 25 phr crumb rubber loaded rubber composite. (a) Percent of elongation at break (b) Rebound Resilience (c) Modulus at 100 % elongation (d) hardness (e) volume loss and (f) compression set%.

The comparison of mechanical properties of pristine rubber, 25 phr crumb rubber loaded composite, silica reinforced composites are presented in Fig. 6.

As presented in Fig. 6a, the percent elongation at break decreases further after incorporation of silica. Pristine natural rubber samples have approximately 650% elongation at break, which decreased about 46% after incorporating 25 phr of crumb rubber. It is interesting to see that addition of 10 phr silica the percent elongation is further decreased by 44% with respect to 25 phr crumb rubber loaded sample. However when silica content was further increased from 10 to 25 phr, the decrement in % elongation at break is about 49% with respect to 25 phr crumb rubber loaded sample. This improvement is approximately 69% and 72% respectively for 10 phr and 25 phr silica loaded sample with respect to pristine natural rubber sample. The change in percent elongation at break is not subtle when silica loading is increased above 10 phr. These results indicate that crumb rubber and silica is a very good combination to reinforce natural rubber based materials.

The rebound resilience of the sample also changes slightly in presence of silica as shown in Fig. 6b. In presence of silica, the resilience decreases as expected. This could be due to the retardation of the motions of the rubber chains.

The modulus at 100% elongation increased about 210% when 25 phr crumb rubber was introduced. This was slightly increased in presence of 10 phr silica. However, a prominent increment, which is about 566% with respect to natural rubber sample, was observed when 25 phr of silica was introduced. With filler loading the strain decrease and stress should increase which in turn increases the modulus. However, at 25 phr loading the composite must be able to bare more loads than predicted because the strain decreases slightly but modulus increases significantly.

Silica is an inorganic material and hardness of silica clusters should be higher than that of both rubber samples and crumb rubber particles. Thus, as expected in presence of silica, hardness further increased as presented in Fig. 6d. However, the changes are not subtle even in presence of 25 phr loading.

The volume loss of the composite decreases in presence of 10 phr silica. The decrement is approximately 16% with respect to the 25 phr crumb rubber loaded sample. However, volume lost increases with further increasing the silica loading. At higher loading, the silica particles may not bound by the matrix rubber chains. Thus, loosely bound silica can easily peel off. This results confirms that wear resistance of rubber composites can be improved by incorporating just about 10 phr silica.

Fig. 6f shows the comparison of compression set results. Compression of the material decreased with crumb rubber loading which was further decreased with silica loading. However when silica loading is changed from 10 to 25 phr it was increased. This results indicate that 10 phr of silica is sufficient to have minimal compression. It means that 10 phr silica is sufficient to fill the voids spaces of 25 phr crumb rubber loaded sample. When more silica is available they may loosely bound to the matrix owing to the insufficiency of natural rubber.

3.4 Morphology of Composites

Cross-sections of the composites were investigated using scanning electron microscopy. Fig. 7a present the cross-section of 25 phr crumb rubber loaded composite whereas Fig. 7b present after reinforcing the same sample with 10 phr of silica. The Fig. 7a illustrates the homogeneous dispersion of crumb rubber throughout the natural rubber matrix. However, it is clearly see that sub-micrometer voids are also present in the sample, which was not observed after loading 10 phr of silica. This investigation supports the improved mechanical properties in silica-loaded samples. Silica particles have quite a variety of geometries, however they have approximately the same dimensions in all directions. Since the silica particles are small, they fill the voids and evenly distributed throughout the matrix. Both crumb rubber and silica particles restrain movement of the matrix phase in the vicinity of each particle and form a continuous phase. Consequently, rubber matrix transfers some of the applied stress effectively to the filler particles, which bear most fraction of the load. Strong bonding at the matrix–filler interface, further supported these enhanced properties.

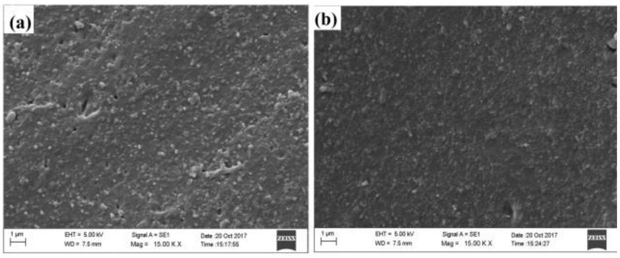

Fig. 7 Scanning electron microscopic images of a) natural rubber and 25 phr crumb rubber loaded composite b) natural rubber, 25 phr crumb rubber and 10 phr Silica loaded composite.

4. Conclusion

This study has shown that the possibility of use of agro-industrial waste materials as fillers in rubber composites while having very competitive mechanical properties. Series of silica and crumb rubber loaded natural rubber composites were prepared. Silica was extracted from rice husk ash and crumb rubber was prepared using waste tyre treads. The extracted silica has comparable chemical and physical characteristics to commercially used silica in tyre compounding industry. Crumb rubber having particle size smaller than 125 μm were used. Composites were first prepared using natural rubber as the matrix and crumb rubber as the filler. Mechanical properties were getting better with increasing the crumb rubber loading up to about 25 to 50 phr and then it started to show retarded behavior. Extracted silica was incorporated to crumb rubber composites having 25 phr crumb rubber loading. At very high silica loading composites cannot be made. The study showed that 10 phr of silica was sufficient to get optimum reinforcement. This work has great potential to utilize agro-industrial waste to lower the factors cause for environmental pollution.

Acknowledgments

The authors thank Uva Wellassa University of Sri Lanka for financially support provided under the grant number UWU/RG/2016/11. A portion of this research used resources at Instrument Center operated by Faculty of Applied Sciences of University of Sri Jayewardenepura and Center of Advance Material Research at University of Sri Jayewardenepura. The authors greatly acknowledged the support given by those facilities to make this work success. Also Authors glad to express their acknowledgement to the all staffs at Department of Rubber Technology and of Rubber Research Institute of Sri Lanka for their guidance throughout the study.

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https://doi.org/10.30799/jacs.190.18040302

Cite this Article as: T. Kirushanthi, Thusitha N. Etampawala, Dilhara Edirisinghe, Jagath Pitawala, D.R. Ratnaweera, Development of agro-industrial waste reinforced natural rubber composite: A potential formulation for rubber flooring product, J. Adv. Chem. Sci. 4(3) (2018) 571–575.