Impacts of rice husk ash filler loading on curing, morphological characteristics and tensile properties of natural rubber/ethylene propylene rubber blends

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Abstract. Rice husk is an agro industrial waste that potentially used in biomaterial application. It contains high silica that could be utilized as a reinforcing filler to substitute commercially precipitated silica or other commercial fillers in rubber compounding application. This research studied the effect of rice husk ash (RHA) loading as reinforcing filler in Natural Rubber/Ethyene Propylene rubber (NR/EPR) blends. The effects were observed by applying 40, 50, 60, 70 phr of RHA filler in the rubber compounding for NR (100) and also for blending NR/EPR (70:30). The rubber compounding and vulcanization for the rubber compounding was conducted according to the ASTM D3182. The results showed that RHA were successfully prepared and introduced as a new alternative filler for rubber industrial application. Based on the curing characteristics, the RHA loading influence slightly $t_{12}$, $t_{90}$, ML, MH and cross link density on the NR blending with and without EPR addition. At the same RHA composition, the torque and cross link density of NR-EPR blending were much higher compared to only NR. RHA loading on NR with and without EPR composites caused a slight decreasing on tensile strength, elongation at break, and compression set. Conversely, the hardness of those composites tended to increase due to the RHA addition. The SEM micrographs provided additional evidences on the poor tensile properties as well as the matrix tearing and voids due to EPR addition into NR composites. To sum up, RHA may be used as an alternative filler for NR composites along with the EPR addition in order to improve compression set and hardness properties without losing tensile properties of the composites.

Keywords: NR-BR Blends, Rice husk Ash Filler, Silica, Tensile Properties, Morphological Characteristics

1. Introduction

Rice husk is an agro industrial waste particularly in rice production. The utilization of rice husk ash has been developed a few years ago for farming, bio mass fuel, and polymer application. For instance, burnt rice husk ash (RHA) has been used as fillers for polymer application namely white RHA and black RHA. It is stated that white RHA contains higher silica 77\%, whilst the black only about 41\% and the rest of it is a substantial carbon content such as loss of ignition, \text{CaO}, \text{MgO}, \text{Fe}_2\text{O}_3, \text{K}_2\text{O}, \text{Al}_2\text{O}_3, \text{TiO}_2, \text{P}_2\text{O}_5 [1, 2]. In addition, in terms of carbon content and the effect of its addition into rubber vulcanizates, the white RHA also has the properties nearly similar as commercial carbon black [3].

Fillers addition for rubber compounding has many advantages such as improve physical-mechanical properties (tensile strength, hardness, compression set and elongation at break) and reduce material costs during processing [4]. The common fillers used for rubber industries is carbon black, the petroleum
derivation products [5]. However, the carbon black usages has many disadvantages like scarcity, high price, and non-renewable products [6]. Therefore, the utilization of RHA that contains high silica may substitute the CB consumption for rubber industries. This is because silica that extracted as RHA possibly may be applied as reinforcing fillers for rubber compounding [3, 7]. Compared to commercial silica such as zeosil, chemisil, and precipitated silica, RHA has lower silica content and less purity. However, the utilization of RHA has benefits such as greener, reduce agro industrial waste, as well as decrease the material cost.

It is reported that incorporation rice husk ash into polypropylene lead to a significant increase in the flexural modulus of the composites, one that is comparable to that imparted commercial fillers such as mica [2]. It is also reported that effect of rice husk ash used as filler for epoxidized natural rubber compounds and the multifunctional additives and Silane 69 improved some mechanical properties of the rubber compounds [3]. Moreover, many researchers reported that RHA could be applied as a partial replacement for carbon black or commercial silica, particularly for NR compounding with some modifiers chemicals such as MFA, coupling agent, and silane 69 [1]. However, researches focus on the utilization of RHA on natural rubber (NR) and ethylene propylene rubber (EPR) blending are still limited.

However, in rubber application, it is necessary to blend one or more polymer types to obtain a desired physical-mechanical process that meets a certain product specification. In this research, the rubber formula was designed for heat resistance products that has high tensile properties, particularly for rubber in cable and wires application. Therefore, the polymers that designed were natural rubber (NR) and ethylene propylene rubber (EPR) using the RHA as alternative filler. Theoretically, NR has the best tensile properties, while EPR has good heat resistance. However, in this research, the heat resistance properties of the composites were not explored deeply.

In addition, in order to support the greening campaign for rubber industry to substitute petroleum based chemicals, this research applied natural based plasticizer namely Jatropha oil rather than commercial (petroleum based) such as white oil, paraffin wax and other mineral oil. The plasticizer would lubricate and soften the mixing of EPR, NR and RHA to obtain best fine rubber compounding [8]. It was reported that Jatropha oil was a compatible plasticizer for NR application using Calcium Carbonates as filler [9]. However, the investigation for the compatibility between EPR, NR and RHA as filler using Jatropha oil is still needed.

Bearing in mind the agro-industrial waste namely rice husk along with the environmental issues for limited petroleum based products for rubber industries, the importance of observation of new alternative renewable fillers is an urgency. With regard to some weakness of using NR for wire rubber applications, this research studied the effect of using RHA as filler in NR along with the EPR rubber blending to improve some particular properties. In order to observe the compatibility blending between RHA, NR and EPR using Jatropha oil, curing characteristic and morphological (SEM) were investigated. In addition, to examine the effect of RHA as a reinforcing filler for NR with and without EPR addition, physical mechanical and tensile properties namely hardness shore A, tensile strength, elongation at break, and compression set were tested. Consequently, the research would contribute new practical information on the utilization of RHA filler in the rubber composites like NR with or without EPR blending particularly for hose, construction rubber products, electrical wire and other rubber products.

2. Experimental Procedures

2.1. Materials
Polymers: Ethylene Propylene Rubber (EPR-70), Natural Rubber/NR (Standard Indonesian Rubber/SIR 10). The chemicals including: Activator: Zinc Oxide (NC 105 Global Chemical Co, Ltd); Accelerators: CBS, MBTS (Brathaco Chemical); Vulcanizer: Sulphur (Brathaco Chemical); Peptisizer: Stearic Acid (SA 1806) and Jatropha Oil, Oleochemicals Industry, PT Sumi Asih); Tackifiers: Cumaron resin (Brathaco Chemical); Anti-oxidant: BHT (Brathaco Chemical); Fillers: Caoline (PT Supersilica Indo Semesta).
2.2. Methods

2.2.1. Rice Husk Ash Preparation. The following procedure is in accordance with [7, 10]. Rice husk was soaked and washed using pure water to clean any foreign materials. Meanwhile, an amount of 0.4 M of Hydrochloric acid solution was prepared, then 100 g cleaned rice husk was mixed in 1:1 (weight/weight) of prepared acid solution and then boiled at 100-105°C for 30-45 minutes. After the reaction, the husk was washed using pure water until pH neutral. The purified husk was dried in an oven at 105-110°C for 3-5 hours. The treated husk was burned in furnace at 600°C for 6 hours; silica was obtained as white ash and black ash, in this research both or ashes was used as rice husk ashes (RHA). In order to obtain 38 um particle size, the RHA was ground and sieved.

2.2.2. Rubber Compounding. Laboratory two roll mill XK-160 (Shanghai Rubber, Machine Worker, China) was used to conduct the rubber compounding. The polymer of NR with or without EPR was masticated first at about 3-4 minutes, then chemical ingredients as listed in table 1 were compounded in accordance with ASTM D3182 Standard Practice for Rubber (Materials, Equipment, and Procedures) for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets and ASTM D3184-89 Standard Test Methods for Rubber Evaluation of Natural Rubber. In order to examine the physical mechanical, tensile properties and morphological characteristics, the compounded rubber sheet was vulcanized for about 10 minutes at 140°C using heating press hydraulic (YG-220, Shanghai Rubber Machine Worker, China). These procedures were replicated 3 for each formula in order to obtain highly accuracy data.

2.2.3. Curing Characteristic. The procedures of testing were according to ASTM 5289 Standard Test Method for Rubber Property- Vulcanization Using Rotorless Cure Meters. Before, the vulcanizates were conditioned for 24 hours at room temperature 25-27°C. It was tested using an oscillating Rotorless Rheometer type UR2010 at set temperature of 160°C, a cure time of 4 minutes, and a pressure of 4.0-4.5 kg cm⁻². The scorch time t₂, t₇₀ and t₉₀, torque minimum (MH), torque maximum (ML), cure rate index (CRI) and cross link density were observed accordingly.

2.2.4. Physical Mechanical and Tensile Properties. Each vulcanizates were tested for parameters as follows: hardness Shore A (ASTM D 2240-05 (ra 2010) using hardness IHRD, Germany; tensile strength & elongation at break testing in accordance with ASTM D 412 at 26°C room temperature at

| Table 1. Formulation of NR/EPR using RHA filler. |
|-----------------------------------------------|
| Material                        | Formula (phr) |                  |
| NR (SIR 10)                     | 100           | 70               |
| Ethylene Propylene Rubber       | 0             | 30               |
| RHA Fillers                     | 40; 50; 60; 70| 40; 50; 60; 70   |
| Kaoline                         | 50            |                  |
| Jatropha Oil                    | 2             |                  |
| ZnO                             | 5             |                  |
| Stearic Acid                    | 2             |                  |
| Cumaron Resin                   | 1             |                  |
| CBS                             | 1.5           |                  |
| MBTS                            | 1.5           |                  |
| Sulphur                         | 2             |                  |
| BHT                             | 1.5           |                  |


500 mm/min cross head speed using INSTRON UTM Taiwan Tearing machine JT-420; and compression set was tested in accordance with ASTM D395.

2.2.5. Morphological Studies. Studies on the morphology (dispersion, fracture surfaces, and adhesion) of NR/EPR/RHA composites were conducted using Scanning Electron Microscope (SEM) JEOL JSM 6510.

3. Results and Discussion

3.1. Curing Characteristics of RHA-NR/EPR Blends

![Curing characteristics graphs](image)

**Figure 1.** Curing characteristics (optimum cure time, scorch time, torque graphs) of NR and NR/EPR blends using different loading of RHA.

Fig. 1 shows the comparison of curing characteristics of NR/EPR composites and NR filled with RHA. The data was discussed and observed with the increasing loading of RHA used in the only NR and the blends between NR and EPR. It is seen clearly that the scorch time and optimum cure time of NR/EPR blends much higher than the single NR without EPR. It is indicated that the non-polar EPR that has big molecular weight restricts molecular motion of chemicals in rubber matrix during rubber processing [11]. Furthermore, by the higher RHA loading, the optimum cure time and scorch time also increase slightly. The higher cure time showed that the EPR along with the RHA filler may prolong the vulcanization process because the surface are of RHA is highly specific to adsorb the chemicals agents and charges on lattices [12]. Other researcher also reported that RHA also still have small number of hydroxyl groups and bound waters on its surface that possibly delay the compounding [13]. In addition, as the content of RHA loading for both composites increase, the curative agents such as sulphur become less soluble in NR/EPR compared with NR [6]. Consequently, the curing rate of the blend decreases with the increasing RHA loading along with the EPR addition. The presence of cross linked precursors and the un-reacted curative agents in NR/EPR also may cause the lower cure rate.
The decreasing of cure time due to RHA loading may be caused by the excess fillers, that shows the denser of vulcanization and cross link site occurred with the RHA and Jatropha oil in NR and NR/EPR composites. Consequently, the competition for cross-liking agents between NR, EPR and the overloading RHA, so that cause in the decreasing of vulcanization rate. Hence, the less mobility matrix polymer due to the EPR addition may cause the vulcanization rate of NR/EPR/RHA is lower than the NR/RHA composites. As the decreasing cure time, the vulcanization process may be explained as follows: both rubber filler (NR or NR/EPR with RHA) network formation and rubber network (NR or NR/EPR) degradation occur simultaneously, but the initial network formation rate was lower than the degradation rate, so that, an optimum level can be reached before the rate of network structure degradation finally became shorter for the NR without EPR than NR/EPR composites [14, 15].

As the RHA loading increases along with EPR addition, the cure time index relatively decreases. It could be explained that the more heat generated from shear force during melt mixing due to high viscosity of RHA and EPR. The EPR has high molecular weight produce high melt viscosity during melt mixing and this effects will generate high shear stress for the shearing and breaking layers of RHA filler [16]. The higher viscosity of RHA loading and EPR addition which may shorten the optimum cure time and scorch time [17]. Furthermore, the RHA contains silica and also the metal oxides like MgO, Fe₂O₃, and other organic material impurities that may also increase the chain mobility in NR or NR/EPR blends [1, 2].

Fig. 1 also depicts that increasing RHA filler loading causes an increasing in torque (ML and MH) for NR/EPR blends and only NR. However, the value of ML and MH for NR without EPR addition much lower than the NR/EPR blends. It is indicated that the process ability on mixing RHA in NR is much easier so that it only needs small energy (the ML and MH less than 5 kg.cm) on the compounding process. In addition, 2 phr of Jatropha oil may be able to lubricate and soften the mixing NR/EPR with the RHA filler by increasing the flow resistance, reducing the melt viscosity of the blends, increasing the proccessability NR-EPDM (non polar rubber) with RHA [8, 15]. MH generally correlates with durometer hardness and/or modulus.

In addition, homogenizer/compatibilizers that used for NR compared to the NR/EPR blends, the torque value for MH and ML are about 5-10 kg.cm. The cross link density that assumed as ΔF (MH-ML), as depicted in Fig. 1, the value of ΔF also increasing similarly by the RHA loading. It is predicted due to the stronger bonding interaction between EPR and NR at high RHA filler loading. The strengthening of the rubber network is consistent with the ΔF, demonstrating the increase in the density of the dispersed EPR phase. Hence, the rubber network has significant effect on the mechanical property and elasticity [6].

3.2. Physical-Mechanical Properties of RHA-NR/EPR Blends

The effect of RHA loading on physical-mechanical of NR with and without EPR is shown in Fig. 2. It can be seen that tensile properties decrease with increasing RHA filler (40-70 phr), whereas the hardness experiences a significant increasing. Meanwhile, the EPR addition cause a significant increasing hardness on the composites with same loading of RHA. Conversely, the EPR addition give negative effect on decreasing tensile properties slightly. EPR composition in the blends and the presence of EPR/RHA. According to El-Sabbagh [18] compatibilizers often referred to as inter facial agents which were able to improve inter facial adhesion between otherwise gross-phase-separated polymer pairs by reducing the inter facial energy between the phases.
The effects of RHA on the tensile strength and hardness are shown in Fig. 2. It can be seen that the tensile strength decreased with the increasing the RHA composition (40 to 70 phr) in both NR with EPR and without EPR. The 70 phr RHA addition for both composites yields the lowest tensile strength value about 5-8 N/mm² whereas the 40 phr RHA gives the highest tensile strength value about 8-9 N/mm². This may due to the overloading of RHA in the compounding or carbon black particles not be were homogenized blended. Furthermore, the higher amount of RHA had caused reinforcement effects between the RHA particles themselves, NR and EPR chains. This finally leads to the interfacial interaction reduction between the rubber chains due to the agglomeration of RHA particles. The RHA presence induces rigidity to the matrix rubber, in which an increase in RHA will increase the rigidity [17].

The decreasing trend of tensile strength may be caused by the weak interaction and bonding between rubber particles and RHA particles. However, at the same blend ratio of RHA loading, the tensile strength of NR blends without EPR addition was slight higher compared to NR-EPR blends. NR (cis, 1-4 isoprene) has better tensile properties compared to EPR rubber [18, 19]. Aforementioned, the EPR is non polar rubber that has huge molecular weight, the EPR addition reduces the NR composition in the composites, decrease its compatibility of RHA-NR blending, and restricts the mobility of rubber filler matrix [20]. Furthermore, those phenomena might possibly cause the weak bonding between NR-EPR and NR-EPR-RHA, the incorporation of EPR in NR-RHA was less compatible so that the decreased interfacial crosslinking because the worse adhesion between RHA and NR-EPR. Consequently, the tensile strength of NR-EPR-RHA blending decreased even slightly.

Fig. 2 shows that RHA loading cause the increasing hardness on both NR without EPR and NR with EPR blending. It could be seen clearly that EPR addition on NR-RHA blending at the same number of filler give significant effect on hardness value which about increased 10 shore A. Aforementioned that EPR has properties such as hardness about 40-90 Shore A and it can be compounded until the density 2 g/cm³ [21]. Therefore, it causes the hardness of RHA filled NR EPR has much higher hardness value compared to NR blending without EPR. In addition, EPR is amorphous polymer that contains ethylene 45-85% that enable the blending becomes stiffer [22]. While, NR has an excellent tensile range 500-3500, elongation up to 700% but lower durometer shore A [18]. Thus, based on its properties, the NR filled RHA without EPR may have better tensile properties, whereas it has lower hardness value. In addition, RHA filler added as amorphous with spherical shaped agglomerated, and RHA particles always exceeds one. Therefore, the hardness would increase when it dispersed in EPR that has big molecular size. Other reason for the increasing hardness may possibly due to the improvement in crosslink density of the blend. The elasticity and flexibility of the rubber chain were less when more EPR was incorporated into NR, which also resulted in more rigid and an increase in hardness.
3.3. Tensile Properties of RHA-NR/EPR Blends.

![Figure 3. Compression set and elongation at break of NR and NR/EPR blends using different loading of Rice Husk Ash (RHA).](image)

Fig. 3 illustrates that the increasing RHA filler on both NR with and without EPR addition would decrease the compression set slightly. Similarly, the addition of RHA composition for both composites also cause a significant decrease on elongation at break. At the same number of RHA composition, the EPR addition on RHA-NR composites give significant effect on increasing the value of compression set from about 30% up to approximately 60%. Conversely, EPR addition give a slight negative effect on decreasing elongation at break on NR filled with RHA. This could be explained that EPR has advantages properties such as a good compression set and tear resistance but a fair elongation due to the highly ethylene content in the polymer [12, 21]. Thus, the elongation at break of NR/EPR filled RHA has much lower than NR without EPR. Aforementioned that NR has excellent in tensile properties and elongation at break but fair in compression set, thus the EPR addition yield increasing on compression set in the same way with the hardness and modulus properties of NR-EPR filled with RHA. The sudden decrease of elongation at break at 60 phr RHA may due to the uneven distribution of excessive RHA in NR/EPR blends. The overloading RHA sometimes cause a detachment of filler interaction and rubber filler interaction due to the less adhesion between them in the rubber matrix [23]. The high RHA loading enhance the rigidity of NR-EPR. This is due to the higher brittleness of RHA into the composites and attractive forces between silica in RHA and polymer molecules. This forces restricts the free mobility of NR-EPR chains to form cross linked network. Since the RHA particle size used in this work is [2], it produces more interfacial bonding and cause decreasing molecular mobility so that resulting in the stiffening of the matrix [23].
3.4. Morphological Properties of RHA- NR/EPR Blends

![Figure 4. SEM Micrographs of: 1) 500x magnification NR/EPR/RHA (100/30/70) phr; 2) 200x magnification NR/EPR/RHA (100/30/70) phr; 3) 500x magnification NR/RHA (100/70)phr; 4) 200x magnification NR/RHA (100/70)phr.]

To investigate the dispersed RHA particles in NR and NR/EPR blends and in order to examine the effect of EPR addition on NR and RHA composites, the morphologies of disintegrated samples were observed by using SEM and the micrographs are shown in Fig. 4. It could be seen that less homogenous dispersion in the rubber matrix NR with EPR that contain the 70 phr RHA. Non-homogenous dispersion lead to agglomeration and flocculation between filler and rubber matrix that cause negative effect on physic-mechanical properties [22].

It can be seen that the dispersed RHA phase in the NR/EPR is actually the agglomerate of different particles size of RHA and EPR. As previously reported, the rubber particles with high cross linking degree and elastic modulus could be as additional fillers in the vulcanizes, thus it contributes to the rough surfaces as shown in Fig. 1 and 2. Furthermore, more matrix tearing and fewer voids were observed as shown in Fig. 3 and 4, indicates that good interaction between RHA within NR matrix, thus this results are in agreement with the higher tensile properties of NR filled RHA without any EPR. Conversely, rough surface indicates the weaker interaction between RHA within EPR and NR matrix rubber, thus a brittle-type failure occurs. A slight void as shown in Fig. 4 can be attributed to the easy detachment of agglomerated EPR from NR-RHA matrix. This clearly provides supporting evidence for the poor tensile properties when EPR added in the blending [2, 22].

4. Conclusions

Rice Husk Ash were successfully prepared and introduced as a new alternative filler for rubber industrial application particularly for natural rubber blending with EPR. Based on the curing characteristics, the RHA loading influence slightly the scorch time, optimum cure time, cure rate time index and torque (MH and ML) on NR with and without EPR addition. It is inferred that jatropha oil may possibly use as
plasticizers for RHA blending in NR with or without EPR composites. The EPR addition in NR blending give significant effect on increasing the curing characteristics. Thus, at the same RHA composition, the torque and cross link density of NR-EPR blending were much higher compared to only NR. RHA loading on NR with and without EPR composites cause a slight decreasing on tensile strength, elongation at break, and compression set. Conversely, the hardness of those composites tend to increase due to the RHA addition. Furthermore, At the same RHA loading, the EPR addition could improve the hardness and compression set of NR-RHA blending. However, the EPR addition cause a slight effect on the decreasing of tensile strength and elongation at break due to the reduction composition of NR. SEM micrographs shows morphological and surface area that confirmed the effect of both RHA and EPR addition on its physic mechanical and tensile properties. The micrographs provide additional evidences on the poor tensile properties as well as the matrix tearing and voids due to EPR addition into NR composites. To sum up, RHA may be used as an alternative filler for NR composites along with the EPR addition in order to improve compression set and hardness properties particularly for specific rubber products.

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