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# Studies on the Effect of Rice Husk Semi-Nano Filler on the Mechanical Properties of Epoxidized Natural Rubber Composite

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**ABSTRACT**

*The effect of semi nano filler from rice husk on the mechanical properties of epoxidized natural rubber was studied and compared with carbon black filled epoxidized natural rubber composite. The particle size distribution of the milled rice husk was determined using optical microscopy and digital imaging technique. The semi nano powder obtained by ball milling of the rice husk was characterized for pH, iodine value, cellulose content, lignin content, hemicellulose content, moisture content, bulk density and loss on ignition. The result of the physico-chemical analysis showed excellent values which compared favourably with other literature reports. Optical microscopy and digital imaging of the rice husk nano filler showed a spherical particle of average size 221nm. The results of the mechanical testing presented similar trends and close values for both carbon black filled epoxidized natural rubber and rice husk semi-nano filled epoxidized natural rubber at filler loading in the range 10 - 50phr. Consequently, rice husk nano filler can be a good substitute for carbon black for products where cost, compression set and rebound resilience properties are critical.*

**Keywords:** Epoxidized natural rubber, ball milling, semi-nano filler, carbon black, vulcanizate, flex fatigue

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## INTRODUCTION

Fillers are major additives in rubber compounding as their incorporation enhances mechanical properties, processibility as well as reduce cost of production. Filler can be reinforcing or non-reinforcing. While reinforcing fillers improves the mechanical properties of the rubber products, the non-reinforcing fillers serve only to reduce cost and act as diluents [1]. Examples of the reinforcing fillers are carbon black, silica, etc. while the non-reinforcing fillers include mica powder, barium sulphate, calcium carbonate etc. [1-2]. The efficiency of reinforcement in a vulcanizate depends on the particle size, particle shape, particle dispersion, surface areas, surface reactivity, structure of the filler and the bonding quality between the fillers and the rubber matrix [1, 3]. Hence, fillers with particle size on the nano scale have high specific surface area for better polymer matrix-filler interaction.

Numerous researches have been carried out to develop nano-filled rubber composites through the incorporation of nano-scaled materials such as ceramics and carbon in polymer matrices. For instance, organo clays, carbon nano tubes, alumina nano particles and silica nano particles have been added to polymers. Polymer nano composites exhibit exceptional properties even with the addition of low fillers [3]. The incorporation of nano fillers enhances mechanical, electrical, optical and other properties of polymer composites without sacrificing too much of the good properties such as the toughness being traded for the stiffness as such found in the rubber filled carbon fibres.

Several polymers have been used for preparing polymer nano composites such as elastomers (natural rubber), epoxidized natural rubber (ENR), styrene-butadiene rubber (SBR), chloroprene rubber (CR), ethylene propylene diene monomer rubber (EPDM) etc.), thermoplastics e.g. nylon 6, 6, polypropylene (PP), poly (ethylene terephthalate) (PET), poly (methyl metacrylate) (PMMA), polycarbonate etc., and several polymer blends [3-4]. ENR is miscible with more polar polymers thereby offering unique properties such as good oil resistance, low gas permeability, higher wet grip, rolling resistance, and a high strength. The most common reinforcing filler used in the rubber industry is carbon black. However, the growing concern on environmental sustainability have called for the use of renewable and environmentally friendly reinforcing fillers mostly from agricultural wastes. Rice husk is one of such agricultural wastes with great potentials for industrial applications. Rice husk is a cellulose-based material highly available and demonstrating attractive properties like toughness, resistance to weathering and unique chemical composition.

Many methods and techniques have been employed by different researchers to obtain semi nano or nano fillers from rice husk or rice husk ash. Ludena et al [5] produced nanocellulose from rice husk using chemical treatments. Esterves et al [6] produced from rice husk ash nano silica particles by worms using a bio-digestion process. Nittaya et al [7] obtained nano silica by precipitation method. Several other researchers had also obtained nano silica by mechanical methods of ball milling and grinding [8-9].

This research focuses on obtaining semi nano filler from rice husk by ball milling, incorporating it as filler in epoxidized natural rubber and comparing the mechanical properties with carbon black filled epoxidized natural rubber.

## MATERIALS AND METHOD

### Materials

Rice husk were sourced locally from Bende, Abia State, Nigeria. The Epoxidized Natural Rubber (ENR-50) was obtained from Rubber Research Institute, Iyanomoh, Benin, Nigeria. All chemicals used were analytical grades and products of British Drug House (BDH), England. The chemicals include Tetramethyl thiuram disulphide (TMTD), Mercapto benzothiazole sulphide (MBTS), stearic acid, sulphur, Trimethylquinoline (TMQ), zinc oxide, processing oil (paraffin wax) and N330 carbon black.

The equipment used include Retch New Planetary mill series (Pm 100), Monsanto Tensile Tester Model 1/m (British Company Ltd., England), Wallace Hardness Tester Model c8007/25 (Elektron Technology Series, UK), Wallace Akron Abrasion Tester (Elektron Technology Series, UK), DuPont machine (British Company Ltd., England) etc.

### Method

### Preparation and Characterization of Nano Filler

Rice husk was winnowed to remove sand particles and other adhering foreign bodies and then washed. The washed rice husk was placed in the planetary mill with a spherical grinding media which consists of planetary balls (< 0.1mm diameter) made of hardened steel (0.24 to 0.95cm diameter). The rice husk and the grinding media were placed in a stationary tank followed by an agitation with an armed shaft rotating at 250rpm. The forces of shear and impact exerted by the grinding media on the rice husk reduces it to a dispersion of fine powder. The resultant slurry formed was discharged, air-dried and further oven-dried. The cake was ball milled using the top-down technique (i.e. critical speed grinding under a continuous process of approximately 60hrs) to 200-300nm size. Standard tests method was used to characterize the semi-nano powder for lignin content, hemicellulose content, cellulose content, moisture content (ASTM 1509) at 125$^{o}$C, bulk density, loss on ignition (ASTM D7348), pH (ASTM 1512) and iodine value.

### Determination of the Particle Size of the Rice Husk Semi-nano Filler by Optical Microscopy

Particle size was determined using optical microscopy and digital imaging technique. The images were viewed and digitalized with Olympus digital camera at 4080 x 3072 pixel.

### Processing of the Composite

The compounding was done on a two-roll mill in steam-heated presses using the formulation shown in table 1. Formulation was carried using ASTM D3184-80 method. The mixing cycle is as shown in table 2 and was maintained at 70$^{o}$C. The compounded rubber was vulcanized using a Hydraulic Press (Elektron Technology Series, UK) at a pressure of 3.5bar for 12mins. The moulds were removed immediately and cooled in a large cold water tank for about 10mins. The sheets were wiped dry and stored for 24hrs before testing.

**Table -1 Formulation for Compounding Epoxidized Natural Rubber**

| Ingredient | Parts per hundred rubber (phr) |
|---|---|
| Epoxidized Natural Rubber (ENR-50) | 100 |
| Paraffin Wax | 5.0 |
| Zinc Oxide | 5.0 |
| Stearic acid | 2.5 |
| Sulphur | 1.5 |
| MBTS | 1.5 |
| TMTD | 3.5 |
| Fillers (Rice Husk or carbon black) | Variable (10 - 50) |

A batch factor of four (4) was used.

**Table -2 Mixing Steps and Mixing Time**

| Mixing steps | Time (minutes) |
|---|---|
| Epoxidized Natural Rubber Mastication | 8 |
| Addition of Stearic acid | 1 |
| Addition of Zinc Oxide | 1 |
| Addition of filler | 10 |
| Addition of MBTS | 1 |
| Addition of TMTD | 1 |
| Processing Oil | 1 |
| Addition of Sulphur | 2 |
| **Total** | **25** |

### Mechanical Testing of the Vulcanizates

The mechanical properties of the vulcanizates were determined using standard test procedures tensile test (ASTM D638), hardness test (BS 903 part A26), compression set test (ASTM D385), resilience (ASTM D430), flex fatigue (ASTM D430), and abrasion resistance test (BS903 part 49 method C).

## RESULTS AND DISCUSSION

### Characterization of the Rice Husk Nano Filler

The results of the characterization of the rice husk nano filler are presented in table 3. The value for the cellulose content was within the range (25-35%) reported by Premalal et.al [10] and the range (28-48%) by Naheed et.al [11]. The hemicellulose content compares favourably with the range of values (18-21%) also reported by Premalal et.al [10] while the lignin content deviated from the range of values (26-31%) reported by the authors. However, the lignin content was in tandem with the values (12-16) presented by Naheed et.al [11]. The moisture content was found to be very low compared to values reported by previous authors. Premalal et.al [10] stated values between 5-10% and Ajay et.al [12] reported values in the range of 8-9% in their review. Yaning et al [13] in their study of the physical properties of varieties of rice husk from different continents obtained moisture contents of 4.72% for long grain rice husks, 5.63% for short grain rice husk, 4.60% for cascara de aruz rice husk and 6.07% for japonica rice husk. Generally, the physico-chemical content of rice husk could among other things will depend on rice variety, climatic conditions, soil conditions and locality. Hence, these factors could also account for the very low moisture content obtained in this study for the rice husk semi nano filler.

The value obtained for the bulk density was higher than the range of values (96-160kg/m3) reported by Ajay et.al [12]. Also, Mansaray and Ghali [14] obtained values ranging from 86kg/m3 to 114kg/m3 for rice husks for six varieties of rice. The high value obtained in this study could be attributed to the particle size of the rice husk semi nano filler which was very low compared to the micro sizes of those reported.

The iodine value reveals the amount of iodine adsorbed per 100gram of the nano silica sample. One important application of iodine value is that it elicits the surface area of the rice husk powdered sample and indicates the microstructure of the filler and reflects it reaction and adsorption abilities. Hence, the value obtained shows that the surface area of the rice husk semi nano filler is 19.05 mg/g. A high surface area gives better reinforcing properties and results in higher modulus at higher strain, higher abrasion resistance and lower hysteresis.

Loss on ignition is a measure of the carbon content lost during combustion. The value obtained for the loss on ignition of the rice husk semi nano filler shows that it contains more non- carbon elements and 7.75% of carbon. The larger the non- carbon elements of the agricultural by-product, the lower the reinforcing, as filler for elastomer [1].

The pH result showed that rice husk semi nano filler was slightly alkaline. The pH of filler affects the cure rate of mix of elastomers. pH at alkaline level tends to accelerate cure rate and hence increase the cross-links density.

**Table -3 Physico-Chemical Characterization of Rice Husk Nano Filler**

| Characteristic (Unit) | Value |
|---|---|
| Loss on Ignition (%) | 7.75 |
| Cellulose content (%) | 29 |
| Hemicellulose Content (%) | 21 |
| Lignin Content (%) | 18.25 |
| Bulk Density (kg/m$^3$) | 720 |
| Iodine value (mg/g) | 19.05 |
| Moisture content (%) | 1.80 |
| pH | 7.15 |

### Particle Size of Rice Husk Semi-Nano Filler from Optical Microscopy

The optical microscopy and digital imaging of the nano filler are presented in Fig.s 1a, 2a and 3a. Each dust particle was spherical in shape and the area of the dust particle is the area given in excel as shown in Fig.s 1b, 2b and 3b. For an area past 10 square pixels, the dust particles are now agglomerates or aggregates. From measurements, 1 pixel is approximately equal to 900/4080 = 0.221micrometer (microns) or 221nm.

1 square pixel therefore means that the dust particle has a radius of 49 squared nm.

Minimum radius124nm (0.124microns)

Maximum radius 394nm (0.394microns)

Mean value is 0.184 microns

Distribution shows that though there are aggregates, but independent rice husks particles do indeed dominate the whole system.

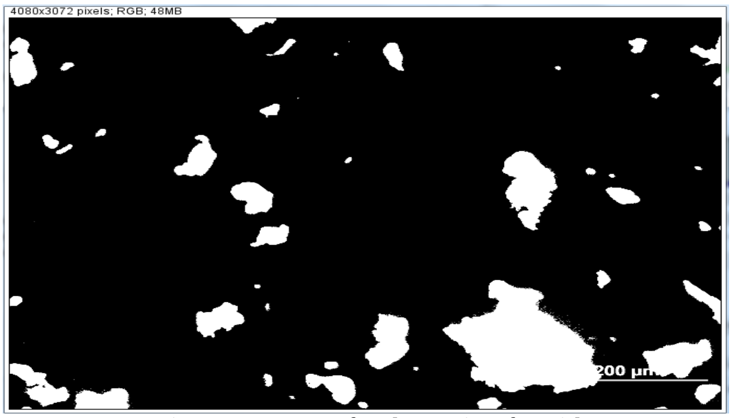


**Fig. 1a OM Images of agglomeration of particle**

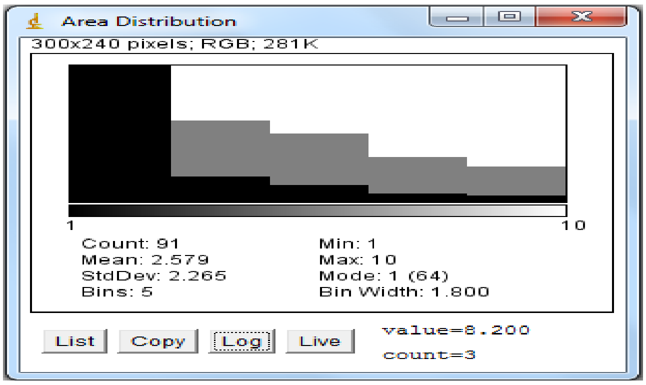


**Fig. 1b OM agglomeration result**

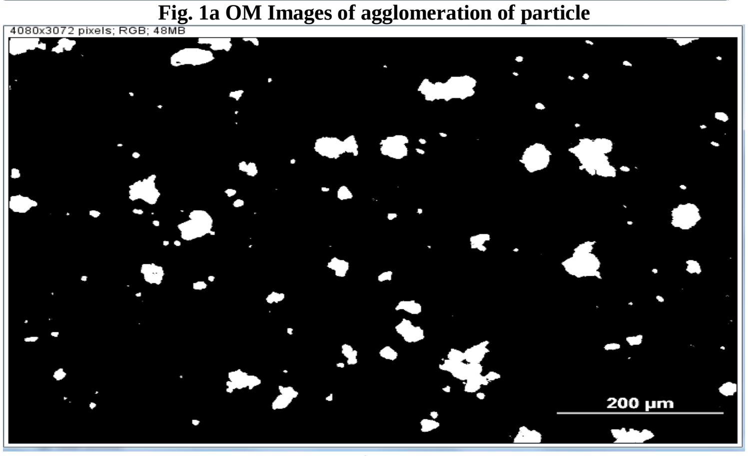


**Fig. 2a OM Images of agglomeration particle**

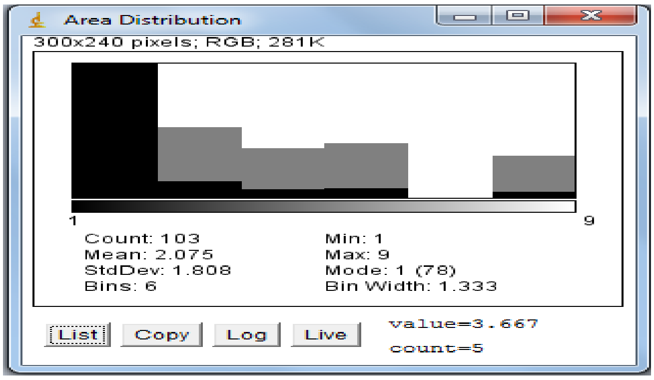


**Fig. 2b OM agglomeration result**

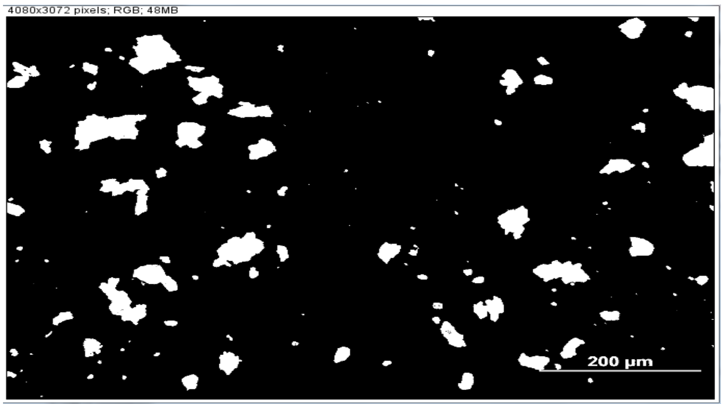


**Fig. 3a OM Images of agglomeration of particle**

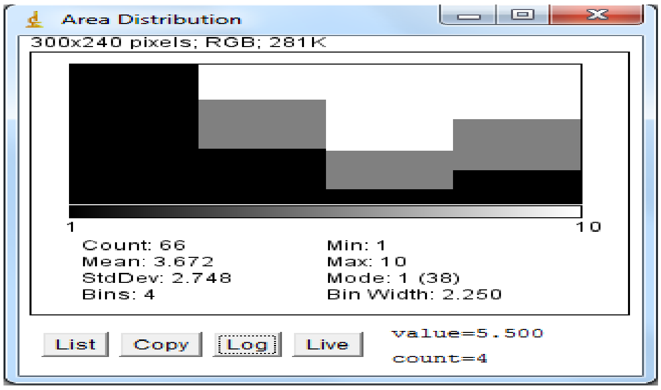


**Fig. 3b OM agglomeration result**

**Table -4 Mechanical Properties of the Vulcanizates**

| Property | Filler Loading (phr) | | | | | |
|---|---|---|---|---|---|---|
| | 0 | 10 | 20 | 30 | 40 | 50 |
| Tensile Strength (MPa) | 17.50 | (18.50) [19.10] | (20.00) [23.30] | (22.50) [24.50] | (23.20) [25.60] | (24.50) [30.20] |
| Modulus (MPa) | 6.15 | (6.90) [8.00] | (8.00) [9.60] | (8.90) [10.20] | (10.10) [11.00] | (12.40) [13.50] |
| Elongation at Break (%) | 550 | (530) [506] | (512) [489] | (502) [474] | (488) [450] | (480) [435] |
| Hardness (IRHD) | 32.60 | (40.60) [46.30] | (44.30) [50.50] | (49.80) [58.70] | (59.10) [66.40] | (65.50) [73.80] |
| Compression Set (%) | 27.00 | (30.80) [28.90] | (29.10) [28.00] | (27.90) [25.20] | (25.60) [23.90] | (23.20) [21.40] |
| Abrasion Resistance ($mm^3$/500rev) | 10.35 | (11.46) [13.40] | (12.80) [14.00] | (14.00) [15.40] | (15.20) [16.00] | (15.50) [16.30] |
| Rebound Resilience (%) | 47.50 | (42.40) [37.80] | (39.80) [35.60] | (39.00) [34.90] | (38.20) [34.00] | (37.10) [32.40] |
| Flex fatigue (kc x $10^3$) | 7.50 | (7.25) [6.25] | (6.02) [5.50] | (5.80) [5.00] | (5.65) [4.50] | (4.50) [4.20] |

Key Semi Nano Rice Husk filled ENR-50 Vulcanizates ( ) Carbon Black filled ENR-50 Vulcanizates [ ]

Epoxidized natural rubber inherently has high tensile strength owing to strain-induced crystallization. On incorporating fillers into the matrix, the regular arrangement of rubber molecules is disrupted resulting in loss of strain-induced crystallization [3]. Tensile strength of epoxidized natural rubber composites as a function of filler loading is shown in Table 4. Introduction of fillers into the epoxidized natural rubber produced a rubber composite with higher tensile strength as compared to the unfilled epoxidized natural rubber with lower tensile strength. The tensile strength of epoxidized natural rubber increased with increase in filler loading as earlier established by previous workers [3, 15]. The effect of filler loading on tensile strength of filled epoxidized natural rubber is presented in Fig. 4. It can be seen that tensile strength increases with increasing filler loading. This is in agreement with the works of previous researchers who used coconut shell and palm fruit fibre fillers [16]. It can be seen that the tensile strength of the filled vulcanizates increases with filler loading from 18.50 – 24.50MPa for semi nano rice husk powder filled ENR-50 vulcanizates and 19.10 – 30.20MPa for carbon black filled ENR-50 vulcanizates as filler loading increases from 10 - 50phr. This implies that carbon black is out performing rice husk semi-nano filler by about 3.14% - 18.87%. This does not take away anything from the potential use of rice husk as a filler since it is a waste product which becomes attractive when cost considerations are taken into account. Other researchers had also reported similar trend in tensile strength of rubber vulcanizates with increase in filler loading when working with palm kernel shell reinforced epoxidized natural rubber [17]. This behaviour could be attributed to enhanced filler dispersion in the epoxidized natural rubber matrix.

Fig. 5 represents the modulus of the filled ENR-50 vulcanizates at filler loading from 10-50phr. A gradual increase in modulus is observed with increasing filler loading for both vulcanizates. It could be noticed also that carbon black filled ENR-50 vulcanizates exhibited a higher modulus than semi nano rice husk filled ENR-50 suggesting better interaction of the carbon black fillers with the ENR-50 rubber matrix.

The reinforcing potential of fillers is affected by factors which include filler dispersions, surface area, surface reactivity, bonding capacity (quality), and particle size [18]. As earlier pointed out, the effectiveness of filler may be measured by its surface area available for compatibility [9]. Fillers with higher carbon content, provide greater reinforcement than those with lower carbon content because carbon itself is very good reinforcing filler [6]. This informs why the carbon black filled ENR-50 vulcanizate exhibited higher tensile strength and modulus compared to the rice husk nano filled ENR-50 vulcanizate. Rice husk fillers are also known for their easy agglomeration, whose presence may generate flaws and create voids resulting to poor interfacial bonding between the filler and the rubber matrix.

The effect of the filler loading on the elongation at break is shown in Fig. 6. The values of elongation at break (EAB) decreases with increase in filler loading for both semi nano rice husk filled ENR-50 and carbon black filled ENR-50 vulcanizates. A decrease in elongation at break is attributed to the adherence of the filler to the rubber polymer matrix leading to the stiffening of the polymer chain and hence resistance to stretch on application of strain [15]. Semi nano rice husk filled ENR-50 rubber vulcanizates exhibited higher values than the carbon black filled ENR-50 vulcanizate. This shows poor adherence of the rice husk filler to the rubber matrix when compared with the carbon black filler.

The hardness of semi nano rice husk filled ENR-50 and carbon black filled ENR-50 vulcanizates increased with increasing filler loading as shown in Fig. 7. This result is expected because as more filler particles get into the rubber, the elasticity of the rubber chain is reduced, resulting in more rigid vulcanizates [9]. The hardness results of carbon black filled ENR-50 vulcanizates are higher than those of semi nano rice husk filled ENR-50 rubber depicting better penetration of the carbon black filler in the ENR-50 rubber matrix as against the rice husk filler. Hardness is also a factor of cross-link density; the more cross linking a material undergoes during vulcanization, the harder the final vulcanizate will be. Therefore, carbon black filler formed stronger crosslinks with the rubber matrix than the rice husk filler.

Compression set is useful in prediction of the service performance of rubber articles. The level of compression determines the service life and area of application of the rubber composites. The results of compression set in Fig. 8 shows that the compression set of the filled vulcanizates decreased with increasing filler loading. The values obtained for the carbon black filled ENR-50 vulcanizate are lower than those obtained for the semi nano rice husk filled ENR-50 vulcanizate. The results support the reports of Ogbeifun et al [19] who also obtained values of compression set for carbon black filled rubber vulcanizate lower than those of carbonized agricultural wastes filled rubber vulcanizates.

The values of rebound resilience decreased with increasing filler loading of the mixes for all the vulcanizates as shown in Fig. 9, with semi nano rice husk filled vulcanizate exhibiting higher values than the carbon black filled counterpart. Resilience is related to the flexibility of molecular chains of the vulcanizate; the more flexible the molecular chains, the better the resilience [20]. Resilience is most critical in dynamic seals and hence semi nano rice husk filled ENR-50 will perform better in such applications.

The abrasion resistance of a solid body is defined as its ability to withstand gradual wearing of its surface caused by mechanical action of rubbing or scraping. The trend of abrasion resistance with filler loading presented in Fig. 10 shows a regular pattern of increase with increasing filler contents for both semi nano rice husk filled ENR-50 and carbon black filled ENR-50 vulcanizates, indicating that filler loading is a function of the measured parameter. Carbon black filled ENR-50 vulcanizates presented higher (though not appreciable) values when compared with semi nano rice husk filled ENR-50 vulcanizates. This could be attributed to the strength of the bond between the fillers and the rubber matrices. A weak bond will cause easy detachment of the filler particles from the rubber matrix leading to wear when subjected to mechanical action. Hence, carbon black filler bonded strongly to ENR-50 than the semi nano rice husk filler.

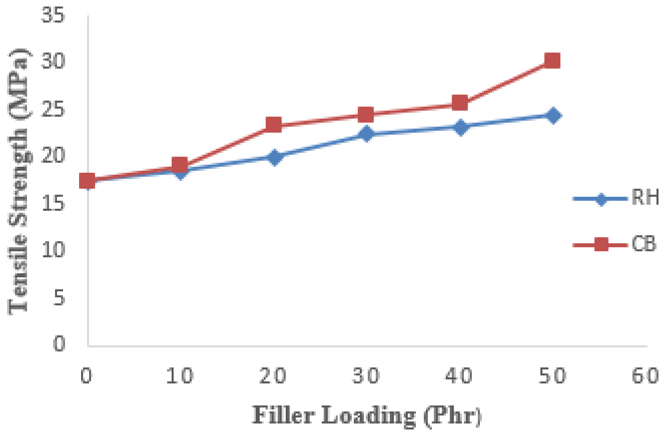


**Fig. 4 Tensile strength of filled vulcanizates at varying filler loading**

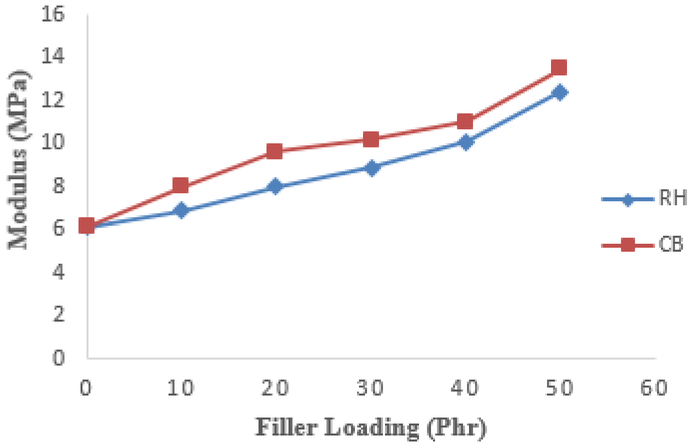


**Fig. 5 Modulus of filled vulcanizates at varying filler loading**

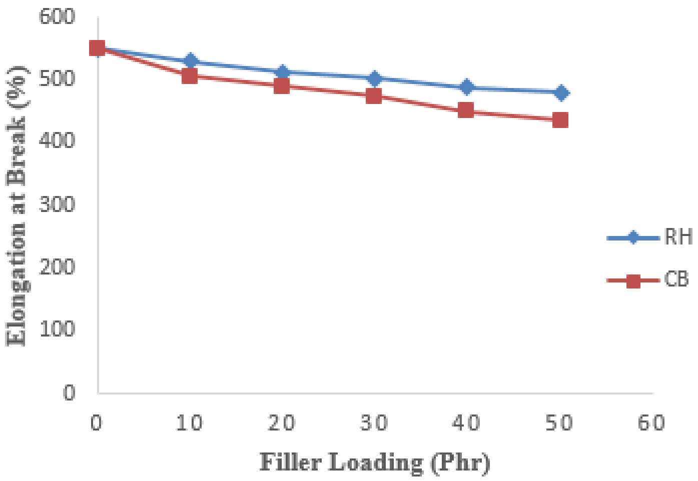


**Fig. 6 Elongation at break of filled vulcanizates at varying filler loading**

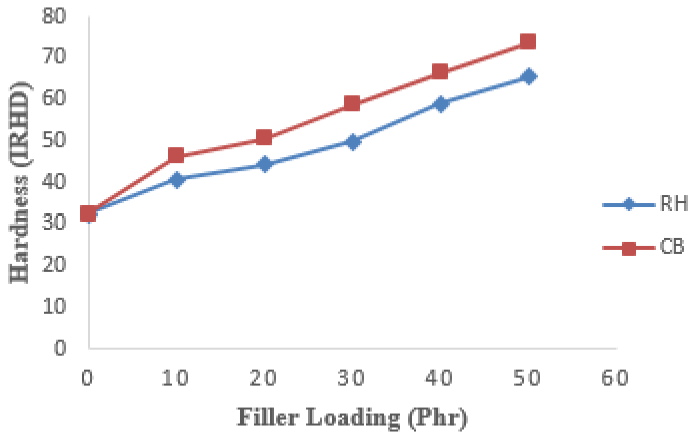


**Fig. 7 Hardness of filled vulcanizates at varying filler loading**

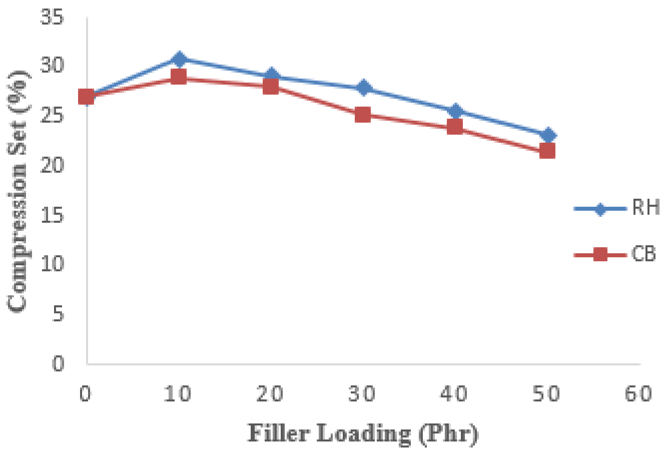


**Fig. 8 Compression Set of filled vulcanizates at varying filler loading**

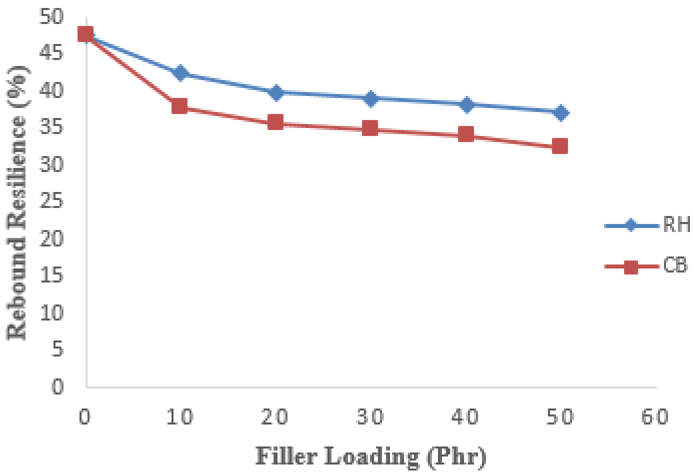


**Fig. 9 Rebound Resilience of filled vulcanizates at varying filler loading**

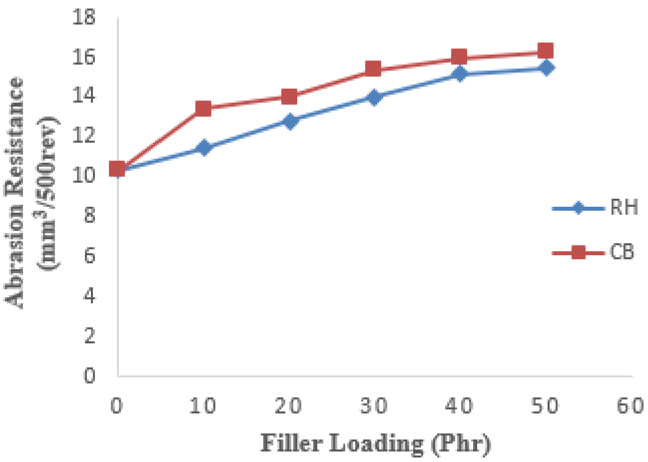


**Fig. 10 Abrasion Resistance of filled vulcanizates at varying filler loading**

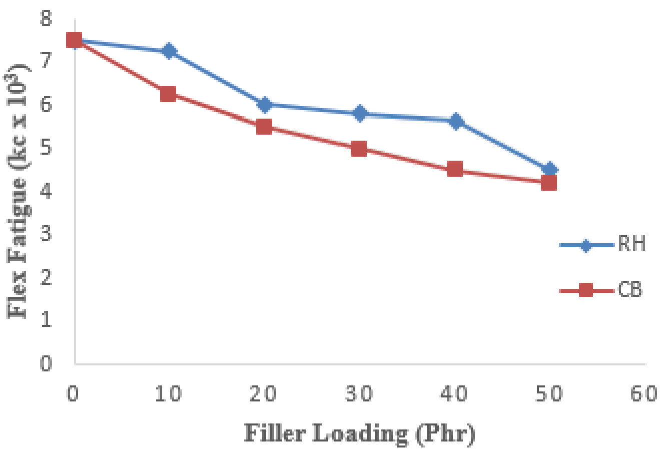


**Fig. 11 Flex fatigue of filled vulcanizates at varying filler loading**

Flex fatigue is the cracking of a rubber component due to cyclical stresses and strains. The cracking starts off at molecular level and with successive cycling, it propagates into macro-cracking that ultimately results in product failure. The incorporation of the fillers reduced the flex fatigue of the rubber material. Fig. 11 illustrates a falling trend in the flex fatigue of the filled rubber vulcanizates with increasing filler loading with rice husk filled vulcanizate exhibiting the highest values. While filler incorporation reduces flex fatigue, it is worthy to note that products made from rice husk filled vulcanizate will fail easily than products made from carbon black filled vulcanizate.

## CONCLUSION

Semi nano powdered filler from rice husk has demonstrated its potential reinforcing ability in epoxidized natural rubber. This research work examined how favourably the mechanical properties of rice husk semi-nano filled epoxidized natural rubber vulcanizate compares with those of carbon black filled vulcanizates. The results obtained showed that mechanical properties of rubber vulcanizates are greatly influenced by filler loading. It was observed that carbon black filled ENR-50 vulcanizates exhibited higher tensile strength, modulus, hardness and abrasion resistance. However, the compression set, elongation at break and flex fatigue were lower than those of the rice husk semi-nano filled ENR-50 vulcanizate.  This indicates that the rice husk semi-nano filled ENR-50 vulcanizate would be useful in the manufacture of products requiring lower stress but higher compression set, rebound resilience and elongation at break in their service life. Nevertheless, when cost and availability seem to be the paramount factors in the choice of filler, rice husk semi-nano filler can serve as a better filler than carbon black.

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