PRODUCTION AND QUALITY ASSESSMENT OF AUTOMOBILE COMPOSITE PART (EXHAUST PIPE SUSPENDER) FROM NATURAL RUBBER FILLED WITH CHEMICALLY TREATED RICE HUSK

Ayo, M. D.
Auchi Polytechnic, Auchi, Edo State, Nigeria
E-mail address: dadaayo2335@gmail.com

Chukwu, M. N.
Auchi Polytechnic, Auchi, Edo State, Nigeria

Momoh. F. P.
Auchi Polytechnic, Auchi, Edo State, Nigeria

ABSTRACT

The research is on the manufacture of automobile part (exhaust suspender) from natural rubber filled with chemically treated rice husk powder. Rice husk were obtained from a mill in Auchi, Edo State, Nigeria and shared into four parts. They were separately treated with 10% benzoyl chloride, 10% sodium hydroxide and 10% hydrogen peroxide and one part was untreated. The various samples were ground, filtered, analyzed in terms of FTIR and SEM then used in compounding natural rubber. The rubber vulcanizates were vulcanized and the mechanical properties were determined. The FTIR results show the reduction in hemicellulose and lignin content for the treated rice husk while the SEM micrograph showed a finer structure for the treated rice husk. This may suggest stronger reinforcement observed in the composites filled with treated filled. There was improvement in mechanical properties of the composites. This show that chemically treated rice husk is a potential substitute for other fillers for automobile exhaust suspender or foot mat and oil seals.

Keywords: Composite, Filler, Rubber, Characterization, Rice-husk

INTRODUCTION

Rubbers as one of the classes of polymers have found great important application in engineering, sport, leisure and domestic. In its raw state, rubbers may not be good enough for any useful application, so there is the need for the addition of additives which help to enhance their properties. Among these additives are varieties of different fillers. Carbon black is the major filler used in the rubber industry. Today, there are lots of researches going on to find alternative for it (Ayo, Ekebafe, Chukwu and Madufor, 2011). Fillers have marked effect on the physical and chemical properties of vulcanisates. Tensile and modulus properties could be enhanced using appropriate fillers. Therefore, selecting a suitable filler for the polymer matrix is a key consideration (Akinlabi, Olayinka, Dare and Oyenekan, 2011).

Rice husk waste is a potential candidate for the development of new composites because of its possible strength and preferred modulus properties. Composites of high strength filler can used in broad range of applications. However, it should be noted that rice husk filler like all other natural fillers suffer certain disadvantages which the present work sought to correct and improve upon. The work therefore emphasizes the modification of rice husk in an attempt at combating these short comings. The reasons for the application of natural fibre in the automobile industry include: low density, which may lead to weight reduction of 10 to 30%, acceptable mechanical properties, favourable processing properties such as high stability, less splintering and eco-balance for part production, occupational health benefits and price advantage (Mizahur, Mubarak and Khan, 2007).

EXPERIMENTAL

Rice husk used in this study were obtained from a rice milling machine in Auchi, Edo State, Nigeria. The natural rubber used was obtained from Nigeria Rubber Research Institute, Iyanomo, Benin, Edo State, Nigeria. Sodium hydroxide, benzoyl chloride, hydrogen peroxide were obtained from Titan Biotech Ltd, India. These materials were used as received. The compounding ingredients such as zinc oxide, stearic acid, sulphur, Trimethylquinoline (TMQ), mercaptobenzothiazole sulphenamide (MBTS) and processing oil were obtained from British Drug House, England.

Sample Preparation

Rice husk from the milling machine were obtained and divided into four parts. One part was treated with 10% sodium hydroxide solution for one hour at room temperature. The solution was then filtered and the residue was thoroughly washed with distilled water, dry at room temperature for 48 hours follow with oven drying at 70^0C for 2 hours.

The second and third parts were separately treated with 10% benzoyl chloride and hydrogen peroxide solutions, filtered and the various residues were washed with distilled water and air dry at room temperature for 48 hours and then oven dry at 70^0C for 2 hours. The fourth part was not treated. The various treated rice husk and the untreated rice husk were ground and filter with a sieve of mesh 75µm.

Characterization of the Filler

The Fourier Transform Infrared of the powdered samples were carried out using the Fourier's Transform Infrared Spectrometer Nicolet IS10 FTIR spectrometer and the morphology of the powdered samples were carried out using Scanning Electron Microscope, Joel-JSM 7600F(1).

Processing of the Composite

This involves compounding of natural rubber with powdered rice husk filler. The formulation used for the compounding of the natural rubber with the treated and untreated rice husk powder filler is given in table 1.

Table 1: Formulation for Compounding Natural Rubber with Powder Rice Husk

Ingredients	**Parts per hundred rubber**
Natural rubber	100.0
Filler (rice husk powder)	10, 20, 30, 40 and 50
Zinc oxide	5.0
Stearic acid	2.5
Sulphur	2.5
MBTS	1.0
TMQ	1.0
Processing oil	5.0

Mixing Procedure

The rubber mixes were prepared on a laboratory size two roll mill according to the mixing cycle shown in table 2. It was maintained at 70^0C to avoid crosslinking during mixing after which the rubber composite was stretched out. Mixing follows ASTMD 3184-80 Standard.

Table 2: Mixing Steps and Mixing Time

Mixing Steps	Time (minutes)
Natural rubber mastication	5
Addition of stearic acid	1
Addition of zinc oxide	1
Addition of filler (rice husk)	10
Addition of MBTS	1
Addition of TMQ	1
Processing oil	1
Addition of sulphur	2
Total	22

Composite Curing

The curing of test pieces was done in a compression moulding machine. The curing was carried out at 140^{0}C for 10 minutes.

Mechanical Properties of the Composite

The mechanical properties of the composites were determined using standard test procedures. The tensile properties determination was carried out in accordance with ASTMD 412-87 method. Hardness Test was done in accordance with ASTMD 785. ASTMD 385 method was used for compression set determination. The flex fatigue determination was carried out using ASTMD 430 using the flex tensometer machine. Wallace Akron abrasion tester was used for abrasion resistance determination.

RESULTS AND DISCUSSION

Characteristics of the Powdered Filler

Figure 1: FTIR of the untreated Rice husk Powder

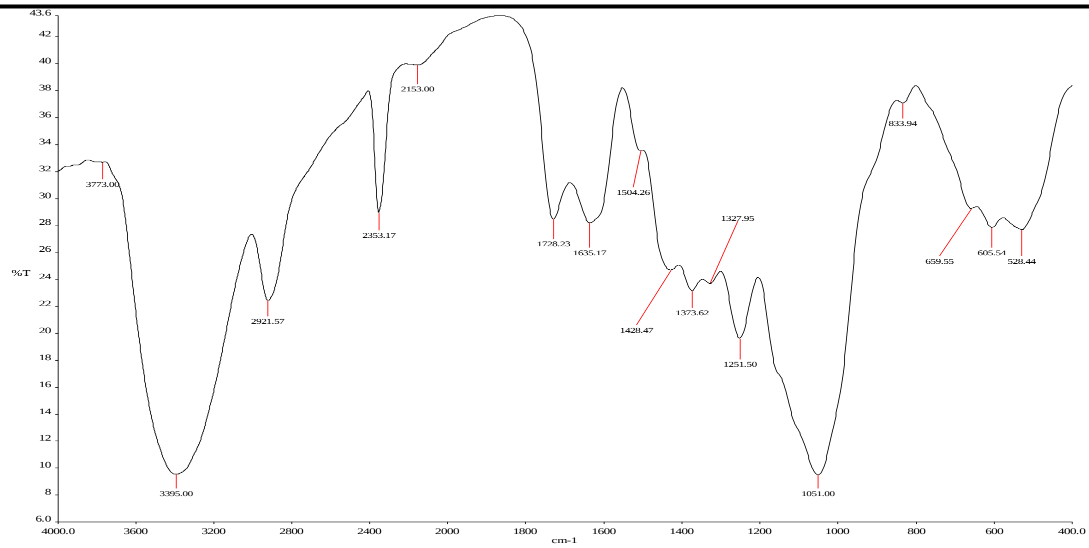

Figure 2: FTIR of the Mercerized treated Rice husk Powder

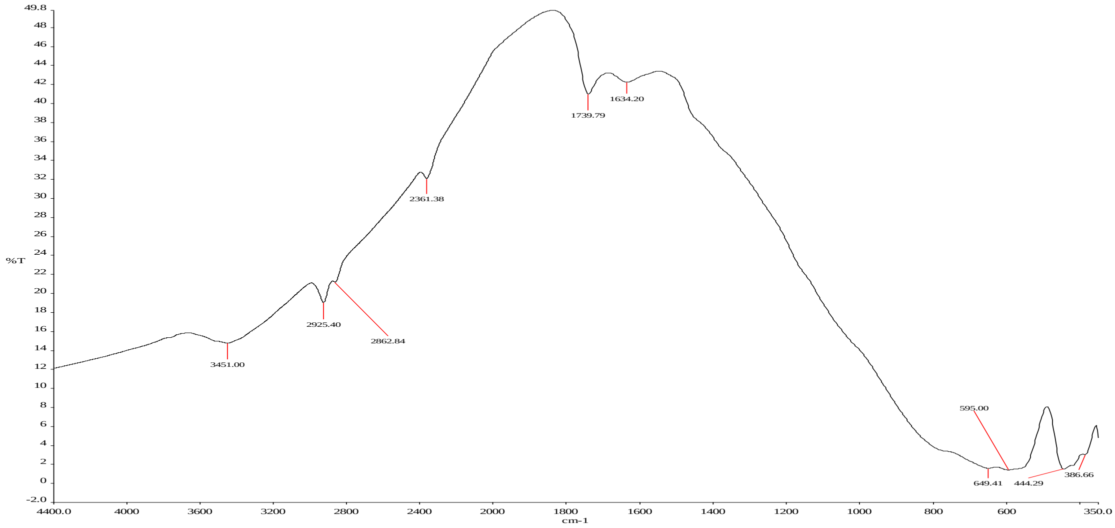

Figure 3: FTIR of the Benzoyl chloride treated Rice husk Powder

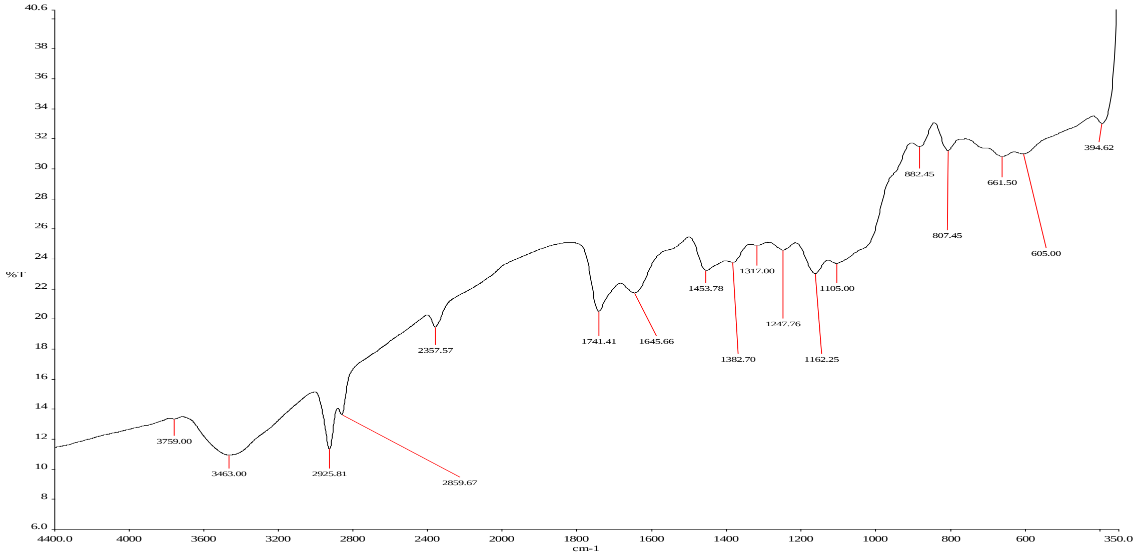

Figure 4: FTIR of the Hydrogen peroxide treated Rice husk Powder

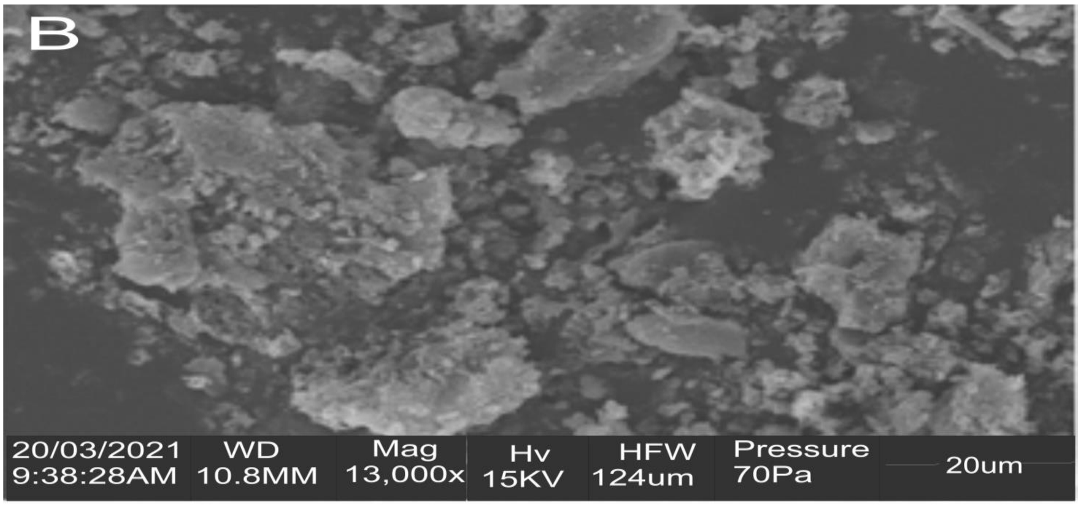

Figure 5: SEM Micrograph of the Benzoyl chloride treated Rice husk Powder

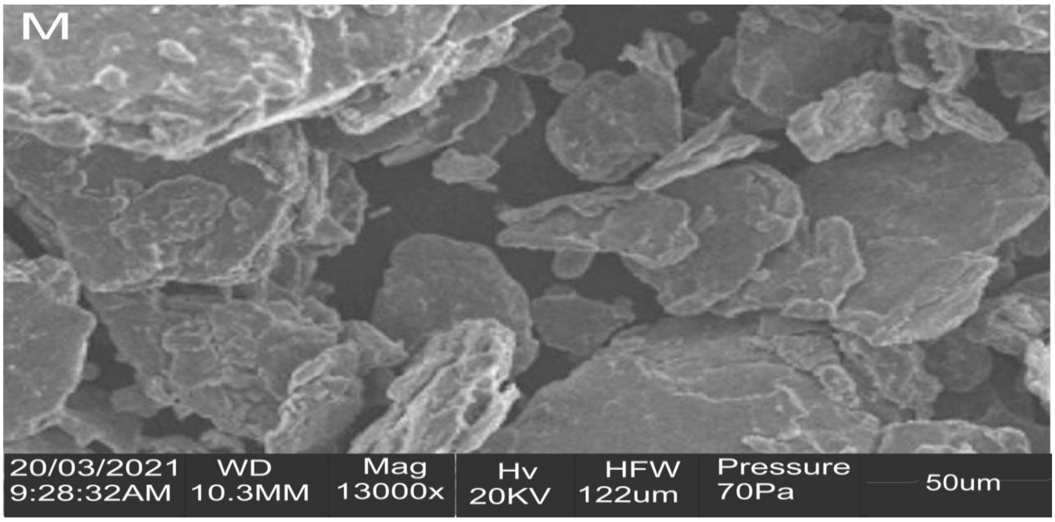

Figure 6: SEM Micrograph of the Mercerized treated Rice husk Powder

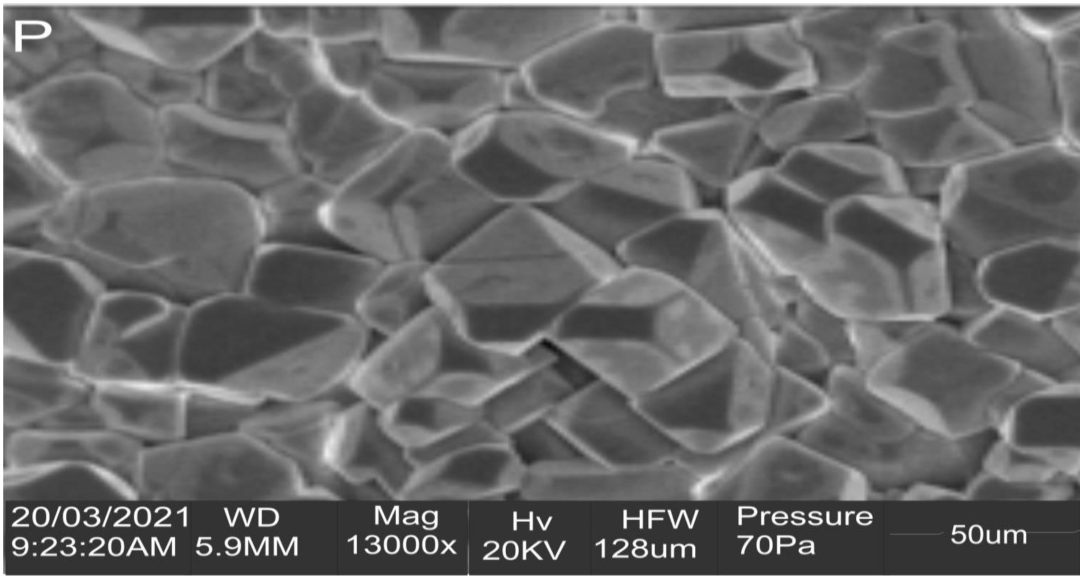

Figure 7: SEM of the Hydrogen peroxide treated Rice husk Powder

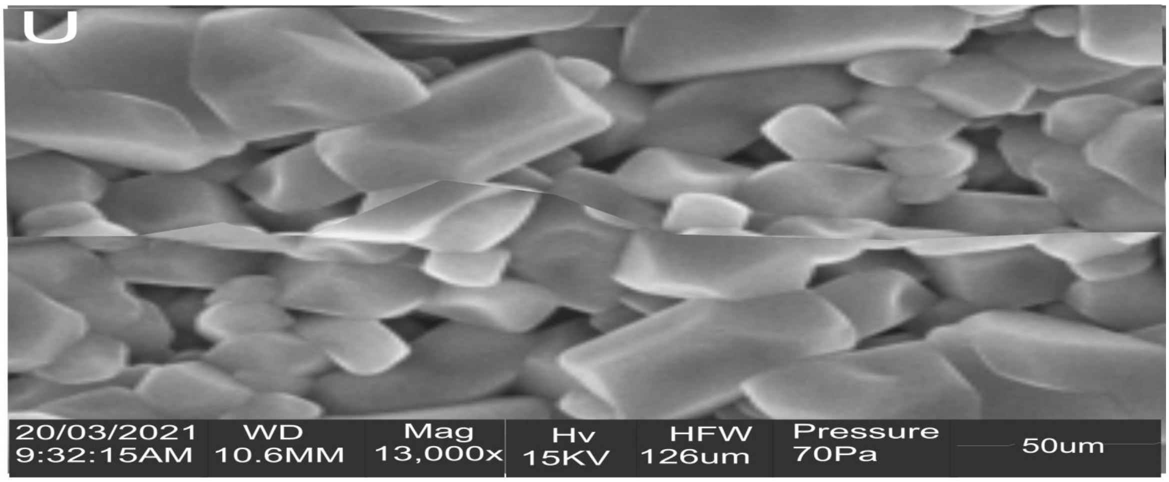

Figure 8: SEM Micrograph of the untreated Rice husk Powder

The results of the characteristic samples are presented in Table 1 above; there was decrease in the moisture absorption for the treated samples. Reduction of about 50% moisture uptake for acetylated jute fibres has been reported by Bledzki and Gassan (1999).

The result of the chemical constituents showed a progressive decrease on percentage of lignin and hemicellulose while there was a significant increase in percent of the cellulose content. This is expected because more lignin and hemicellulose are gradually removed as a result of the chemical treatment thereby increasing the yield of cellulose in the fibre.

The chemical structure of the untreated rice husk powder and the chemically treated rice husk powder are shown in Figure 1-4. The FTIR spectra of untreated rice husk powder show a characteristic carbonyl absorption peak at 1709.63cm^{-1}. This was attributed to the acetyl and uronic ester groups of the hemicellulose (Kamel, 2007). The peak at 1564.36cm^{-1} show the aromatic C=C stretch of the aromatic rings of the lignin (Donescu et al, 2008, Laka and Chemyavskaya, 2007). The FTIR spectra of the mercerized rice husk powder shows the removal of lignin resulting in the reduction of the characteristic band at 1564cm^{-1} to 1504.26cm^{-1}. The FTIR spectra of the benzoylated treatment and hydrogen peroxide treatment show the complete removal of lignin resulting in the varnishing characteristic band at 1564.36cm^{-1}. The spectra for the 10% NaOH, 10% benzoyl chloride and 10% hydrogen peroxide show that the treatment of the rice husk have changed the functional groups onto the surface of absorbent.

The results of the scanning electron microscopy analysis of the untreated and treated rice husk powder are presented in Figure 5-8. The SEM provided microstructural evidence of characteristic cellular morphologies of pore cells as the modification process proceeds. It could be seen that as chemical modification changes from benzoylated to mercerized to hydrogen peroxide, the surface of the filler gets finer and cleaner than the untreated filler. The SEM micrographs revealed that the interfacial bonding between treated elements and filler matrix was significant suggesting that further evaluation of filler on composites should be verified for possible reinforcement along the benzoylated to mercerized to hydrogen peroxide. The optical clarity in untreated rice husk powder showed no interlinks of added elements of modification. Added elements of modification altar the clarity of filler given indication of dark patches of modification.

Presence of tiny particles orifices showed indication of additional elements bonding pores and cells together. This is an indication of regular arrangement, properly aligned morphologies and structure which could have resulted from modification.

There was an indication of better interfacial interactions as modification proceeds from benzoylated to mercerized to hydrogen peroxide. Further evaluations are suggested for indicative strength improvement via reinforcement.

Obviously, large agglomerations of particles as seen in untreated rice husk powder may suggest weaker reinforcement while tiny/fine agglomerates may suggest stronger reinforcement if further probing of physical-mechanical indices are verified.

Table 3: Mechanical Properties of Vulcanizates

Properties	**Filler Loading (pphr)**				
	10	**20**	**30**	**40**	**50**
Tensile Strength (MPa)	(27.86) [24.82] {24.00} <18.10>	(29.94) [27.00] {26.60} <20.25>	(32.80) [31.62] {29.80} <23.20>	(35.64) [33.44] {31.02} <26.40>	(38.23) [35.98] {33.08} <27.50>
Modulus (MPa)	(2.56) [1.86] {1.45} <1.25>	(2.86) [2.15] {1.92} <1.40>	(3.82) [2.90] {2.35} <1.88>	(4.10) [3.18] {2.90} <2.16>	(4.25) [3.28] {3.02} <2.20>
Elongation at Break (%)	(390.00) [415.00] {476.00} <596.00>	(378.00) [402.00] {451.00} <548.00>	(346.00) [388.00] {420.00} <513.00>	(330.00) [380.00] {401.00} <510.00>	(316.00) [374.00] {396.00} <496.00>
Hardness (IRHD)	(61.00) [54.00] {52.00} <48.00>	(63.50) [60.00] {55.80} <49.00>	(68.00) [63.00] {56.00} <50.00>	(69.70) [65.00] {57.80} <51.24>	(71.20) [67.40] {59.90} <52.40>
Compression Set (%)	(14.00) [16.90] {18.00} <20.00>	(13.00) [14.00] {16.00} <19.00>	(11.94) [13.00] {14.96} <18.20>	(11.40) [11.90] {13.00} <16.00>	(10.00) [11.00] {12.40} <15.10>
Abrasion Resistance (%)	(31.55) [28.95] {27.00} <24.20>	(31.80) [30.10] {29.00} <25.00>	(36.40) [34.00] {31.60} <26.25>	(40.20) [38.00] {36.20} <29.90>	(42.50) [39.30] {37.10} <31.00>
Flex Fatigue (kc x 10^3)	(7.00) [8.06] {8.99} <9.14>	(6.10) [7.36] {8.91} <9.00>	(5.36) [6.33] {8.67} <8.10>	(5.00) [5.86] {8.06} <7.80>	(4.82) [5.26] {7.36} <6.10>

( ) = Benzoylated vulcaizate **[ ]** = Mercerized vulcanizate

{ } = Peroxide treated vulcainzate **< >** = Untreated

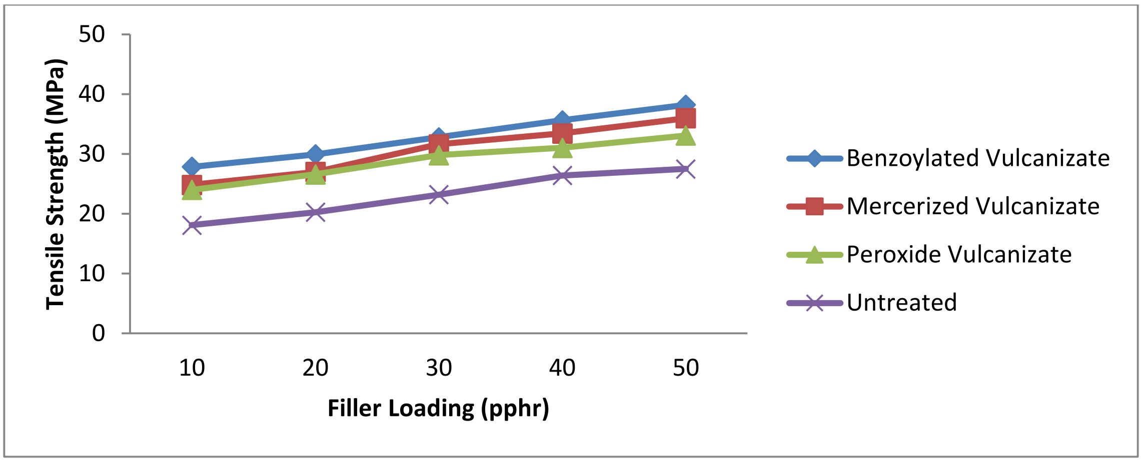

Figure 1: Effect of Treated and Untreated Rice Husk Powder on the Tensile Strength of Natural Rubber Vulcanizates

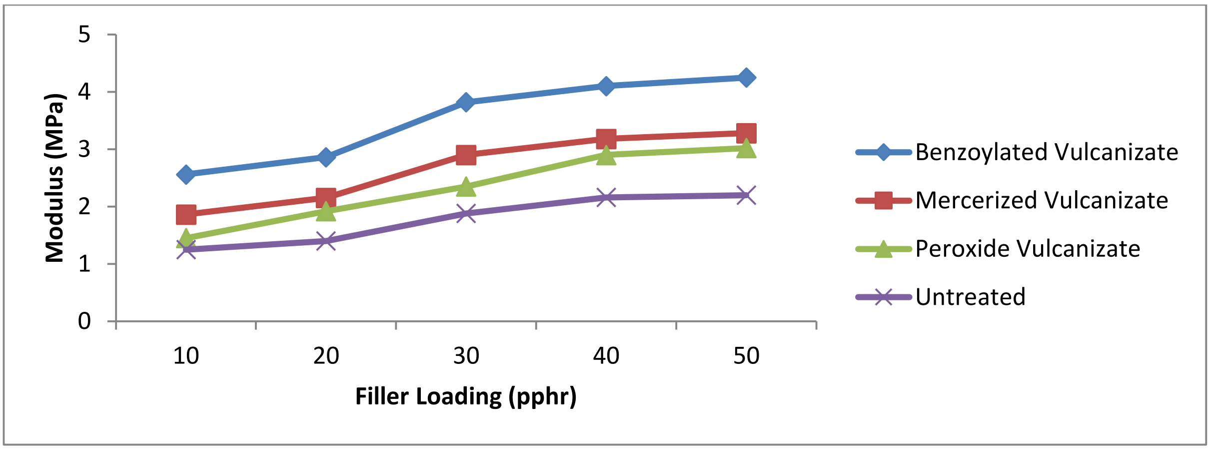

Figure 2: Effect of Treated and Untreated Rice Husk Powder on the Modulus of Natural Rubber Vulcanizates

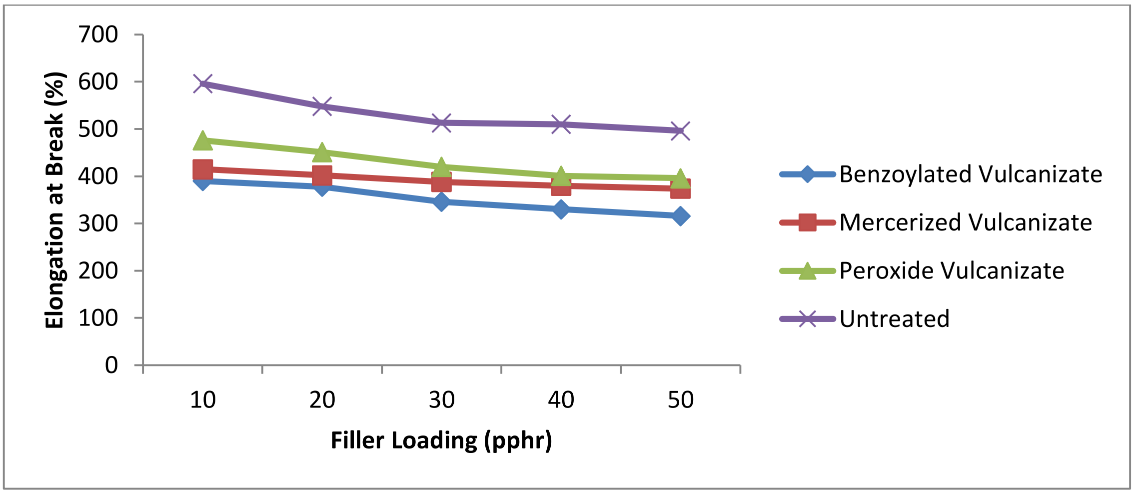

Figure 3: Effect of Treated and Untreated Rice Husk Powder on the Elongation at Break of Natural Rubber Vulcanizates

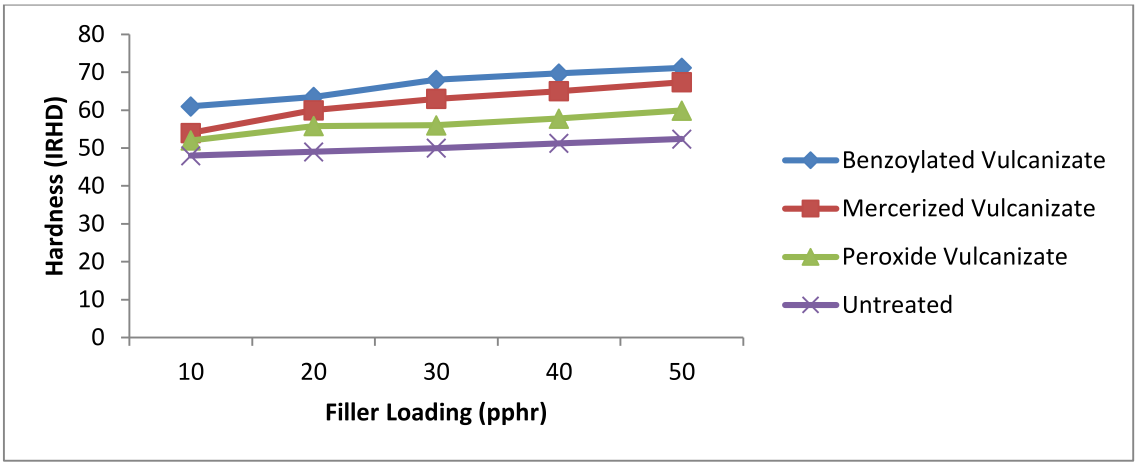

Figure 4: Effect of Treated and Untreated Rice Husk Powder on the Hardnes of Natural Rubber Vulcanizates

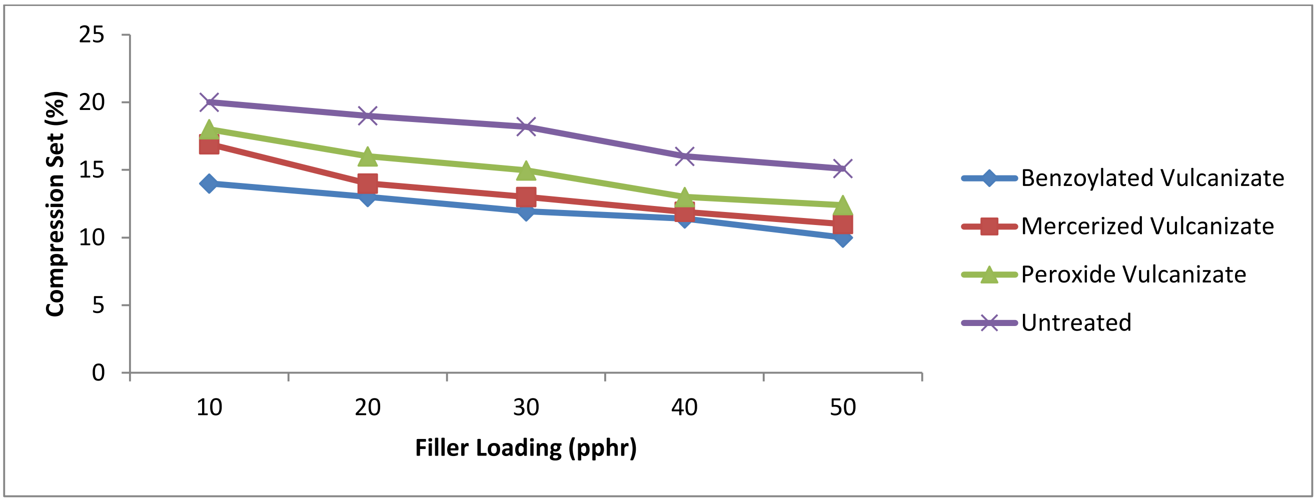

Figure 5: Effect of Treated and Untreated Rice Husk Powder on the Compression Set of Natural Rubber Vulcanizates

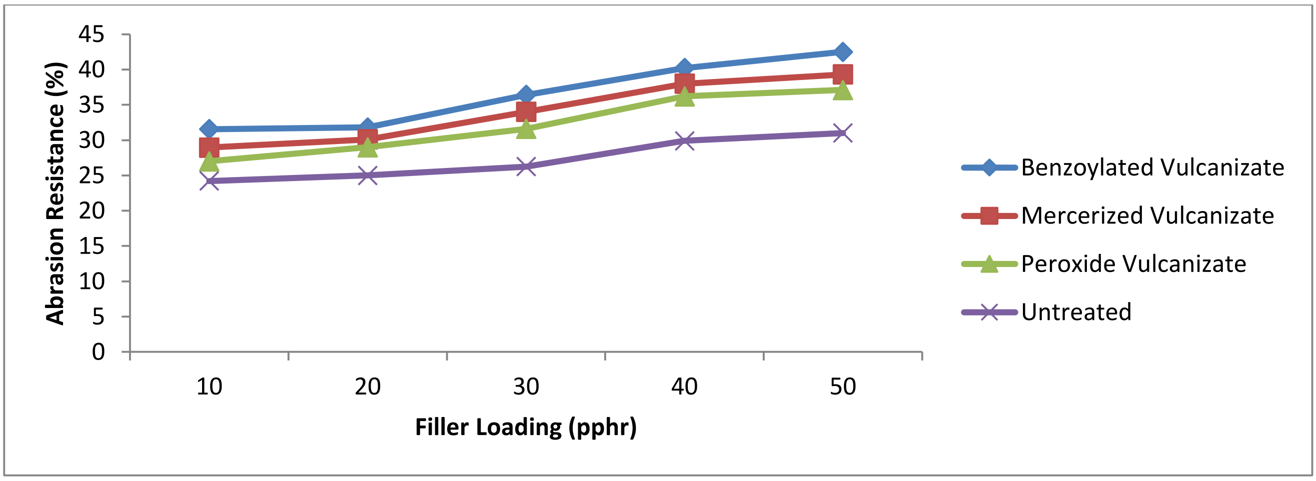

Figure 6: Effect of Treated and Untreated Rice Husk Powder on the Abrasion Resistance of Natural Rubber Vulcanizates

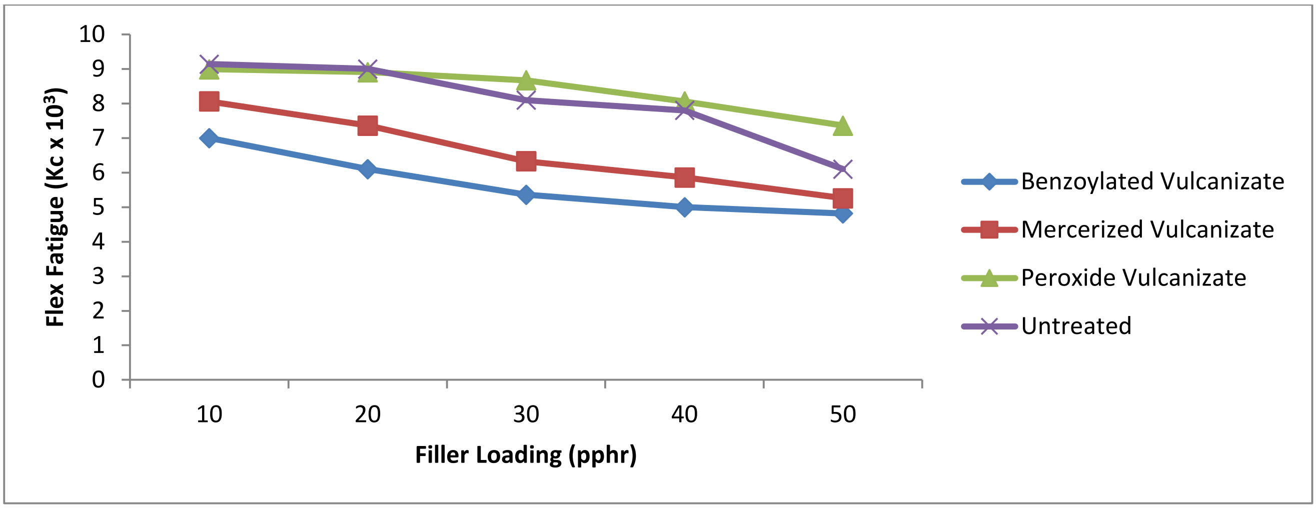

Figure 7: Effect of Treated and Untreated Rice Husk Powder on the Flex Fatigue of Natural Rubber Vulcanizates

DISCUSSION OF MECHANICAL PROPERTIES

The results of the mechanical properties are shown in Table 3 and Figure 1-7 respectively. The variation of the tensile strength of the composites filled with untreated and the treated rice husk powder is shown in Figure 1. The two factors that are apparent are that the vulcanizates containing the treated rice husk powder possessed greater tensile strength when compared to vulcanizates containing untreated rice husk powder. The vulcanizates containing treated benzoyl chloride rice husk powder show better tensile strength than the others. Sreekala, Kumaran, Joseph and Jacob (2000) reported that chemical treatment leads to fibre fibrillation that is breaking down of fibre bundles into smaller fibres which increases the effective surface area available for contact with the matrix. The increased in tensile strength can be explained on the basis of the changes in chemical interaction at the fibre-matrix interface. Chemical treatment results in improvement in the interfacial bonding by giving rise to additional sites of mechanical interlocking thereby promoting more polymer-fibre interpenetration at the surface; hence increase in the tensile strength observed (Jorts, Shay, Imam Glem, Guttmenn and Rovel, 2005).

The results of the modulus of the composites filled with treated rice husk powder were better than the untreated. It is clear that the modulus of a well bonded composite as a result of treatment arise from the fact that the load between the fibre and the matrix occur through the strong fibre-matrix interface (Onyeagoro, 2012). Hence one can see that the modulus of the chemically treated composites exhibit higher modulus values than the untreated composites.

Figure 3 shows the variation of elongation at break of the composites filled with untreated and treated rice husk powder. It can be seen that the elongation at break of the composites filled with chemically modified rice husk powder decreases compared to the composites filled with untreated rice husk powder. The decrease in elongation was due to better strength and stiffness achieved from strong adhesion between treated rice husk powder and rubber matrix, however, higher extension is obtained from weak interfacial adhesion (Onyeagoro, 2012).

The hardness results show a progressive increase with filler type. These results are expected because the treatment increases the interaction between the rubber matrix and the filler hence the increase in hardness. At higher interaction, the elasticity of the rubber chain decreases resulting in more rigid vulcanizates (Ekebafe, 2009).

The compression set in Figure 5 show a progressive decrease with composites filled with treated rice husk powder to the untreated rice husk powder. Compression set is affected by the affinity of the rubber to the filler (Nemour, 1986).

The variation of abrasion resistance with filler type and loading is presented in Figure 6. The composites filled with treated rice husk powder have better abrasion resistance than the untreated. This observation may be as a result of improved affinity between the rubber and the fillers.

The Figure 7 show the values of the flex fatigue as a function of filler type and filler loading. The values decreased with filler type and filler loading. The decrease is as a result of stiffening of the polymer chain due to the adherence of the filler to the polymer matrix.

CONCLUSION

The aim of this study is to find out the possibility of using chemically treated rice husk powder filler to produce automobile exhaust suspender. From the results obtained, chemical treatment of rice husk powder has achieved some degree of success in making a good interface between the filler and the rubber and improved mechanical properties. Chemical treatment led to strong covalent, bond formation, increased fibre-matrix adhesion and thereby enhancing the composite strength. This shows that chemically treated rice husk is a potential substitute for other fillers for automobile exhaust suspender, car foot mat and oil seal.

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