International Journal of Life Sciences Vol.2. No.4. 2013. Pp. 156-161

Full Length Research Paper

Effect of Filler loading on the Mechanical Properties of Natural Rubber filled with Guinea Corn Husk *(Holcus sorghum)* as Fillers

Tenebe O.G[1], Ayo M.D[2], Ichetaonye S. I[3], Igbonazobi L. C[2], Eguare K. O.[2]
[1]*Department of Polymer and Textile Engineering, Federal University of Technology, Owerri.*
[2]*Department of Polymer Technology, Auchi Polytechnic, Auchi.*
[3]*Department of Polymer and Textile Technology, Yaba College of Technology, Yaba, lagos*

Corresponding author: Tenebe O.G

Abstract
Effects of filler loading on the mechanical properties of natural rubber filled with guinea corn husks as fillers were studied. The fillers were sieved through a 100µm screen and were characterized in terms of loss on ignition, moisture content, iodine absorption number and pH and were used in compounding natural rubber using varying filler loading (5phr, 10phr, 20phr, 30phr and 40phr). The mechanical properties of the guinea corn filled vulcanizates present better tensile strength at 20phr (10.87Mpa), modulus at 20phr (3.85MPa), elongation at break at 20phr (400%), hardness increases with filler loading (44.50 - 81.60 IRHD), flex fatigue decreases (6.60 - 3.35 kcx10^3) as well as the compression set (19.20 - 10.20 %). Filler loading between 20 - 25phr was found to be most appropriate to achieve optimum reinforcement beyond which the filler only serve as diluents. This implies that guinea corn husks can serve as replacement for N330 carbon black in the production of low strength products such as shoe soles and foot mats.

Key words: *Guinea Corn, Husks, Screen, Vulcanizate, Characterization, Diluents, Fillers, Sieve.*

Introduction

The search for means and methods of improving the properties and processing of rubber products dates back to over a century ago [Ahmedna et al, 1997]. One way of achieving these extensions of service life of these rubber products is the incorporation of additives into the polymer matrix [Komolafe, 1980]. The use of agricultural by-products as fillers for renewable polymer additives tried by several authors is drastically taken it position in the polymer industry [Asore et al, 2010]. Fillers, as one of the major additives used in natural rubber compounds are either used raw or modified [Asore et al, 2010]. These fillers function to modify the physical and to some extent, the chemical properties of the vulcanizates [Okieimen et al, 2002]. In this research work, the guinea corn husks were characterized and then used for compounding natural rubber [Ayo et al, 2010].

Objective

To investigate the effect of filler loading on the mechanical properties of the nature rubber vulcanizates..

Experimental

Materials and Method

Nigeria standard rubber of grade NSR-10 was obtained from Rubber Research Institute, Iyanomoh, Benin City. The guinea corn husks were obtained from milling house, Agenebode, Edo State while Zinc oxide, processing oil, Tetramethyl thiuram disulphide (TMTD), Mercarpto benzothiazole sulphenamide (MBTS), Stearic acid, Sulphur, Trimethylquinoline (TMQ), Sodium sulphate (99.5%), Analytical grade potassium iodide and N330 carbon black produced by British Drug House (BDH) were obtained from Rovet Chemicals, Benin City.

Characterization of the filler

The guinea corn husks were washed in water and dried in air to removed sand particles and moisture respectively. After drying, the guinea husks were milled to fine powder and sieved through a mesh size of 100µm. The fine particles that passed through were collected, characterized in terms of Iodine value, Moisture content, Loss on ignition, Bulk density and pH respectively [Eichhom et al, 2001] and used for compounding nature rubber.

Equipments

The equipments used in this research work are of international standard obtained from H. W. Wallace and Company Limited, made in England. They include:

i. Monsanto Tensile tester model 1/m was used to test the tensile properties [ASTMD 813, 1983].
ii. Wallace Hardness tester model c8007/25 was used for hardness test [ASTMD 785, 1983].
iii. Wallace Akron abrasion tester was used for abrasion test [Sukru et al, 2008].
iv. Du Pont machine was used for flex fatigue test [ASTMD 430, 1983].

v. 2- Roll Mill was used in mixing the rubber composite [ASTMD 3184- 80, 1983].
vi. Hydraulic press was used for curing the rubber composites [Chukwu, 2002].

Processing of the composites

The formulation used for the compounding of the natural rubber (NSR 10) with the guinea corn husk fillers is shown in Table 1.

Table 1: Formulation for Compounding Natural Rubber.

Ingredients (phr)	A	B	C	D	E	F
Natural Rubber	100	100	100	100	100	100
Filler (GCH)	-	5	10	20	30	40
Zinc oxide	5.0	5.0	5.0	5.0	5.0	5.0
Stearic acid	2.5	2.5	2.5	2.5	2.5	2.5
Sulphur	1.5	1.5	1.5	1.5	1.5	1.5
TMTD	3.5	3.5	3.5	3.5	3.5	3.5
MBTS	1.5	1.5	1.5	1.5	1.5	1.5
TMQ	1.5	1.5	1.5	1.5	1.5	1.5
Processing Oil	5.0	5.0	5.0	5.0	5.0	5.0

GCH = Guinea Corn Husks
A batch factor of six (6) was used to multiply the weight of the ingredients in parts per hundred of the rubber [Chukwu, 2002].

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^{o}c to avoid cross-linking during mixing after which the rubber composite was stretched out. Mixing follows [ASTMD 3184–80, 1983].

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 (GCH)	10
Addition of MBTS	1
Addition of TMTD	1
Processing Oil	1
Addition of Sulphur	2
Total	22

Compounded composite curing

The curing of test pieces was done in a compression moulding machine. The curing was carried out at 130^{o}C for 25 minutes [Ekebafe et al, 2010].

Mechanical properties of the vulcanizates

The mechanical properties carried out include; tensile properties,
Hardness test, Compression set, Flex fatigue and Abrasion resistance respectively

Results

The results of the characterization of the fillers and mechanical properties of the guinea corn husks filled vulcanizates are presented in table 3 and tale 4 respectively.

Table 3: Characteristics of the powdered guinea corn husks

Parameter	**Value**
Loss on ignition (%)	23.50
Moisture content (%)	22.30
pH of slurry	5.10
Iodine adsorption number (g/mg)	30.01
Particle size distribution (µm)	100

Table 4: Mechanical properties of the guinea corn husks filled vulcanizates

Property	**SampleA (0phr)**	**SampleB (5phr)**	**SampleC (10phr)**	**SampleD (20phr)**	**SampleE (30phr)**	**SampleF (40phr)**
Tensile strength (MPa)	2.08	2.95	6.20	10.87	9.40	8.10
Modulus (MPa)	1.92	2.00	2.40	3.85	2.50	1.70
Elongation at break (%)	98	100	200	400	100	50
Hardness (IRHD)	41.18	44.50	52.20	60.00	73.30	81.60
Flex fatigue ($kcx10^3$)	5.86	6.60	6.00	5.32	4.25	3.35
Compression set (%)	19.01	19.20	16.75	13.04	11.50	10.20
Abrasion resistance (%)	11.91	12.50	25.00	45.55	56.45	60.50

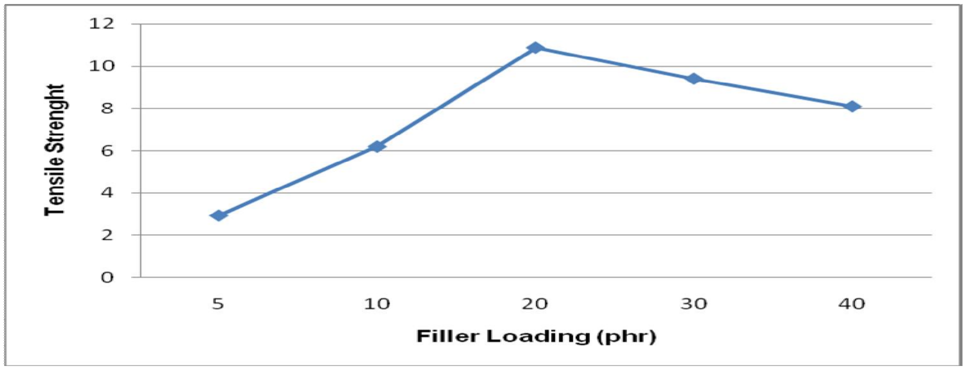

Figure 1: Tensile Strength Vs filler loading of GCH-filled Vulcanizate

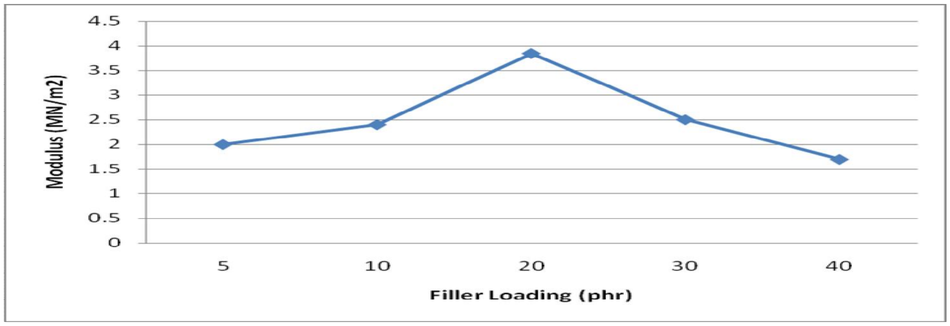

Figure 2: Modulus Vs filler loading of GCH-filled Vulcanizate

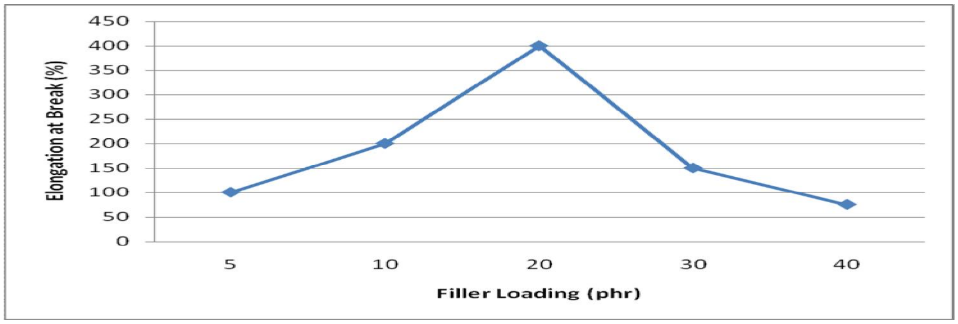

Figure 3: Elongation at break Vs filler loading of GCH-filled Vulcanizate

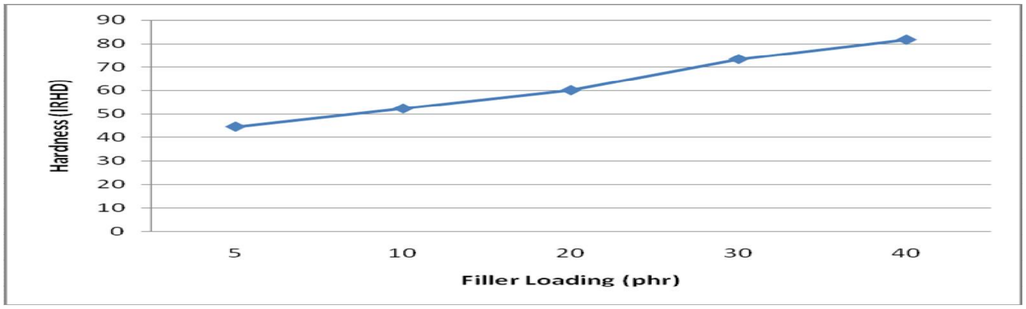

Figure 4: Hardness Vs filler loading of GCH-filled Vulcanizate

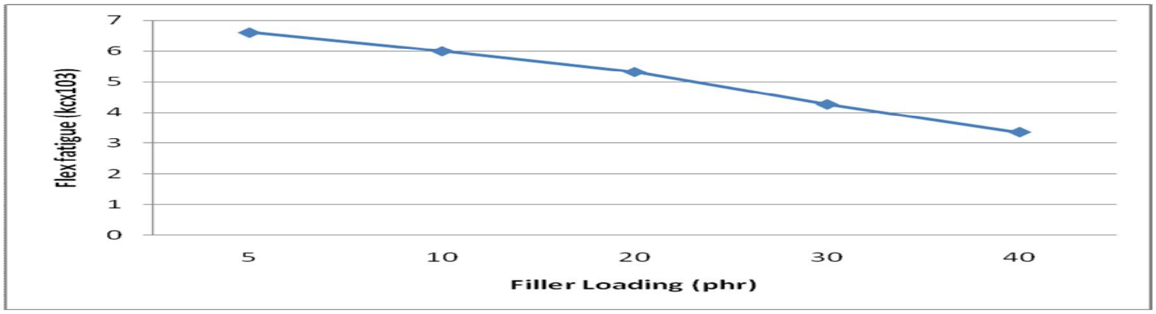

Figure 5: Flex fatigue Vs filler loading of GCH-filled Vulcanizate

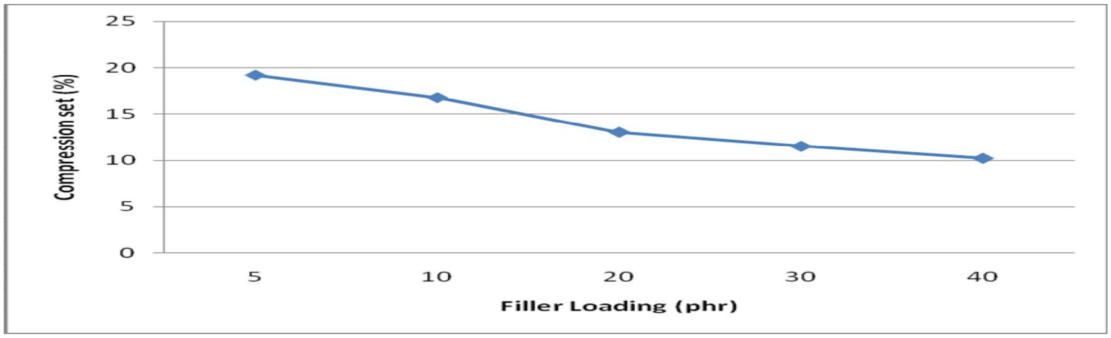

Figure 6: Compression set Vs filler loading of GCH-filled Vulcanizate

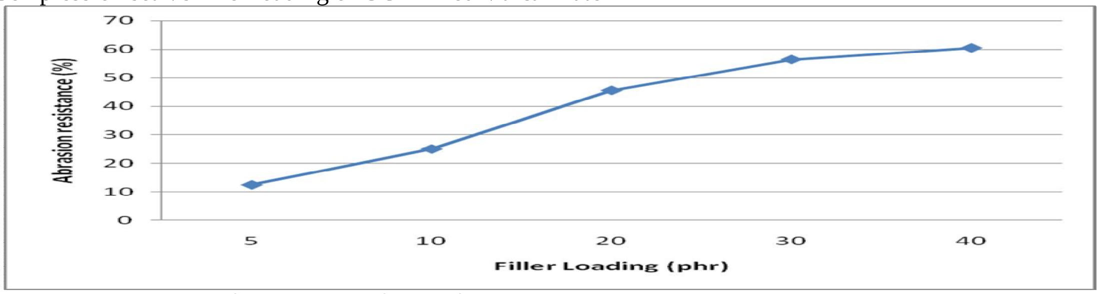

Figure 7: Abrasion resistance Vs filler loading of GCH-filled Vulcanizate

Discussion

Natural fibres are derived from lignocelluloses containing strongly polarized hydroxyl group, hence hydrophilic in nature. Most fibres contain cellulose, lignin, water-soluble compounds, waxes, hemicelluloses etc., where lignin, hemicelluloses and celluloses are the major constituents [Ekebafe et al, 2011]. The hydrophilic nature of fibres causes the fibre to swell considerably and ultimately rotten through fungi attack. The major causes of this biodegradation are the presence of hemicelluloses in natural fibres, whereas lignin is prone to Ultra-violet degradation but thermally stable [Ekebafe et al, 2011].

In all filled system as shown in Figures 1, 2 and 3, a gradual increase in tensile strength, modulus as well as elongation at break with filler loading is noticed up to 20phr before it starts decreasing as the filler content increases [Imanah et al, 2004]. It may be mentioned here that both tensile strength and modulus are important for recommending any vulcanizate for structural applications.

The increase in tensile strength, modulus and elongation at break is as a result of high surface area of guinea corn husk, suggest better polymer filler interaction and hence enhanced better tensile properties. The factors that affect the reinforcing potentials of fillers include filler dispersions, surface area, surface reactivity, bonding capacity (quality), particle size between the filled and the elastomeric matrix [Imanah et al, 2003].

Table 4 and Figure 2 show that modulus of filled vulcanizates depends on the level of filler dispersion in the polymer matrix. The reduction in particle size accounts for the moderate moduli and tensile strength since high particle size reduces modulus and tensile strength because it impedes polymer-filler interaction which consequently decreases the ability of the filler to restrain gross deformation of the rubber matrix. At filler loading beyond 20phr there may not be rubber to hold the filler particle together leading to a phenomenon refer to as phase inversion [Ishak et al, 1995].

Table 4 and Figure 3 shows that elongation at break (EAB) decrease with increasing cross-links of the mixes for all fillers below 20phr, above which there was a gradual rise in the value of EAB. This has been attributed to the spacing between cross-links which put more of the stress on a relatively few of the network chains. These highly stressed chains break first and their loads are then distributed to other chains forcing them to either break or slip so as to relieve the stress on them. A decrease in EAB has been explained in terms of adherence of the filler to the polymer phase leading to the stiffening of the polymer chain and hence resistance to stretch when the strain is applied [Imanah et al, 2003].

The hardness of guinea corn husks filled vulcanizates shown in Table 4 and Figure 4 increased with increasing filler loading. Since hardness measures small deformation at the surface of the vulcanized elastomer and an approximate index of stiffness, longer shelf service life may be expected of products made with guinea corn husks as fillers. 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 [Nemour, 1986].

Flex fatigue of a material is the failure or degradation of its properties due to oscillatory deformation or stresses, representing the number of cycles of oscillation before a specimen fractures at an applied stress or strain. At high stress the specimen breaks after a few cycles and vise-a-vise. The values of flex fatigue decreases with increasing filler loading of the mixes presented in Table 4 shown in Figure 5. A decrease in flex fatigue has been explained in terms of adherence of the filler to the polymer phase leading to the stiffening of the polymer chain and hence resistance to stretch when strain is applied. As filler loading increases, it could be expected that more filler particles and aggregates will not be dispersed and wetted efficiently by the rubber matrix [Sukru et al, 2008].

When an elastomer is compressed and allowed to relax, it does not return to its original dimension. This divergence is refers to as compression set. The results of compression set in Table 4 and Figure 6 show that as filler loading increases, the compression set of guinea corn husk filled vulcanizates decreases. This observation is connected with the degree of filler dispersion and its particle size which may have enhanced it performance [Sukru et al, 2008]. This is expected because as filler concentration increases in the polymer matrix, the void spaces are proportionately reduced [Zareiropoulosa et al, 2002].

The abrasion resistance of a solid body is defined as its ability to withstand the progressive removal of the material from its surface as a result of the mechanical action of rubbing, scraping or erosive nature. The trend of abrasion resistance with filler loading presented in Table 4 and Figure 7 show a regular pattern of increase with increasing the filler loading of the guinea corn husk filled vulcanizates. This indicates that filler loading is a function of the measured parameter attributed to the degree of dispersion of the fillers [Asore, 2000].

Conclusion
The main aim of this research work is to examine how the filler loading may influence the mechanical properties of natural rubber vulcanizates filled with guinea corn husks as fillers [Zareiropoulosa et al, 2002]. The initial result shows that guinea corn husks can serve as reinforcing filler for natural rubber compound. This reinforcing potential of guinea corn husks is best compared to that of N330 carbon black in some measured properties. The results indicate that mechanical properties of the vulcanizates are greatly influenced by filler loading and are therefore significant factors in determining the application in rubber compounding [Nemour, 1986]. The result also predicts the potential applications of guinea corn husks as low cost filler in natural rubber products. The vulcanizates exhibit high quality characteristics. This indicates that for high quality vulcanizates using guinea corn husks as fillers, filler loading should not exceed 20 - 25phr.

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