Investigation of the Effect of Coir Fibre Particulate Fillers on Physical Properties of Natural Rubber Vulcanizates

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***Abstract*—The focus of this work was to investigate the effect of coir fibre particulate filler on the physical properties of rubber vulcanizate. Coconut fibre was separated from the outer shell of coconut fruit shell, it was then converted to particulate coir fibre (PCF) filler by grinding to 125μm diameter particle size. The particulate coir fibre was used as a filler in compounding natural rubber, varying the loadings between 0 and 60 phr at the interval of 15 phr. The convectional filler carbon black (CB) was also used within the same filler loading range for the purpose of comparison. The fillers were characterized on the basis of pH, density, and moisture content. The physical properties of the vulcanizates were investigated. Results of tests obtained for vulcanizates filled with PCF were compared with those obtained from carbon black filled vulcanizates. It was observed that for hardness, PCF based vulcanizates compared very well with the CB filled vulcanizates. The hardness for all vulcanizates maintained an increasing trend with the increase in the filler loadings. Abrasion resistance and compression set revealed an irregular increase with increasing filler loading for vulcanizates filled with both fillers while elongation at break showed a falling trend as filler loading were increased. Tensile strength increase with increasing filler loading for both filler type based vulcanizates, however vulcanizates filled with PCF fell below that of carbon black at every point of filler loadings. The scanning electron micrograph obtained for vulcanizate filled with PCF showed high degree of filler dispersion in the rubber matrix. The thermogravimetric analysis (TGA) also showed that the vulcanizates are thermally stable up to 250 ^{o}C.**

Keywords—Coir fibre, rubber, vulcanizate, particulate filler, carbon black

INTRODUCTION

The search for possible alternative to carbon black filler in the manufacture and application of rubber article has witnessed a steady growth over the years. Carbon black filler is produced through heavy industrial combustion of hydrocarbon process. This process requires tremendous energy ultilisation and constitutes a potential source of pollution and global warming; this has led to the search for more cost effective and eco – friendly materials for replacement. Raw rubber is usually transformed into a range of materials suitable for application in various uses and in different service environment through compounding and vulcanization. Natural rubber alone does not possess the necessary physico-mechanical properties that are required by rubber manufacturers. Fillers are widely used additives and the largest in quantity among others in the manufacture of rubber product. Particulate fillers such as carbon black, calcium carbonate and china clay are widely used as reinforcing filler in the industries. Calcium carbonate ($CaCO_3$) has attracted considerable interest in recent years due to its low cost and availability. Agricultural residues are low cost materials and readily available in large quantity for use everywhere [1]. In previous report, the use of sugarcane bagasse, cocoa pod husk, rubber seed shell etc. as filler in natural rubber has been investigated [2], [1] and [3]. As for coir fibre few works have been reported so far on its use as fillers and all of them employed carbonized coir [4], [5]. These study present results of assessment and utilization of particulate coir fibre as filler for the compounding of rubber.

MATERIALS AND METHODS

The main materials used in this work include the crumb natural rubber of grade NSR-10 purchased from Alhson Lab Equipment consult, Zaria Kaduna State, Nigeria and the coir fibre obtained from Ekpoma, Edo state Nigeria. Other compounding additives and chemical used which are of industrial grade were sulpur, stearic acid, carbon black (N330 HAF), trimethyl quinoline (TMQ) and mercaptobenzothiazole (MBT) and were all purchased from Alhson Lab Equipment consult, Zaria Kaduna State, Nigeria. The coconut shell fibre was obtained from a waste point in Zaria Nigeria.

Preparation and Characterization of Particulate Coir Fibre (PCF) Fillers

The waste outer part of coconut fruit shell were soaked in water for five (5) hours in order to soften it and to facilitate easy extraction of the coir fibre. The fibre so obtained was then sun dried. The dried coir fibre were then converted to particulate coir fibre filler by grinding and sheaving it to 125µm particle size with the aid of laboratory grinder (Thomas Wiley lab mill model 4). The particulate coir fibre (PCF) filler were characterized based on pH value, density and moisture content

Formulation of Rubber Composite

Ten (10) formulations of rubber composite were prepared. Five of them were loaded with 0, 15, 30, 45, and 60 parts per hundred (pph) of carbon black as fillers and the other five loaded with 0, 15, 30, 45 and 60 pph of particulate coir fibre (PCF). All the formulations were based on the conventional sulphur vulcanization system (high sulphur to low organo-accelerator level). The formulation tables are shown below.

Table 1: Rubber Formulation with Carbon Black Filler

INGREDENT	CONTENT (pph)
Natural rubber	100
Zinc oxide	5
Stearic acid	2
TMO	1
MBT	2
Sulphur	3
Carbon black	0, 15, 30, 45, 60

Table 2: Rubber Formulation with Particulate Coir Fibre Filler

INGREDENT	CONTENT (pph)
Natural rubber	100
Zinc oxide	5
Stearic oxide	2
TMO	1
MBT	2
Sulphur	3
Particulate fibre	0, 15, 30, 45, 60

Compounding

The rubber was masticated and mixed with chemicals/additives using open roll mill manufactured by Reliable Rubber and Plastics New York, Model 5189 in accordance with standard methods in the American Society for Testing Materials (ASTM D3184-80) for all the composites.

Vulcanization

Appropriate test pieces were vulcanized using 11 metric tons Carver inc. model 3851-0 lab curing press at temperature of 150 °C for 30 minutes.

Determination of the Properties of Rubber Vulcanizates

Tests piece were prepared from the vulcanizates into appropriate shapes and dimensions for various tests and analysis which includes hardness, abrasion resistance, compression set, elongation at break, tensile strength, scanning electron microscopy (SEM) and thermogravimetry analysis (TGA). All tests were carried out as specified by American Society for Testing Materials (ASTM).

RESULTS AND DISCUSSIONS

Results

Table 3: Characteristics of Fillers

PARAMETERS	PARTICULATE COIR FIBRE (PCF)	CARBON BLACK (CB)
Moisture content	7.41	2.4
pH	7.18	6.5
Density (gcm^{-3})	0.36	0.92
Particle size (µm)	125	

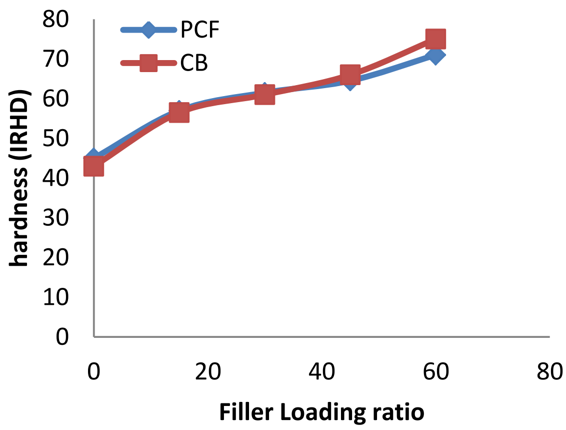

Fig. 1: Effect of Filler Loading on Hardness

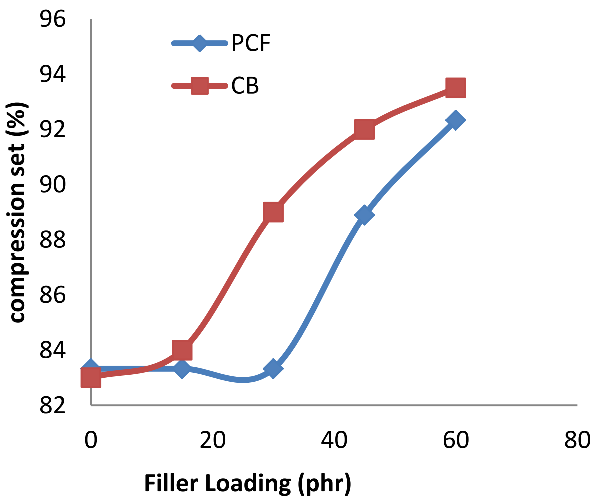

Fig 2: Effect of Filler Loading on Compression Set

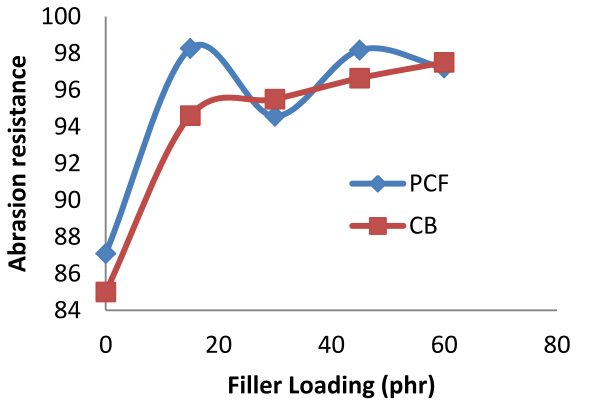

Fig.3: Effect of Filler Loading on Abrasion Resistance

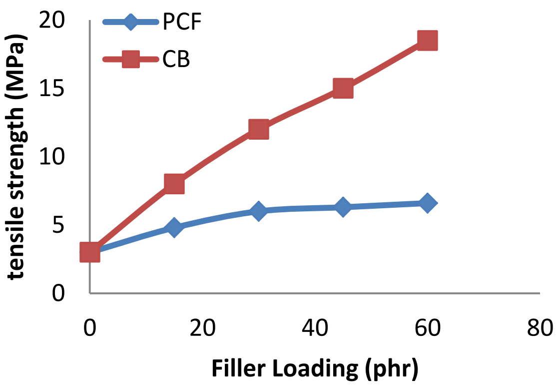

Fig.4: Effect of Filler Loading on Tensile Strength

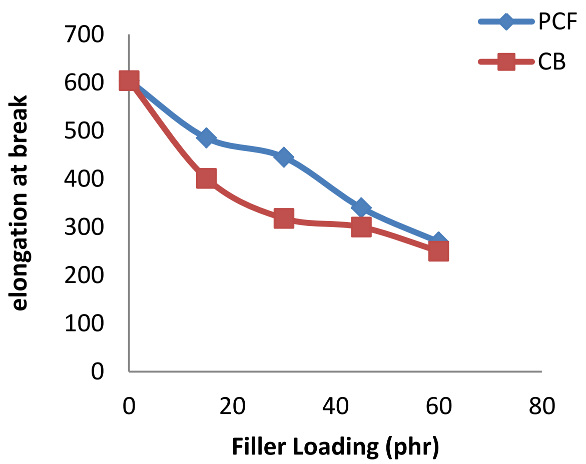

Fig. 5: Effect of Filler Loading on Elongation at Break

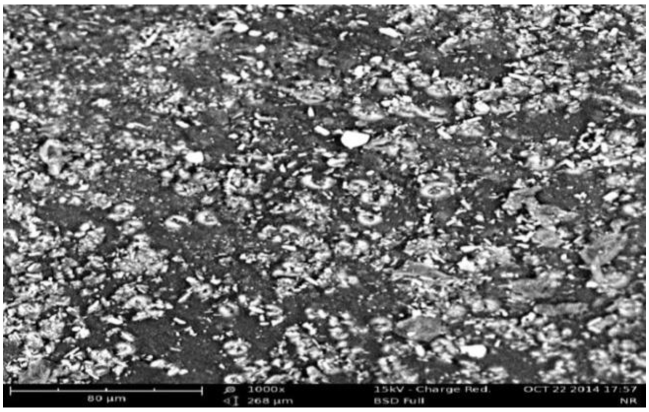

Plate I: Micrograph of Unfilled NR

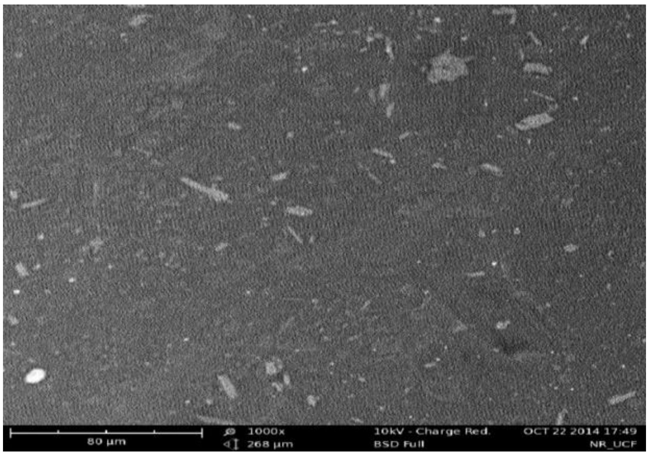

Plate II: Micrograph of PCF-NR

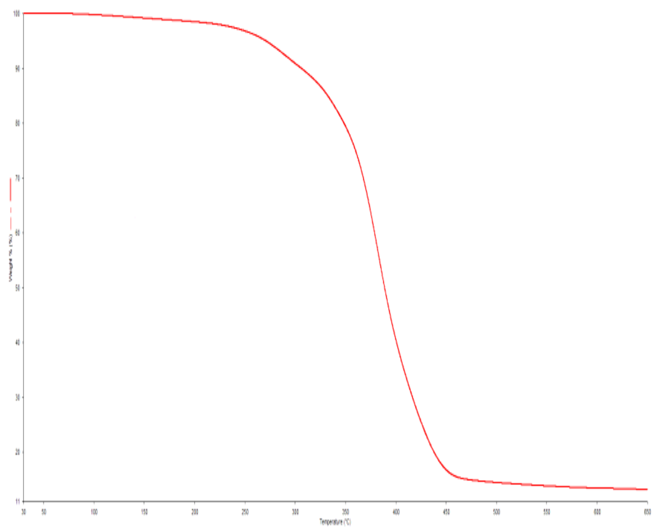

Fig.6: Thermogravimetric Analysis (TGA)

Discussion

The physical characteristics of particulate coir fibre (PCF) and that of carbon black are shown in table 3. The pH value of PCF is 7.18 while that of CB was 6.5, moisture content and density values were 7.41 % and 0.36 gcm^{-3} for PCF and 2.4 % and 0.92 gcm^{-3} for CB. The pH and moisture content affects cure rate [4]. Generally alkalinity accelerates cure rate while acidity retards it [6]. Also faster cure rates are usually favored by high moisture content [7]. The density of the PCF is lower than that of the CB. The density and particle size determines the level of distribution and dispersion of fillers in the rubber matrix; this essentially affects the reinforcing properties of the final vulcanizates. The smaller the particle size and the lower the density, the higher the reinforcing potential of filler.

The hardness for both PCF and CB filled vulcanizates were measured [8] and found to increase with increasing filler loading (fig.1). The reason for this is that as more filler is incorporated into the rubber matrix the elasticity of rubber chain is reduced, resulting in more rigid vulcanizates. Vulcanizates filled with PCF showed more improvement in the degree of hardness between 0 to 45 phr of filler loading while that of carbon black was higher from 45 to 60 phr. This trend of increasing hardness with increase in filler loading is expected and it is in agreement with many workers such as Egwaikhide et *al,* 2007, Egwaikhide et *al,* 2008, Aguelle and Madufor , 2012.

In the case of compression set which was determined [9] and presented (fig.2), there was no traceable increase in compression set as filler loading was increased from 0 to 15 phr and 0 to 30 phr for vulcanizate filled with carbon black and that filled with PCF respectively. Beyond these points the compression set drastically increased for both filler filled vulcanizates. However the rate of increased is higher for the carbon black filled one. The percentage compression set shown in the curves represents the percentage thickness retained after the removal of the deforming stress. This is dependent on the chain elasticity of the rubber [4]. As the filler loading is increased it becomes more rigid and less susceptible to compressive loading. This is in agreement with the findings of [5].

The abrasion resistance result as shown in fig. 3 reveals that at lower loadings between 0 and 17 there is a noticeable rise in the abrasion resistance which is the same for both filler- filled vulcanizate. At higher loading however ther is no tangible increase in abrasion resistances of both vulcanizates. This is an indication that abrasion resistance is only slightly affected by filler loading. Other workers such as [3], [5] also reported similar trend and they attributed it to be more dependent on the degree of filler dispersion and the adhesion between the polymer phase and the filler particles.

The tensile strength (fig.4) of both PCF and CB filled vulcanizate was measured [10] and found to increase with increasing filler loading from 3 MPa until a maximum level is reached at 6.3 MPa and 18.5 MPa for PCF and CB respectively for the loading employed in this work. The influence of filler loading on the tensile strength of rubber vulcanizates is determined by the extent of filler dispersion, adhesion between polymer phase and filler and the filler surface reactivity.

The elongation at break of both PCF and CB as shown in fig. 5 shows a corresponding decrease as filler loading is increased, however the values of elongation at break of CB was found to be lower at every point of loading. This phenomena can be explained in terms of the adherence of the polymer phase leading to the stiffening of the polymer chain and hence resistance to stretch when strain is applied.

Figures 6 and 7 showed the scanning electron micrographs of filler filled and unfilled vulcanizates. The micrograms showed showed that the unfilled vulcanizates had large numbers of pores and contours while the micrograph of vulcanizates revealed micrograph devoid of pores and contours. This is an indication of high degree of distribution and dispersion of filler phase in polymer matrix.

The thermogravimetric analysis (TGA) of the vulcanizate filled with PCF showed that the thermal decomposition began near 250 ^{o}C and was complete at 370 ^{o}C, this is similar to the trend observed by South, 2001. In his study he found out that vulcanizate filled with carbon black began thermal decomposition near 200 ^{o}C and complete near 400 ^{o}C. This is an indication that PCF filled vulcanizates can be applied in the same thermal environment similar to that filled with carbon black

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