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Cure Characteristics and Physico-Mechanical Properties of Carbonized Bamboo Fibre Filled Natural Rubber Vulcanizates

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ABSTRACT: *Bamboo fibres were carbonized at 600^0C and used as particulate filler in natural rubber vulcanizates. Carbon black was used as the reference filler, while maleic anhydride-grafted-polyisoprene was used as a compatibilizer. The natural rubber vulcanizates were compounded on a two-roll mill, and test samples were prepared by compression moulding. The cure characteristics and physico-mechanical properties of the natural rubber vulcanizates were studied at filler loadings of 0, 2, 4, 6, 10, and 15 phr (parts per hundred parts rubber). The carbonized bamboo fibre was characterized for filler properties and sieved to 0.08µm and 0.35µm particle sizes. Results obtained showed that compatibilized carbonized bamboo fibre filled vulcanizates exhibited improvement in the cure properties investigated over the non-compatibilized vulcanizates. Carbon black exhibited higher tensile strength, modulus, hardness and elongation at break in the vulcanizates than carbonized bamboo fibre. However, at any given loading of carbonized bamboo fibre, these properties increased with a reduction in particle size of the filler. Specific gravity of the vulcanizates increased with increases in filler loading, and was found to be dependent on filler particle size. The smaller particle-sized carbonized bamboo fibre filled vulcanizates exhibited higher specific gravity than those vulcanizates containing the larger particle-sized filler.*

Keywords: *bamboo fibre, carbonization, cure characteristics, natural rubber, physico-mechanical properties.*

I. Introduction

Natural rubber (NR) is an interesting material with commercial success due to its excellent physical properties, especially high mechanical strength, low heat build-up, excellent flexibility, and resistance to impact and tear, and above all its renewability [1]. However, raw dry rubber is seldom used in its original state for any engineering and domestic application. Consequently, rubber manufacture involves the addition to rubber many ancilliary materials called additives to allow the rubber compounds to be satisfactorily processed and vulcanized in order to improve the application properties of the rubber compound. Additives used in rubber manufacture include vulcanizing agents, accelerators, activators and/or retarders, fillers, anti-degradants, among others.

Fillers represent one of the most important additives used in rubber compounding. Fillers are added to rubber formulation in order to optimize properties needed for service application [2]. Reinforcement of rubber polymers with particulate fillers is a subject that has captured the interest of a large number of researchers [3, 4, 5, 6, 7]. Property advantages obtainable from filler reinforced rubber vulcanizates include design flexibility, improved physico-mechanical properties such as tensile properties, hardness, and processing economy. Due to strong environmental regulations worldwide and increased interest in the proper utilization of renewable natural resources, efforts have been made to find alternative reinforcements that are environmentally friendly while providing the same performance as their synthetic counterparts [8].

With their low cost, easy availability, ease of chemical and mechanical modification, and high specific mechanical properties, natural fibres represent a good, renewable and biodegradable alternative to the most common synthetic reinforcement [9].

Carbon black is always considered the most commonly consumed reinforcing filler in the rubber industry [10]. Considering its problems such as its non-renewable petroleum origin, dark color, contamination and pollution, researchers are seeking an adequate alternative [11],[12]. Thus, the use of nano-fillers such as nano-ZnO, nano-Al_2O_3, and nano-$CaCO_3$ as substitutes for carbon black in rubber compounding has been suggested [13]. The authors reported that the use of these fillers exhibited superior physical and mechanical properties in the vulcanizates when compared to the conventional micro-composites. Excellent reports exist in the literature on the use of different fillers to reinforce natural/synthetic rubber and their blends. Osman et al. [14] studied the effect of maleic anhydride-grafted polypropylene (MAPP) on the properties of recycled newspaper (RNP) filled polypropylene (PP)/natural rubber (NR) composites. The authors found that the incorporation of MAPP reduced the water uptakes of the composites. In a study by Ansarifer et al. [15] on the properties of natural rubber reinforced with synthetic precipitated amorphous white silica nano-filler it was reported that compression set, tensile strength and harness were improved on addition of filler into the rubber, while elongation at break, tear strength and cyclic fatigue were adversely affected. Yang et al. [16] in their studies on the influence of graphite particle size, and shape on the properties of acrylonitrile butadiene rubber (NBR) found that graphite with the smallest particle size possessed the best reinforcing ability, while the largest graphite particles exhibited the lowest function coefficient of the composites among four fillers investigated.

The reinforcing effects of coal shale-based fillers on natural rubber on the basis of filler particle size have been investigated [17]. The authors reported that the ultra micro coal-shale powder exhibited excellent filler properties. In other reports, the use of renewable agro-based materials such as cocoa pod husk and rubber seed shells [18], palm kernel husk [19], short pineapple leaf fiber [20], and snail shell powder [4] as fillers in natural rubber were examined.

The present study reports the use of carbonized bamboo fibre as filler in natural rubber. One of the most important aspects of composite manufacture is to achieve adequate adhesion between filler and the rubber matrix. In this work, the cure characteristics and physico-mechanical properties of carbonized bamboo fibre filled natural rubber vulcanizates using maleic anhydride-graft-polyisoprene (MAPI) as compatibilizer are reported. Bamboo fibre was selected as the reinforcement because bamboo is an abundant natural resource in Nigeria and its overall mechanical properties are comparable to those of wood [21]. The use of carbonized bamboo fibre in reinforcing natural/synthetic rubber had not been reported in the scientific literature to my knowledge. However, the effect of filler carbonization temperature on the tensile properties of natural rubber compounds filled with cassava (Manihot esculenta) peel carbon filler was reported by Stella et al. [22] who revealed that the physico-mechanical properties of the composites were greatly influenced by filler loading and filler carbonization temperature. Similarly, the influence of carbonized Dika (Irvialgia Gabonensis) nutshell powder on the vulcanizate properties of natural rubber/acrylonitrile-butadiene rubber blends was studied by Onyeagoro [23]. The author found that synchronous use of carbon black and carbonized Dika nutshell produced significant improvements in the vulcanizate properties of the blends at 10 phr filler loading, and suggested that carbonized Dika nutshell powder could serve as potential substitute filler for carbon black in the rubber industry, especially in the production of low-cost/high volume rubber products where strength is not critical.

II. Experimental

2.1. Materials

Natural rubber (Standard African Rubber, SAR 3) having the properties given in Table 1 was obtained from the Rubber Research Institute of Nigeria (RRIN), Iyanomo, Benin City. The rubber compounding ingredients such as carbon black (N330), sulphur, accelerator (MBT), zinc oxide, stearic acid, wax and anti-oxidant (TMQ) were of commercial grade and supplied by Dunlop Plc, Lagos, Nigeria. The bamboo used in this work belongs to the species of Bambusa Paravariabili*s* and was obtained from Forest Reserve Areas in Umuokanne in Imo State, Nigeria. Maleic anhydride-graft-Polyisoprene used as compatibilizer was purchased from Rovet Chemicals Ltd, Benin City, Nigeria.

Table 1: Properties of Standard African Rubber (SAR 3) [25]

Parameters	
Volatile matter	0.40
Dirt content retained on 45µm sieve (%)	0.02
Nitrogen (%)	0.23
Ash content (%)	0.32
Initial Plasticity (P^0)	36
Plasticity Retention Index (PRI)	67
Plasticity after aging for 30 min @ 140^0C (P^{30})	24
Mooney Viscosity, ML (1+4), 100^0C	70

2.2. Carbonization and Characterization of Bamboo Fibres

Bamboo chips were produced by means of a wood planar which were then air-dried to a constant weight. Portion of the dried bamboo chips was milled to fine powder, sieved through a mesh of 150µm, and collected as unmodified bamboo fibre in a dessicator until required. The unmodified bamboo fibre was weighed and carbonized at a temperature of 600^0C for 3 hours [26] using a Muffle furnace. The carbonized bamboo fibre (CBF) portion was then milled to fine powder and sieved through a mesh size of 0.08, and 0.35µm. It was kept in a dessicator and allowed to cool to room temperature until required. CBF and carbon black (CB) were then characterized and used for compounding.

2.3. Characterization of Carbonized Bamboo Fibre and Carbon Black (N330)

Carbonized bamboo fibre (CBF) and Carbon black (CB) were characterized in terms of loss in ignition, P^H, bulk density and surface area. The loss on ignition was determined gravimetrically according to ASTM 1509 standard test method. The P^H and bulk density were determined by methods described by Ahmedna et al [27]. Iodine adsorption number method was used to measure the surface area as described by Ahmedna et al [28]. Moisture content and oil absorption were measured according to standard procedures described by ASTM D 1510, 1983 and BS 3483, Part B7, respectively. The results obtained are as presented in Table 2.

Table 2: Characterization of Carbonized Bamboo Fibre (CBF) and Carbon black (CB)

Parameter	CBF	CB (N330)
Loss on ignition	79.3	91.3
P^H of slurry @ 32^0C	8.45	6.35
Iodine adsorption number (mg/g)	56.10	82.43
Bulk density (g/ml)	0.47	-
Moisture content (%)	5.13	2.72
Oil absorption (kg)	5.83 (0.35µm) 6.25 (0.08µm)	8.76

2.4. Preparation of Natural Rubber Vulcanizates

Table 3 gives the recipe used in the formulation of natural rubber compound. Mixing was carried out on a laboratory two-roll mill size (160 x 320 mm) in accordance with ASTM–D 3184-80. The nip gap, mill roll speed ratio, sequence of addition and time of mixing of the ingredients were kept the same for all the composite samples. The sheeted rubber compound was conditioned at room temperature, 32^0C for 24 hours in a closed container and was further processed by curing. Curing was achieved by a method described by Igwe and Ejim [4]. The cured samples were coded accordingly for property testing.

Table 3: Compounding recipe for Filler reinforced Natural Rubber Composites

Ingredients (Phr)	Formulation			
	A	B	C	D
Natural Rubber (NR)	100	100	100	100
Stearic acid	2	2	2	2
Zinc Oxide	5	5	5	5
TMQ*	1	1	1	1
MBT+	1	1	1	1
Sulphur	2	2	2	2
Processing aid	2	2	2	2
Maleic anhydride-graft-Polyisoprene (Compatibilizer)	3.5	3.5	3.5	3.5
Filler# Variable	(2, 4, 6, 10, 15)			

* TMQ = Trimethyl Quinoline
\+ MBT = Mercaptobenzoylthiazole
\# Filler = Bamboo fibres (CBF) and Carbon black (CB)

2.5. Cure Characteristics of Natural Rubber Vulcanizates

The cure characteristics were measured using a Monsanto Moving Die Rheometer (MDR Model). The scorch time, t_2 and cure time, t_{90} were obtained from the rheometer at 150^0C. The Mooney viscosity was also determined at 120^0C using a Monsanto automatic Mooney Viscometer (MV 2000 Model). The testing procedure was done according to the method described in ASTM D 1646 – 94.

2.6. Testing of Natural Rubber Vulcanizates

The following tests were conducted on the rubber samples using standard test methods [14, 16, 17, 22]: tensile strength, tensile modulus, elongation at break, hardness and specific gravity.

III. Results and Discussion

3.1 Characterization of bamboo fibre

Measurement of some characteristics of carbonized bamboo fibre (CBF) and those of carbon black (CB) which served as the reference filler in this study was carried out, and the results are presented in Table 4.The weight loss in ignition is a measure of the carbon content lost during combustion and measures the effectiveness of the filler. The higher the value, the greater the reinforcement effect of the filler [18, 20]. The higher value of loss in ignition recorded for CB when compared to CBF is an indication that CB is more resistant to heat effect. Table 4 shows that the iodine adsorption number of CB is greater than that of CBF. The iodine adsorption number is a measure of the surface area of the filler; the higher the value, the finer or smaller the particle sizes of the filler (larger surface area) and the greater the reinforcing ability.

Table 4 also shows the oil absorption values for the fillers. The values indicate that the aggregate structure of carbon black is only greater than that of carbonized bamboo fibre particle size 0.08 and 0.35μm by about 2.51 and 2.93 kg, respectively. According to ASTM classification, CB consists of modular subunits called particles [18]. The similarity in the oil absorption values for the fillers may be an indication that CBF of particle sizes, 0.08 and 0.35μm have structures that may be closely related to CB, and therefore may produce similar effects in the vulcanizate [4].

3.2 Cure Characteristics of natural rubber vulcanizates

The effects of filler loading and compatibilizer on the scorch time, t_2 and cure time, t_{90} of carbonized bamboo fibre filled natural rubber vulcanizates are shown in Figs. 1 and 2. It can be seen that both properties decrease with increase in filler loading. This is attributed to longer residence time of the rubber vulcanizates in the mill during mixing. Similar observation was reported by Geethamma et al. [29]. According to the authors, the incorporation time of filler into rubber matrix increases as the filler loading increases and consequently generates more heat due to additional friction. However, at similar filler loading, the scorch and cure times of vulcanizates with compatibilizer are shorter than for the vulcanizates without compatibilizer, which is attributed to enhanced fibre-matrix adhesion and more energy required to incorporate the fibres due to increased viscosity.

The effect of filler loading and compatibilizer on Mooney viscosity of carbonized bamboo fibre filled natural rubber is shown in Fig. 3. The result shows that the Mooney viscosity increases with increase in filler loading and addition of compatibilizer. This indicates that the addition of both filler and compatibilizer increase the stiffness of the vulcanizates.

This observation is consistent with the report of Kumar et al. [30] for short sisal fibre filled styrene-butadiene rubber composites.

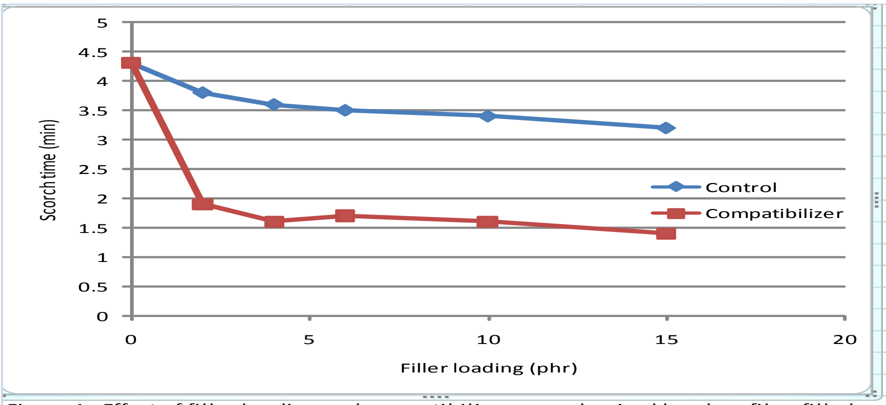

Figure 1. Effect of filler loading and compatibilizer on carbonized bamboo fibre filled natural rubber composites.

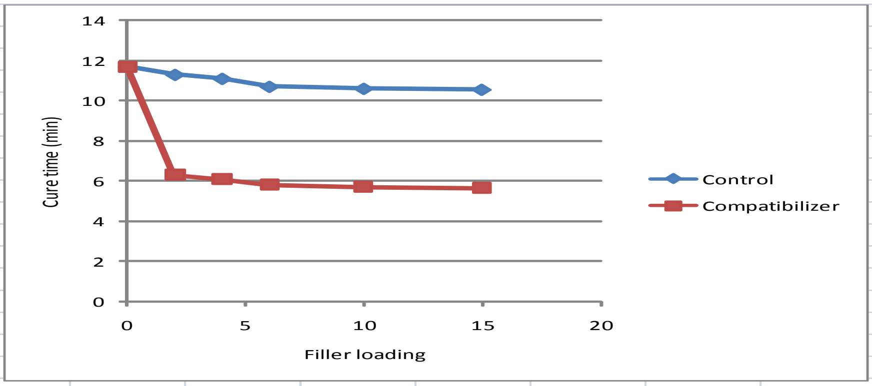

Figure 2. Effects of filler loading and compatibilizer on cure time of carbonized bamboo fibre filled natural rubber.

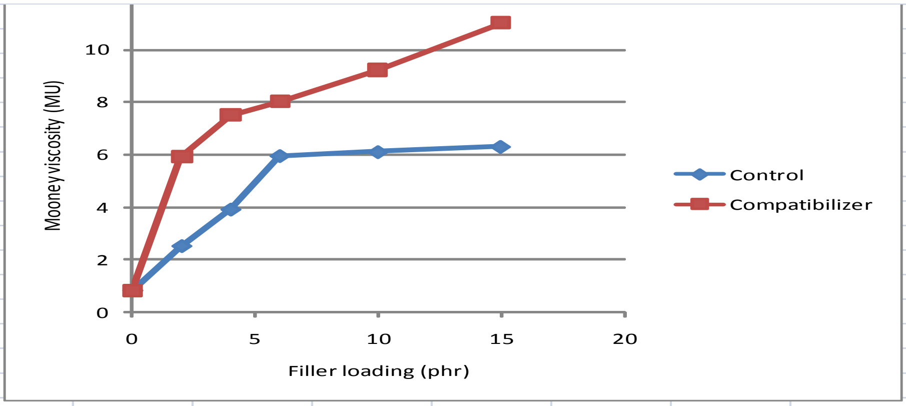

Figure 3. Effect of filler loading and compatibilizer on Mooney viscosity of carbonized bamboo fibre filled natural rubber.

3.3 Mechanical properties of natural rubber vulcanizates

The effect of filler loading and particle size on tensile strength of filled natural rubber is presented in Fig. 4. It can be seen that tensile strength decreases with increasing filler loading. The decrease in tensile strength of rubber vulcanizates with increase in filler loading has been reported by Arumugam et al. [31] who worked on coconut fibre reinforced rubber composites and found that the tensile strength of rubber vulcanizates decreased with increases in coconut fibre content. Similarly, Ismail et al. [12] reported e decrease in tensile strength of rubber vulcanizates with increase in filler loading when working with oil palm wood flour reinforced epoxidized natural rubber. The authors attributed the decrease in tensile strength to poor filler dispersion with filler addition. This behavior can be related to the probable tendency of the filler to form agglomerates. However, other researchers [32, 33] reported increases in tensile strength with increase in filler loading.

The tensile strength of rubber vulcanizates filled with CBF, particle size, 0.08µm showed higher tensile strength than those filled with CBF, particle size, 0.35µm. This observation is expected, and it is attributed to better filler dispersion and filler-matrix interaction. Generally, the smaller the particle sizes of filler, the greater the tensile strength of the vulcanizates [12]. Carbon black (CB), used as reference filler in this study steadily showed significant increases in the tensile strength of the vulcanizates, and produced higher tensile strengths than CBF filled NR. As earlier pointed out, the effectiveness of filler may be measured by its carbon content. Fillers with higher carbon content, provide greater reinforcement than those with lower carbon content because carbon itself is a very good reinforcing filler, Okieimen and Imanah [18]. It can be seen from the weight loss on ignition (Table 2) that CB has more carbon content than CBF and this partly explains the better reinforcing ability shown by CB over CBF. Tensile strength is affected by filler particle size, filler surface area and filler geometry. The filler geometry may be responsible for the poor strength property shown by carbonized bamboo fibre. Excellent reports by Mishra and Shimpi [32] have shown that irregularly shaped fillers could cause decreases in the strength of composites. The authors attributed this observation to the inability of the filler to support stresses transferred from the polymer matrix.

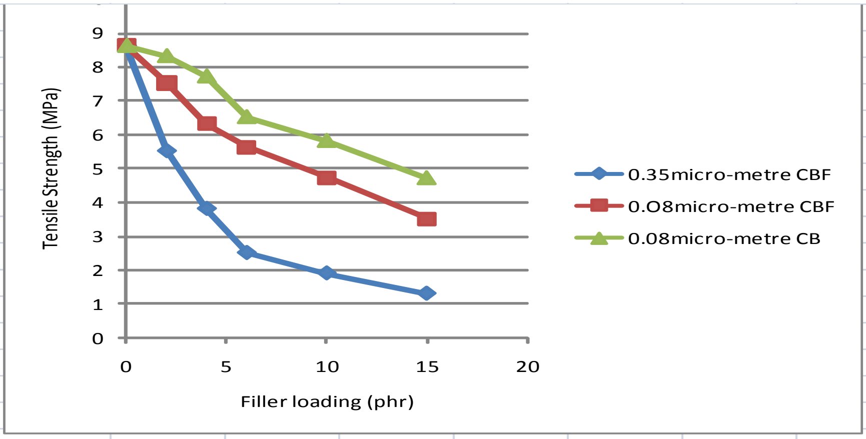

Figure 4. Effect of filler loading and particle size on tensile strength of filled natural rubber.

Figs. 5 and 6 show the effect of filler loading and particle size on tensile modulus and hardness of filled natural rubber, respectively. The results show that both properties increase with increasing filler loading, indicating increases in stiffness of the composites with the incorporation of filler into the rubber matrix. It is evident from the figure that the tensile moduli and hardness of carbonized bamboo fibre filled natural rubber are less than that of carbon black filled natural rubber, at all the filler loadings and particle sizes investigated. However, the carbonized bamboo fibre (CBF) with the smaller particle sizes (0.08µm) showed higher values in both properties in the vulcanizates than the CBF with larger particle sizes (0.35µm). This is because a reduction in particle size provides a greater surface area and reinforcement [36]. Carbon black has a very high surface activity, which provided greater reinforcement in comparison with carbonized bamboo fibre. Thus, the observed trends reveal that surface activity and polymer matrix-filler interaction are important factors controlling the tensile modulus and hardness. Ahmad et al. [3] reported that natural rubber forms a strong adsorptive bond with carbon black. In their independent studies, Kohjiya and Ikeda [34] and Poh et al. [35], reported that the modulus and hardness of filled vulcanizates can be enhanced by improving the surface and surface reactivity of fillers, filler dispersion and filler-polymer matrix interaction.

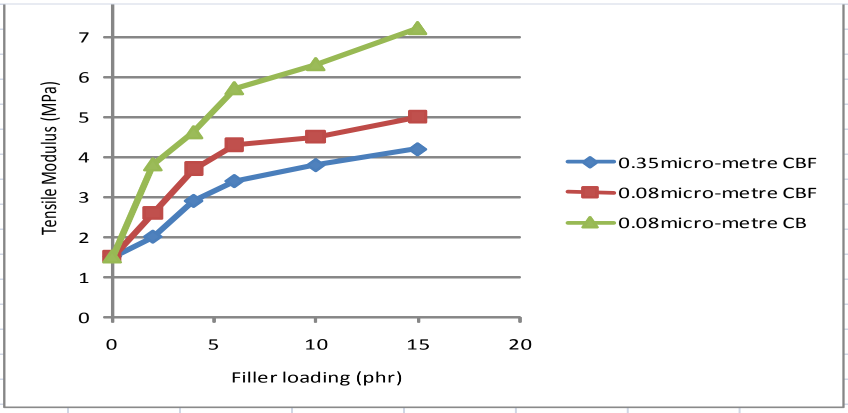

Figure 5. Effect of filler loading and particle size on tensile modulus of filled natural rubber.

Fig. 7 shows the effect of filler loading and particle size on elongation at break (EB) of vulcanized natural rubber. The result shows that the EB of carbon black filled natural rubber vulcanizate is higher than the EB of carbonized bamboo fibre filled natural rubber vulcanizate at the filler loadings studied. In general, the incorporation of reinforcing or non-reinforcing (inert) fillers into natural rubber produces decreases in elongation at break of rubber vulcanizates [18]. The decreasing trend in EB with increasing filler loading is attributed to increase in stiffness and brittleness, which decreased the resistance to stretch on application of strain.

However, at any given carbonized bamboo fibre loading investigated, the EB of the vulcanizates increases with decrease in the particle size of carbonized bamboo fibre. The increase in the EB with a reduction in the filler particle size can be attributed to more uniform dispersion of the smaller sized filler in the rubber matrix, which resulted in greater absorption and more efficient transfer of stresses to the matrix.

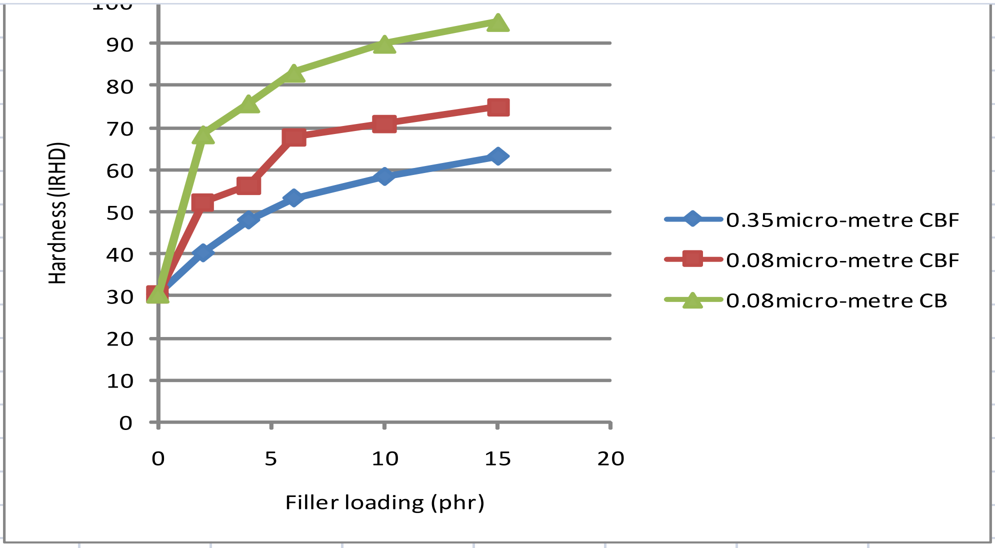

Figure 6. Effect of filler loading and particle size on the hardness of filled natural rubber.

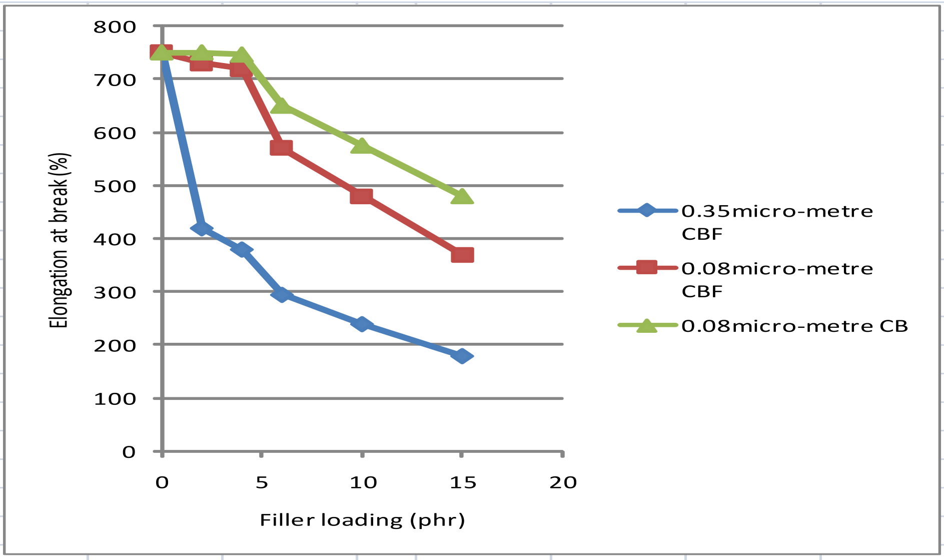

Figure 7. Effect of filler loading and particle size on the elongation at break of filled natural rubber.

Fig. 8 shows the effect of filler loading and particle size on the specific gravity of natural rubber vulcanizates. The figure shows a general increase in specific gravity with increases in filler loading, irrespective of the type of filler considered. This observation is consistent with the reports of Igwe and Ejim [4] and Mishra and Shimpi [32] who revealed a general increase in the specific gravity of rubber vulcanizates with increasing filler loading. However, at all particle sizes investigated, the specific gravity of the carbon black filled natural rubber vulcanizates was greater than those of rubber vulcanizates filled with carbonized bamboo fibre. This may be due to more uniform dispersion of carbon black in the matrix, with the resultant increase in filler-matrix interaction.

At any given loading of carbonized bamboo fibre considered, the specific gravity of the vulcanizates increases with a reduction in the particle size of the filler. The increasing trend in the specific gravity with decrease in the filler particle size may be due to the filler size effect, in which case the smaller sized filler became more uniformly dispersed in the rubber matrix to keep the rubber chain intact on crosslinking.

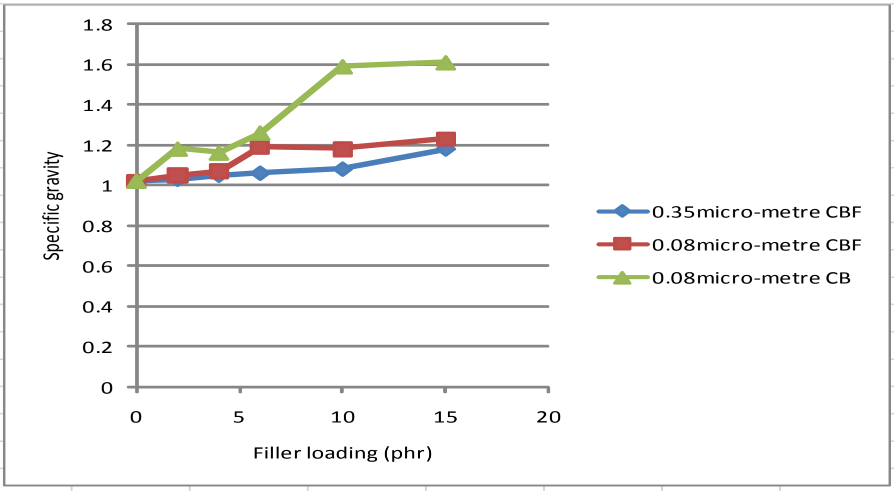

Figure 8. Effect of filler loading and particle size on specific gravity of filled natural rubber.

IV. CONCLUSION

The cure characteristics and physico-mechanical properties of carbonized bamboo fibre filled natural rubber vulcanizates were studied as a function of filler loading, filler particle size and compatibilizer. The scorch time, t_2 and cure time, t_{90} of carbonized bamboo fibre filled natural rubber vulcanizates decreased with increase in filler loading and the presence of compatibilizer.

Carbon black (CB), the reference filler, exhibited higher tensile strength, modulus and hardness in the vulcanizates than carbonized bamboo fibre (CBF). However, at any given loading of CBF considered, these properties increased with a reduction in particle size of the filler. An increasing trend in specific gravity with increases in filler loading was observed, irrespective of the type of filler considered. However, rubber vulcanizates containing smaller particle- sized CBF exhibited higher specific gravity than those containing the larger particle-sized filler.

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