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## ***Calamus deerratus* fibre reinforced natural rubber vulcanizates**

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### **ABSTRACT**

This research study investigates the potentials of *Calamus deerratus* fibre (CDF) as a reinforcing filler in natural rubber, Standard Nigerian Rubber (SNR<sub>10</sub>) vulcanizates. The *Calamus deerratus* was cut, dried, pulverized, characterized and incorporated into the natural rubber compounds and the effects on the cure characteristics and physico-mechanical properties of the CDF-reinforced natural rubber vulcanizates were determined. The results of the analyses, in comparison with a standard carbon black, CB(N330)-filled vulcanizates showed that the CDF manifested a reinforcing effect on the SNR<sub>10</sub> vulcanizates but inferior to carbon black, N330-filled vulcanizates. The maximum torque, abrasion resistance, modulus, specific gravity and hardness increased while the scorch time, cure time, elongation at break and rebound resilience decreased with increasing filler content. The cure time and scorch time were however higher in CDF-filled SNR<sub>10</sub> vulcanizates compared to those filled with carbon black, N330. Tensile strengths of CB(N330)- and CDF- filled vulcanizates increased to optimum levels at 40 phr and 30 phr respectively and decreased with increasing filler content. The results however showed that CDF imparted lower reinforcing potential as shown by the lower tensile strength, abrasion resistance and modulus. The hardness results of the CDF-filled vulcanizates showed slight superiority over that of CB(N330)-filled vulcanizates.

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**Keywords:** Natural rubber, *Calamus deerratus*, fillers, reinforcement and vulcanizates.

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### **INTRODUCTION**

The strong influence of reinforcing fillers on the physical and mechanical properties of elastomers has been a key research area in polymer science and technology for several decades. It has been acknowledged that the incorporation of filler particles to gum elastomers improved the processibility and a variety of vulcanizate properties such as the tensile and tear strengths, abrasion resistance, hardness,

specific gravity, and ageing behaviour, among others, hence significantly contribute to increased uses of polymeric materials in many engineering and technical applications. Several research studies have shown that without fillers, few polymers would have been adequate other than mere trivial applications (Boonstra, 1984; Hoffman, 1989; Okoh et al., 2014).

Carbon black appears to be the oldest and the most important reinforcing filler for

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polymers. However, many research and development activities have increased significantly since the 1990s to develop prototype materials from renewable resources such as plant materials which can achieve similar or higher reinforcing ability as carbon black in order to substitute or supplement its use as filler. Carbon black is derived from non-renewable resources such as petroleum and natural gas at ever increasing price. The environmental concerns are that carbon black reinforced polymeric products are largely non-biodegradable and they constitute among others, the world's greatest environmental pollution problems, and the need to conserve the already dwindling petroleum and natural gas reserves calls for these type of research studies. Several research reports have shown successful use of various plant materials, and that natural plant filler-reinforced composites are better than synthetic filler-reinforced composites in some properties such as enhanced biodegradability, ease of recycling, light in weight, non-corrosiveness, temperature resistance, cost reduction and decreased environmental pollution (Wang et al., 2003; Osabohien et al., 2007; Abu Bakar et al., 2012; Chaiwat et al., 2013; Okoh et al., 2014). Thus, natural plant fibre-reinforced biocomposites are a new area in polymer technology researches.

*Calamus deerratus*, (family, Arecaceae) is a dioecious, wild, tall, rattan palm, climbing up to 20 m high, it is a plant with prickly stems widely distributed in the rain forest regions of Nigeria and other African countries. They may have little or no known medicinal values, but, the apical bud (palm heart) is eaten in Sierra Leone and Ghana, the roasted young shoots are eaten in Ghana while in Senegal, the leaves are grilled over a fire and then macerated and the liquid is drunk to promote weight loss. The stems are used to make a wide range of articles including sponges, ropes, baskets and construction materials (Abbiw, 1990; Sunderland, 2001).

This study is intended to investigate the potential of *Calamus deerratus* fibrous material as filler in natural rubber compounds as a part of the ongoing search for renewable, value-added materials from locally available natural plant resources as alternative reinforcing fillers for polymers. The study also is to determine the cure characteristics and physico-mechanical properties of natural rubber vulcanizates filled with *Calamus deerratus* fibre in comparison with the natural rubber vulcanizates filled with a standard carbon black, CB (N330).

## MATERIALS AND METHODS

### Materials

The materials used for this study include *Calamus deerratus* obtained from local farmers at Agbor, Delta State, Nigeria. Standard Nigerian Rubber (SNR<sub>10</sub>) used was obtained from Foot-wear Accessories, Manufacturing and Distribution (FAMAD), Benin City, Nigeria. Industrial grade carbon black, CB(N330) was obtained from Nigerian National Petroleum Corporation (NNPC), Warri, Delta State, Nigeria, Industrial grade rubber compounding additives and rubber testing equipment were obtained from the Department of Polymer Technology, Auchi Polytechnic, Auchi, Edo State, Nigeria and rubber research institute of Nigeria, Iyanomo, Benin City, Nigeria, respectively.

### Methods

#### *Preparation and characterization of CDF, CB (N330) and SNR<sub>10</sub>*

The stems of *Calamus deerratus* were cut and the green cover (epidermis) carrying the leaves were removed to expose the slender, tough, whitish stems. These stems were twisted with hands into white strands (sponges), and were dried in an oven maintained at 120 °C. These sponges were removed from the oven, cooled and ground into fine whitish powder using the corona grinding machine. The pulverized specimen

was screened with sieves of upper mesh size, 200  $\mu\text{m}$  and lower mesh size of 15 $\mu\text{m}$ . The screened sample was characterized in terms of moisture content, loss on ignition, iodine adsorption number, density and pH of its aqueous slurry, relative to those of CB(N330) using standard test methods (Vogel, 1964; ASTM D1510, 1983; AOAC, 1990). The SNR<sub>10</sub> used in this study was characterized in terms of its dirt, ash, nitrogen and volatile matter contents, Mooney viscosity and plasticity retention index (PRI) using standard techniques (RRIM, 1989; SAR, 1998).

#### **Compounding and curing the mixes**

The formulation given in Table 1 was used to compound the natural rubber, SNR<sub>10</sub> and efficient vulcanization (EV) system was employed. Each of the compounds was mixed and masticated using the laboratory two-roll mill of size 160 x 320 mm maintained at 80 °C. From this stock, unvulcanized samples were cut to allow testing of cure characteristics by using the Monsanto Rheometer, MDR 2000 model. The compounded natural rubber was cured by compression moulding in a steam heated, hydraulically operated press with a pressure of 150 kg/cm<sup>2</sup> at a temperature of 180 °C at the respective cure times derivable from the Monsanto rheographs.

#### **Determination of the physico-mechanical Properties of vulcanizates**

The tensile properties (tensile strength, modulus and elongation at break) of the vulcanizates were measured with the Monsanto Instron Tensometer (model 4301) at a crosshead speed of 500 mm/minute at room temperature using the dumb-bell shaped test pieces in accordance with standard procedures (ASTM D412, 1983). The rebound resilience was determined by the Wallace Croydon Resilometer 2A, while specific gravity and hardness of the vulcanizates were measured

by the Monsanto Densitron 2000 and the Wallace Croydon hardness tester respectively. The abrasion resistance was determined using the Akron abrader in accordance with standard procedures (BS 903, 1982).

#### **RESULTS**

Table 1 shows the recipe for the formulation of the standard Nigerian rubber (SNR<sub>10</sub>) compounds. Two different fillers were used in the formulation: they are the test filler, *Calamus deerratus* fibre (CDF) and standard carbon black, CB(N330) filler. Table 2 depicts the physicochemical properties of the test natural rubber, SNR<sub>10</sub> in comparison with other used natural rubbers such as the standard African rubber, SAR<sub>10</sub> and the standard Malaysian rubber, SMR<sub>5</sub>. The ageing resistance test result that is the plasticity retention index (PRI), the Mooney viscosity or flow resistance of the rubbers were presented together with the purity test results such as the dirt, ash, volatile matter and nitrogen contents. Table 3 itemizes the physicochemical properties of the fillers used; the *Calamus deerratus* fibre and standard carbon black (N330). The percentage moisture content, loss on ignition, iodine adsorption number which is a fair measure of surface area of filler, the pH of the aqueous slurry, metal and non-metal contents and particle size range of the fillers were shown in Table 3.

Figures 1-10 presented the physico-mechanical properties of the test natural rubber, SNR<sub>10</sub>-CDF vulcanizates in comparison with the natural rubber, SNR<sub>10</sub>-CB(N330) vulcanizates. The cure characteristics such as scorch time, cure time and maximum torque, the tensile properties such as tensile strength, modulus and elongation at break and the physical properties such as specific gravity, abrasion resistance, rebound resilience and hardness results were presented.

**Table 1:** Recipe for the formulation of SNR<sub>10</sub> compounds.

| <b>Ingredients</b>                  | <b>phr</b> |
|-------------------------------------|------------|
| Natural Rubber (SNR <sub>10</sub> ) | 100        |
| Zinc oxide                          | 4.0        |
| Stearic acid                        | 2.0        |
| Filler                              | 0.0-70.0   |
| Processing oil                      | 2.0        |
| CBS <sup>a</sup>                    | 2.0        |
| TMQ <sup>b</sup>                    | 1.5        |
| Sulphur                             | 1.5        |

<sup>a</sup>CBS = N-Cyclohexylbenzothiazyl sulphenamide

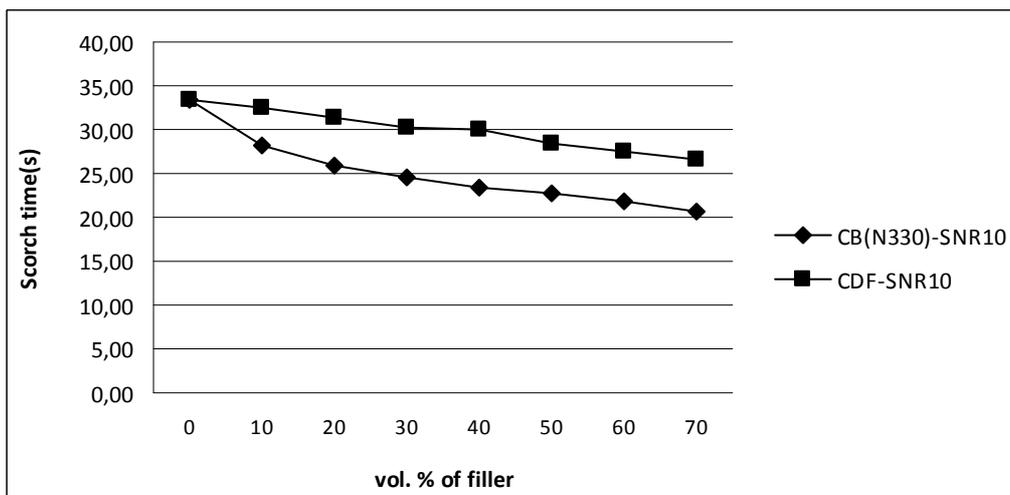
<sup>b</sup>TMQ = 2, 2, 4-trimethyl-1, 2-dihydroquinoline.

**Table 2:** Physicochemical properties of SNR<sub>10</sub>, SAR<sub>10</sub> and SMR<sub>5</sub>.

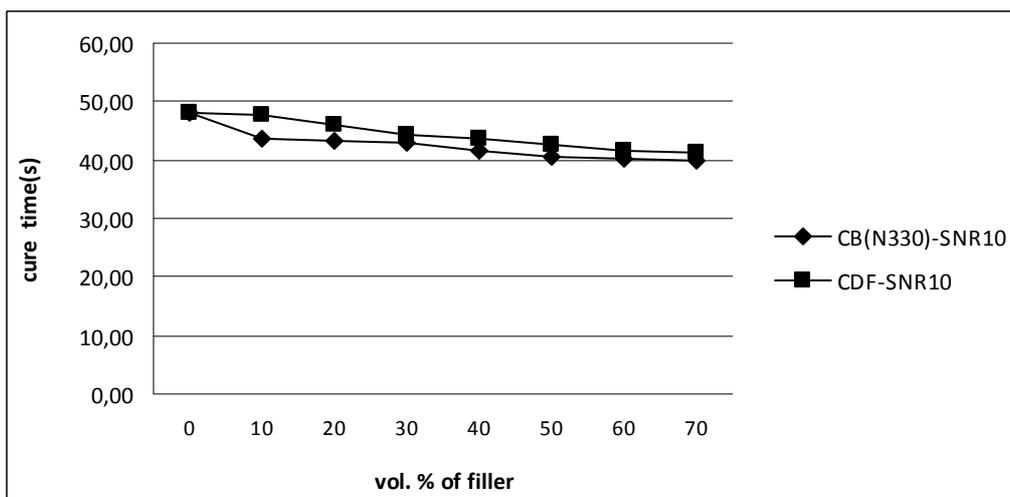
| <b>Natural rubber</b>            | <b>SNR<sub>10</sub></b> | <b>SAR<sub>10</sub></b> | <b>SMR<sub>5</sub></b> |
|----------------------------------|-------------------------|-------------------------|------------------------|
| Dirt content (%)                 | 0.03                    | 0.02                    | 0.05                   |
| Ash content (%)                  | 0.20                    | 0.22                    | 0.50                   |
| Volatile matter                  | 0.41                    | 0.40                    | 1.00                   |
| Nitrogen content                 | 0.25                    | 0.25                    | 0.70                   |
| Plasticity retention index (PRI) | 70.86                   | 67.00                   | -                      |
| Mooney viscosity ML(1+4) 100 °C  | 85.00                   | 70.00                   | 60.00                  |

**Table 3:** Physicochemical properties of CB (N330) and CDF.

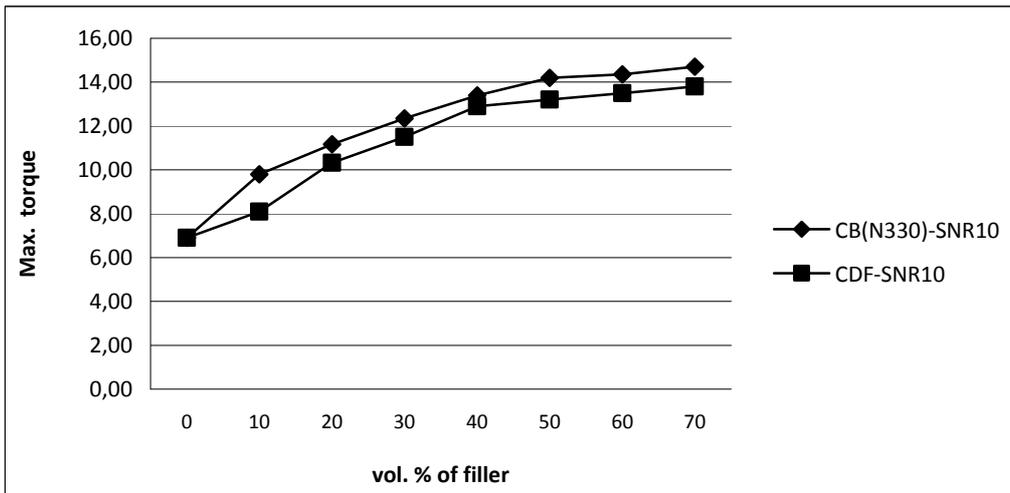
| <b>Filler</b>                             | <b>CB(N330)</b> | <b>CDF</b> |
|---|-----------------|------------|
| % Moisture content at 120 <sup>0</sup> C  | 1.30            | 2.25       |
| % Loss on ignition at 1000 <sup>0</sup> C | 93.0            | 80.00      |
| Iodine Adsorption number (mg/g)           | 78.50           | 56.20      |
| pH of aqueous slurry                      | 6.80            | 5.90       |
| Magnesium (ppm)                           | trace           | 53.10      |
| Sodium (ppm)                              | Trace           | 51.20      |
| Potassium (ppm)                           | Trace           | 16.10      |
| Iron (ppm)                                | Trace           | 23.40      |
| Chlorine (ppm)                            | Trace           | 17.80      |
| Density (g/cm <sup>3</sup> )              | 1.80            | 1.30       |
| Particle size range                       | 30-35 nm        | 15-200 μm  |



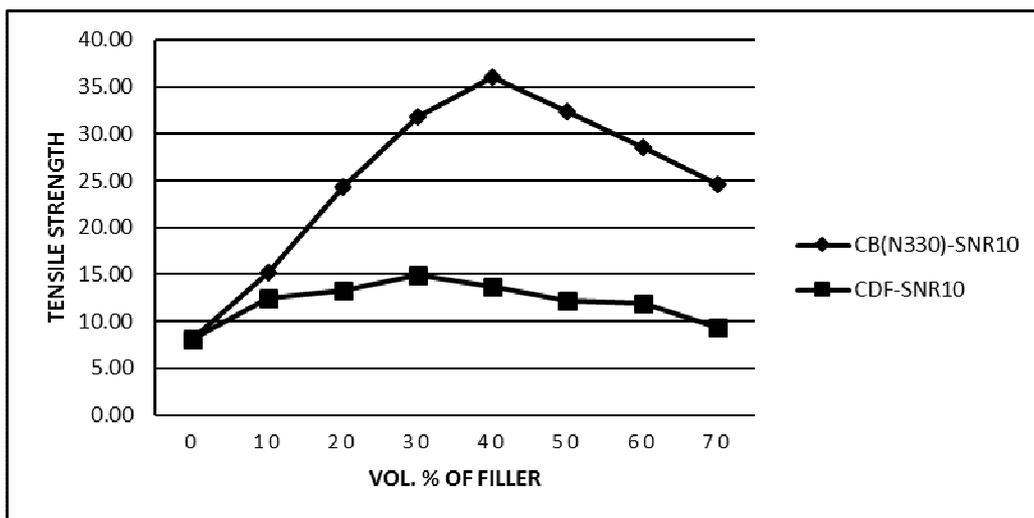
**Figure 1:** Scorch time of rubber/filler composites as functions of volume fraction of fillers.



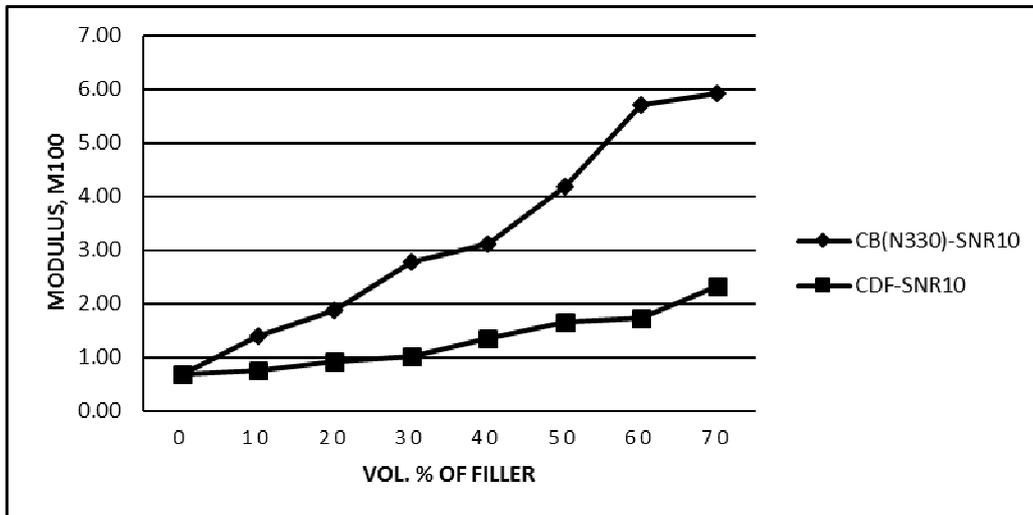
**Figure 2:** Cure time of rubber/filler composites as functions of volume fraction of fillers.



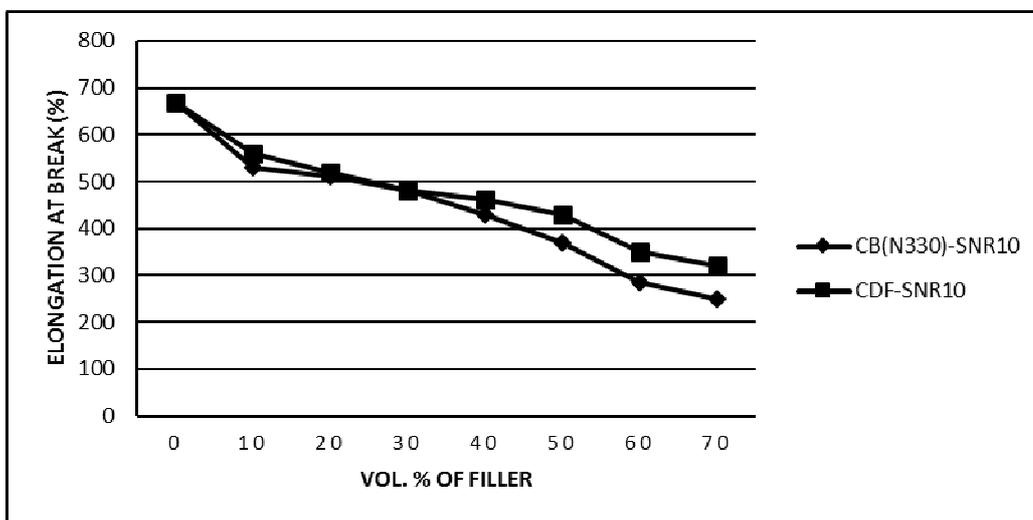
**Figure 3:** Maximum Torque,  $T_{max}$  (lb-in) of rubber/filler composites as functions of volume fraction of fillers.



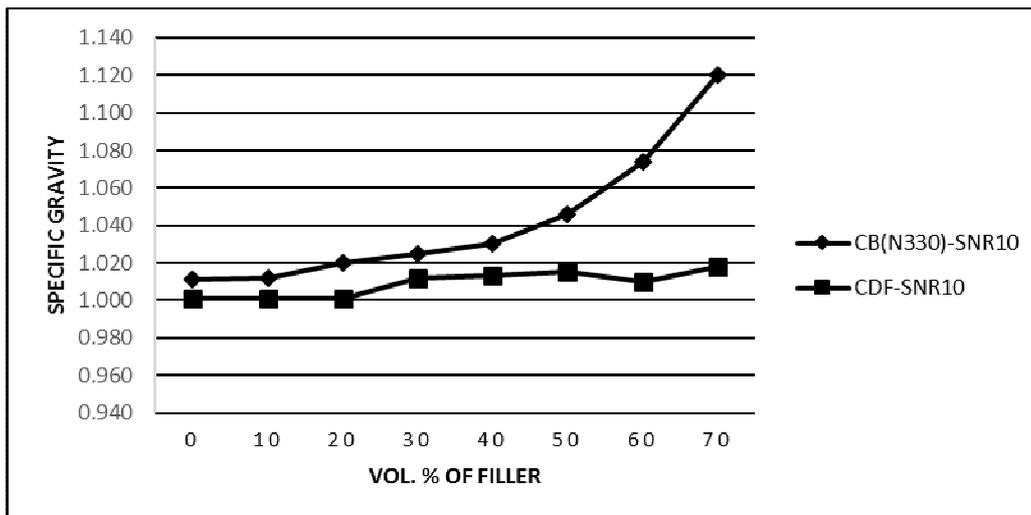
**Figure 4:** Tensile strength (MPa) of rubber/filler composites as functions of volume fraction of fillers.



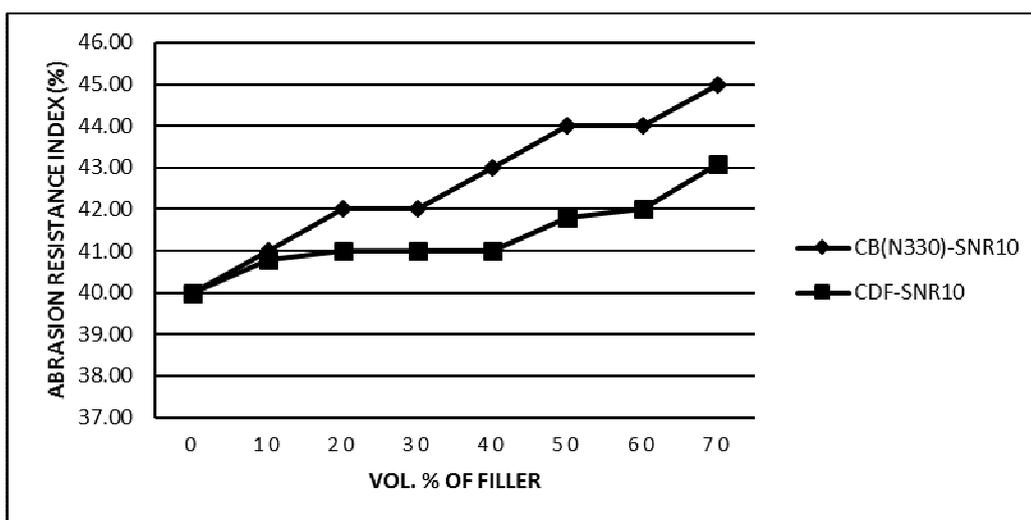
**Figure 5:** Modulus, M100 (MPa) of rubber/filler composites as functions of volume fraction of fillers.



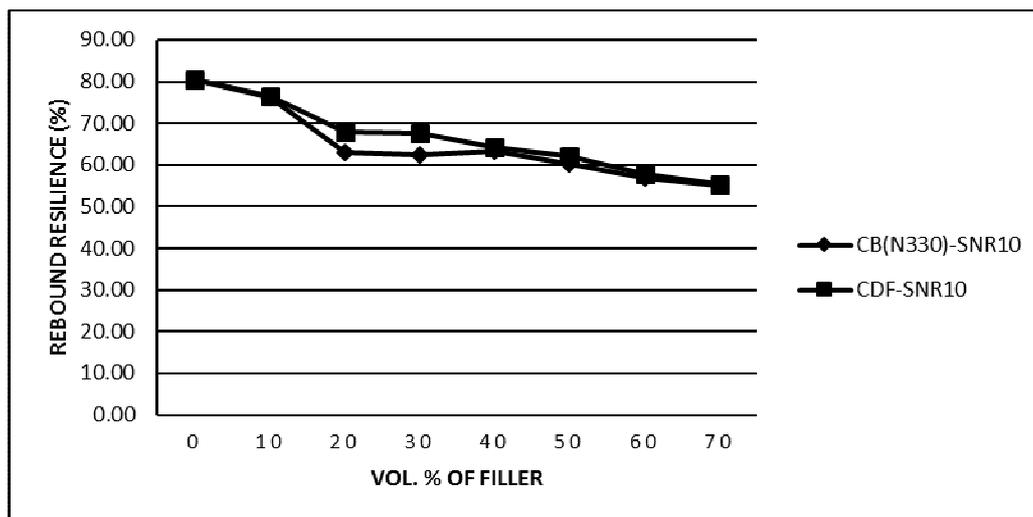
**Figure 6:** Elongation at break (%) of rubber/filler composites as functions of volume fraction of fillers.



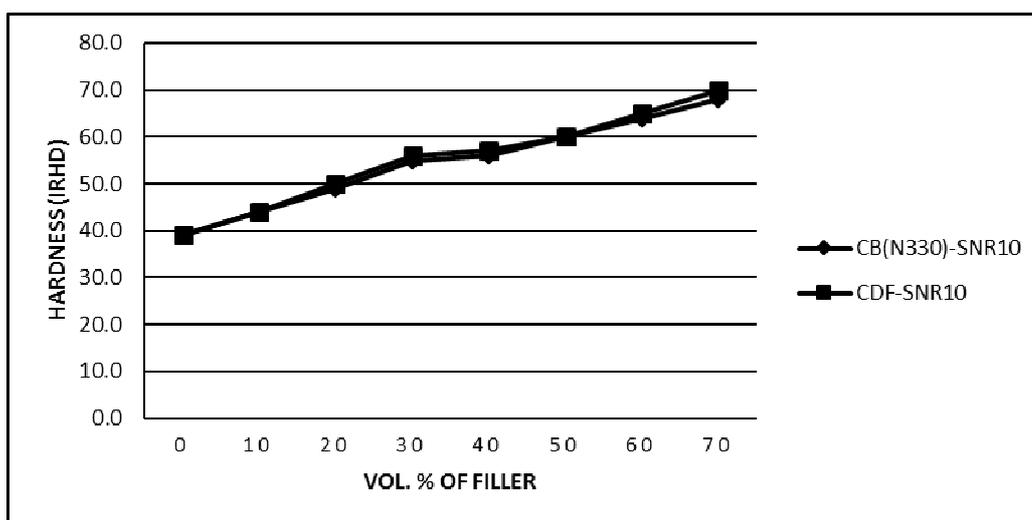
**Figure 7:** Specific gravity of rubber/filler composites as functions of volume fraction of fillers.



**Figure 8:** Abrasion resistance Index (%) of rubber/filler composites as functions of volume fraction of fillers.



**Figure 9:** Rebound resilience (%) of rubber/filler composites as functions of volume fraction of fillers.



**Figure 10:** Hardness (IRHD) of rubber/filler composites as functions of volume fraction of fillers.

## DISCUSSION

### The Physicochemical properties of SNR<sub>10</sub> and the CDF fillers

The results of the foregoing analyses are shown in Tables 2–3. The values of the physicochemical properties of SNR<sub>10</sub> (Table 2) compared favourably with those of other standard natural rubbers such as Standard

African Rubber, SAR<sub>10</sub> and Standard Malaysian Rubber SMR<sub>5</sub> (RRIM, 1989; SAR, 1998; Akinlabi and Egbon, 2003). The values obtained in this study suggest that the raw natural rubber, SNR<sub>10</sub> was of a high quality material. The high values of the plasticity retention index (PRI) and the Mooney viscosity indicate a high resistance of the

resulting vulcanizates to aging and flow respectively (Blow and Hepbum, 1982; Okoh et al., 2014).

The results in Table 3 showed that CB(N330) had lower moisture content at 120 °C than the *Calamus deerratus* material. High moisture content of a filler can lead to poor filler-rubber matrix interactions resulting in poor mechanical strength properties of rubber vulcanizates (Ishak and Bakar, 1995; Puglia et al., 2005; Osabohien et al., 2006). The results also showed that the loss on ignition at 1000 °C was higher for CB(N330) thus suggesting that CB(N330) has higher content of carbon and other combustible materials than CDF. It has been postulated that fillers with high content of carbon exhibit high reinforcing effect (Osabohien and Egboh, 2007a and 2007b). The results further showed that CB(N330) has higher iodine adsorption number and hence larger surface area (which implies smaller particle size) than the CDF filler. It has been documented that the most important factor that determines the reinforcing ability of a filler is its particle size (or surface area); a reinforcing filler must have very small particle size (large surface area) in order to maintain a strong surface contact with the polymer matrix (Boonstra, 1984; Osabohien and Egboh, 2007b; Yamashita and Tanaka, 2014). Both fillers are acidic as shown by the pH of their aqueous slurries, but the CDF is more acidic. The CDF filler also has higher metallic and non-metallic contents than CB(N330). These surface active groups can contribute to the surface reactivity of a filler which can influence its reinforcing potential. It has been shown that they could have a catalytic effect on the rate of cure, modulus, electrical and thermal conductivity of vulcanizates (Blow and Hepbum, 1982; Osabohien and Egboh, 2007b; Osabohien, 2010).

#### **Cure characteristics of SNR<sub>10</sub> vulcanizates**

The data in Figures 1-3 summarize the results of analyses of the cure characteristics of SNR<sub>10</sub> filled separately with CB(N330) and CDF at different filler loadings. The results revealed that the scorch times and cure times of both systems decreased while the maximum torques increased with increasing filler content which is consistent with observations made in earlier studies (Osabohien et al., 2007; Mohamad et al., 2008; Okoh et al., 2014). The CDF-filled SNR<sub>10</sub> vulcanizates had higher scorch times and cure times and lower maximum torque than CB(N330)-filled SNR<sub>10</sub> vulcanizates probably due to the larger particle size, smaller surface area, higher moisture, metals or non-metal contents, presence of lignin and other impurities of the CDF compared to the CB(N330). The metals or their oxides present in the CDF filler could have a retarding effect on the accelerator activity which in turn can slow down the sulphur vulcanization process leading to increase in cure time and scorch time (Osabohien and Egboh, 2007b). The increase in the torque values suggests that there is a good interaction between the filler surface and rubber matrix which results in some reinforcement in the vulcanizates. Thus, the lower maximum torque values of CDF compared to CB(N330)-filled SNR<sub>10</sub> vulcanizates is probably due to a weaker CDF filler-rubber matrix interactions which results in lower crosslink density and lower restrictions to flow of the polymer chains, and this may be attributed to the larger particle size and higher moisture content of the CDF filler.

#### **Physico-mechanical properties of SNR<sub>10</sub> vulcanizates**

Figures 4-10 showed that the tensile strength, modulus, specific gravity, abrasion resistance and hardness of both filled systems

increased while rebound resilience and elongation at break decreased with increasing filler content. These observations are consistent with similar research studies (Ishak and Bakar, 1995; Okoh et al., 2008; Mohamad et al., 2008; Okoh et al., 2014). The results also showed that the tensile strengths of both the CB(N330)- and CDF-filled SNR<sub>10</sub> vulcanizates increased to optimum at 40 phr and 30 phr filler contents respectively and then decreased with increasing filler content. This is consistent with earlier findings (Wang et al., 2003; Osabohien and Egboh, 2007b; Osabohien, 2012; Okoh et al., 2014). The decrease in tensile strength after attaining an optimum level is a phenomenon of “phase inversion”, also taken as a dilution effect resulting from the fact that there is not enough polymer matrix to wet or hold the filler particles thereby creating voids and reducing strength. This is also a case of agglomeration of filler particles due to filler overloading (Boonstra, 1984; Wang, 1999; Osabohien and Egboh, 2007b and 2008). The results showed that CB(N330)-filled vulcanizates had higher tensile strength, modulus, specific gravity and abrasion resistance but lower elongation at break and rebound resilience than the CDF filled vulcanizates. The superior tensile properties may be due to the finer particle size and larger surface area of CB (N330) (Table 3). The finer particle size and hence the larger surface area of CB(N330) would ensure better dispersion and wetting of the filler particles by the rubber molecules. The higher the filler-polymer matrix interactions, the higher the degree of crosslinking and the higher the reinforcement. The higher rebound resilience of the CDF-filled SNR<sub>10</sub> vulcanizates implies lower heat build-up and hysteresis in the rubber products (Osabohien and Egboh, 2007b; Osabohien, 2012; Okoh et al., 2014). The higher values of elongation at break and rebound resilience of the CDF-filled SNR<sub>10</sub>

vulcanizates compared to the CB (N330)-filled system implies a weaker rubber matrix-filler adhesion and hence a lower restriction to flow of the macromolecular chains so that the vulcanizates can be easily stretched on the application of strain (Blow and Hepburn, 1982; Boonstra, 1984; Osabohiem and Egboh, 2007b; Okoh et al., 2014).

However, the hardness of the CDF-filled natural rubber vulcanizates is slightly higher than that of CB (N330)-filled types. Therefore, this is one superior property CDF has over CB (N330) filler, especially for rubber articles requiring high hardness and stiffness properties. This result is in conformity with previous observation made for bowstring hemp fibre reinforced natural rubber vulcanizates (Osabohien and Egboh, 2008).

### **Conclusion**

This research study has shown that the CDF filler influenced the cure characteristics and physicochemical properties of the natural rubber, SNR<sub>10</sub> vulcanizates, although it imparted lower reinforcing effects than the commercial grade carbon black, CB(N330) as shown by the lower maximum torque values, tensile strength, abrasion resistance, and modulus results. This may be largely due to the larger particle size, smaller surface area and higher moisture content of the CDF filler as compared to that of carbon black, CB (N330). These defects could be overcome by pretreatment of the CDF material with a suitable coupling agent and also employing modern methods of wet and dry grinding techniques to obtain finer particle size before application. These could enhance the reinforcing potentials of the CDF for applications requiring light weight polymeric materials.

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