



# PREPARATION OF KERATINOUS PARTICLES FROM CHICKEN FEATHER FIBRES AS POTENTIAL SUBSTITUTE FOR CARBON BLACK REINFORCED NATURAL RUBBER COMPOSITES



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**Abstract:** Several biowastes have been recently researched as possible substitute for carbon black as filler in natural rubber composites. This work shifted from the usual cellulosic waste to a keratinous waste-chicken feather currently posing environmental threats worldwide. Keratinous nanoparticles were prepared via top – bottom approach and validated via Dynamic Light Scattering analysis. The fillers and vulcanizing additives were mixed and compounded using a two roll mill and a compression moulding machine, respectively. The thermal stability and physicochemical properties: shore A hardness, tensile strength, modulus 100 and elongation at break of natural rubber vulcanizates when filled with chicken feather fibre nanoparticles in comparison with carbon black filler were studied. The filler loadings ranged from 0 to 10 ppH at intervals of 2. From the results, nanoparticles from chicken feather fibres gave superior thermal stability with a residue of 15.55% as against 8.77%, increase in hardness by 6%, enhancement in modulus (M100) by 71% at 10 ppH, higher elongation at break of 49% at 2 ppH and wear percent of 0.3% as against 0.16% at 10 ppH, when compared with carbon black filled rubber vulcanizates.

**Keywords:** Feather fibres, shore A hardness, thermal stability, rubber vulcanizate

## Introduction

Recently, polymers reinforced with eco-friendly biodegradable and low cost agricultural waste are gaining grounds in polymer composite development. Improved properties are also recorded when micro to nano sized fillers are incorporated in polymer matrices.

Natural rubber (NR) is a green biopolymer with high molecular weight of isoprene (2, 4 methyl-1, 3 butadiene) from agricultural source. NR in its virgin state possesses low strength and dimensional instability under thermal and loading conditions making it practically useless. Hence, virgin rubber is usually reinforced and vulcanized in order to meet useful applications. The inherent strength and stability of natural rubber vulcanizates can be enhanced by incorporating a reinforcing phase in order to obtain improved modulus and durability required for structural and engineering applications. In a bid to replace carbon black which is one of the most dominant yet harmful reinforcing fillers in natural rubber composites, several agricultural wastes have been recently exploited. Carbon black is obtained from the incomplete combustion of heavy petroleum products and is associated with negative environmental (such as global warming) and health (cancer) implications (Verma *et al.*, 2012). It is also known to limit the aesthetic design of rubber products due to its characteristic black colour. Alternative rubber reinforcing fillers to carbon black include sugarcane bagasse (Okele *et al.*, 2015), carbonized coir and uncarbonized coir (Aguale *et al.*, 2014), velvet tamarind seed shell (Okoh *et al.*, 2014), maize stalk (Chigondo *et al.*, 2013) and bamboo fibre (Onyeagoro, 2012).

Keratinous sources have nevertheless received low attention and exploitation as compared to their cellulosic counterparts. Keratinous fillers among other natural fillers display unique viscoelastic behavior with an initial linear region followed by a plastic region. This makes them capable of absorbing energy and withstanding forces (Wang *et al.*, 2016). A few exploited keratinous fillers include cow bone (Oyetunji *et al.*, 2018), feather meal (Hergenrother *et al.*, 2015) and snail (Igwe and Ejim, 2011).

Feather keratin possesses relatively low density making them attractive in several industries (Ghosh *et al.*, 2017; Ghassamieh, 2011). The cysteine molecules in keratin give

it high strength (Wrześniewska-Tosik and Adamiec, 2007). The twisted microfibrils and unique honeycomb of feather structure yield fibres of high strength and thermal resistance properties (Oladele *et al.*, 2018). The flexible nature and high aspect ratio of feathers' fibre provides toughness and tensile strength required for reinforcement in composite development (Uviersherhe *et al.*, 2015; Salehuddin *et al.*, 2014). The use of chicken feather as a replacement to carbon black in rubber will provide an economic and sustainable use of the agricultural waste (Staron *et al.*, 2011).

Hitherto, feather particles have not been reported as reinforcement in natural rubber matrix. Thus, this research focused on the use of keratinous particles obtained from chicken feather fibre as reinforcement in natural rubber composites and studied the effect of their loading ratio on several mechanical and thermal properties in comparison with carbon black reinforced composite prepared under the same conditions. The fillers and vulcanizing agents were masticated with natural rubber using a two roll mill while vulcanization was carried out with a compression moulding machine. Several physicochemical analyses were carried out and their thermal stabilities were characterized using thermogravimetric analysis (TGA).

## Materials and Methods

### Materials

Chicken feathers were collected from a slaughter house in Sabon-gari Local Government Area, Zaria, Nigeria. The carbon black (N330) was manufactured by Warri refinery and petrochemical a subsidiary of Nigerian National Petroleum Corporation (NNPC) with particle size distribution of 50 to 500 nm and was used as received. Dry natural rubber crumb, sulphur, trimethylquinolone (TMQ), stearic acid, zinc oxide and mercaptobenzothiazole disulfide (MBTS), were procured from Tony West rubber factory, Lagos, Nigeria. The specifications of the dry rubber were  $\leq 1.00\%$  volatile content,  $\leq 0.05\%$  dirt content,  $\leq 0.6\%$  ash content,  $\leq 0.7\%$  nitrogen content, minimum of 30 initial plasticity, minimum of 60 plasticity retention index (PRI) and blackish brown in colour.

**Development of nanoparticles from chicken feathers**

The sourced chicken feather fibres were stripped off their quill, washed and sun dried. They were further subjected to oven drying at 80°C for 2 h. The conventional top-down approach of manufacturing micro to nanoparticles using a ball mill was employed (Thakur *et al.*, 2012). The dried feather fibres milled repeatedly in a Thomas-Wiley laboratory mill through 0.05 mm mesh sieve. The ground particles recovered from this process was further screened through a 125 µm (sieve mesh number 120 according to BS410) sieve mesh and then ball milled at an estimated vessel speed of 300 rpm at ball to particle ratio (BTP) of 1:20 on weight basis with an average porcelain ball diameter of 2 cm for several hours. The particles were subjected to particle size distribution measurement based on the principle of dynamic light scattering (DLS) with the aid of Malvern mastersizer 3000 (Malvern instrument UK) using water as the dispersant at viscosity of 0.8872 Cp and the results shown in Fig. 1.

**Compounding and vulcanization of composites**

Particles of chicken feather (CF) and carbon black (CB), vulcanizing agents and natural rubber matrix were compounded using a two roll mill, followed by an in-situ moulding and curing in a compression moulding machine. The formulation used was given on Table 1. The compositions of the particles ranged from 0 to 10 ppH with zero standing for the unfilled rubber vulcanizate. The compounded composites were moulded and vulcanized using locally fabricated metallic moulds of appropriate test shape and dimensions. The cure conditions for the sulphur vulcanization were 140°C for 13 min using compression moulding technique in an 11 tonne hydraulic press.

**Table 1: Formulation for reinforced natural rubber composites**

Ingredients	Parts per hundred of rubber
NR	100
Zinc oxide	5
MBTS	2
TMQ	1
Stearic acid	2
Sulphur	3
Fillers (carbon black, chicken feather)	0 – 10 (at an interval of 2 ppH)

**Physicomechanical and abrasion resistance analyses**

**Tensile properties (tensile strength, elongation at break, modulus of elasticity)**

The tensile strength was conducted and other parameters calculated in accordance to ASTM D412. Tensile tests were carried out at room temperature using universal Instron machine model 3366 with a load cell 1 kN. Pre-moulded dumb bell shaped specimens were used to perform the experiment at a loading speed of 80 mm/min. The maximum breaking stress (tensile strength) ( $\sigma_t$ ) was calculated using the equation below;

$$\sigma_t = \frac{F}{b \times d}$$

Where F is the breaking force (N), b is the width (mm) of the sample and d is the thickness (mm) of sample. The effect of varying filler loading and filler type on tensile strength of natural rubber composites is shown in Fig. 2.

The elongation at break was also calculated using the equation below.

$$\text{Elongation at break} = \frac{\text{Final length at break} - \text{Initial gauge length}}{\text{Initial gauge length}} \times 100\%$$

The effect of varying filler loading and filler type on elongation at break of natural rubber composites is shown in Fig. 3.

The modulus at 100% elongation was calculated as given in the equation below. Modulus of;

$$\text{Elasticity} = \frac{\text{Maximum stress at break}}{\text{Strain at break}}$$

The effect of varying filler loading and filler type on Modulus 100 of natural rubber composites at 100% elongation is shown in Fig. 4.

**Shore A hardness**

The hardness of the samples was determined with the aid of a Durometer (Instrol Wilson) according to ISO 7619-1. The samples were cut from each of the vulcanized rubber composite already conditioned. The samples were placed on the foot of Durometer’s indenter to determine the extent of penetration of indenter into the sample at several points. The indenter of the Durometer was then pressed into the specimen and the depth of the indentation was read from the Durometer. Five locations on every sample were penetrated and the average computed as the hardness in IRHD in order to ensure reliability. A mean of the five indentations were calculated and recorded. The effect of varying filler loading and filler type against the hardness values of natural rubber composites were plotted in Fig. 5.

**Wear test**

The abrasion loss of the vulcanized rubber composite samples were determined with the aid of rotary drum abrader in terms of volume loss according to ISO 4649 – 2010 (E) standard using a Martindale Abrasion machine. Three abrasion specimens of each sample with thickness 5 mm and diameter 30 mm were prepared with a drill. The samples were removed from a dessicator and weighed before the procedure. Each sample was mounted against the drum of the abrasion testing machine at applied force of 10 N. The abrasive surface of the wear test machine was covered with an abrasive paper of grade P 220. The machine was programmed to run and stop after 100 revolutions at 40 rev/ min. The samples were reweighed at the completion of each run. The initial,  $w_1$  and final weights,  $w_2$  of the samples in triplicate were measured and the mean weight losses calculated according to equation below;

$$W \% = \frac{w_2 - w_1}{w_1} \times 100 \%$$

The effect of varying filler loading and filler type on weight loss of natural rubber composites were plotted as shown in Fig. 6.

**Thermo-gravimetric analysis (TGA)**

Natural rubber composites reinforced with 10 ppH of treated and untreated fibres were subjected to TGA measurement as against the unfilled composites using TGA 4000, Perkinelmer. An initial weight was taken and heated from 30 to 900°C at a heating rate of 10°C/min. The temperature for different percentages of weight loss, temperature at maximum decomposition and residue at 800°C of all composites samples were determined and the results tabulated on Table 2.

**Results and Discussion**

**Particle size distribution analysis**

The exact sizes of particles were difficult to obtain because of the high number of entanglement and complexity at micro to nano-structural level. Nevertheless, peaks and the percentage volume of each size distribution were used to obtain a range as shown in Fig. 1.

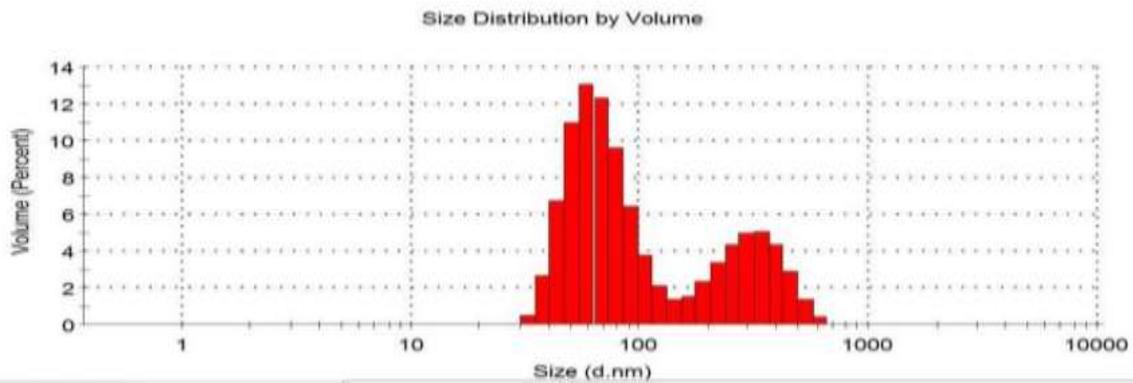


Fig. 1: Particle size distribution of size reduced feather fibres

The particle size of the feather fibre was found to be distributed between 38.4 – 282.9 nm with higher percentage volume of less than 100 nm. This amongst other factors plays an important role in determination of the rubber reinforcement. Generally particles size of less 100 nm is considered to be high reinforcement filler, while particle size of less than 1000 nm is considered to be medium reinforcement filler (Vineetkumar, 2008). Hence, the results of particle size distributions obtained for chicken feather fibre suggest suitability for medium to high reinforcement for rubber matrix.

**Tensile Strength**

The tensile strength of the composites reinforced with particulate chicken feathers (CF) and carbon black (CB) as presented in Fig. 2, measures the maximum force required to fracture a unit area of the composites under static or quasi-static loading conditions at a defined temperature and strain rate (Oboh *et al.*, 2017).

Generally, from the data presented the tensile strength increased with increasing filler loading for both fillers (CF and CB). Improvements from unfilled rubber vulcanizates can be attributed to a high degree of interaction between the rubber chains and the filler particles (Sinclair *et al.*, 2017). Nevertheless, carbon black gave slightly higher values. The higher value of tensile strength for carbon black based composites can be attributed to higher surface reactivity of carbon black with polyisoprene molecules which results to covalent bonds when compared with keratinous particle surface. Previous reports by Vineetkumar (2008) have shown that carbon black is capable of bonding both physically and chemically to rubber phase due to its surface reactivity. Nevertheless, particulate chicken feathers (CF) gave an improvement from 1.35 for unfilled rubber to 2.52 MPa at 10 ppH of CF loading and generally close tensile strength values with regards to carbon black (CB) reinforced natural rubber composites at all filler loadings.

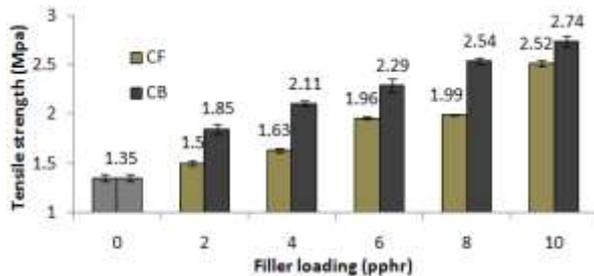


Fig. 2: Tensile strength values for rubber composites reinforced with varying filler loadings of particulate chicken feather and carbon black

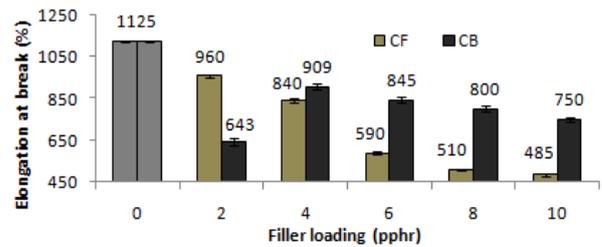


Fig. 3: Elongation at break values for rubber composites reinforced with varying filler loadings of particulate chicken feather and carbon black

**Elongation at Break**

The elongation at break is the strain or percentage increase in the length of the composites when it breaks. The results of the elongation at break for carbon black (CB) reinforced and particulate chicken feather (CF) reinforced natural rubber composites are shown in Fig. 3.

The percentage elongation at break was observed to have decreased from 960 to 485 % as the particulate chicken feather particles increased from 2 – 10 ppH. Similarly, the percentage elongation at break for carbon black first increased from 643 to 909 % (2 - 4 ppH) and subsequently decreased from 909 to 750 % (4 -10 ppH). A decrease in elongation at break as filler loading increases is explained in terms of adherence of the filler to the rubber polymer matrix leading to the stiffening of the polymer chain and hence resistance to stretch when strain is applied (Ekebafe *et al.*, 2010). Particulate chicken feather (CF) reinforced the rubber matrix better than carbon black (CB) at 2 ppH only with regards to elongation at break.

**Modulus of elasticity**

The modulus of elasticity is a measure of stiffness of an elastic material. Fig. 4 shows the results of modulus at 100% elasticity (M100) of the carbon black (CB) reinforced and particulate chicken feather (CF) reinforced natural rubber composites.

Generally, the filled rubber composites gave higher modulus than the neat rubber (0 ppH). An increase in modulus was attributed to the restriction of the rubber molecular chains mobility when fillers were added. Carbon black filled rubber composites exhibited an irregular trend when their loadings were increased. This behaviour was ascribed to the orientation pattern of the particles within the vulcanized rubber matrix. Whereas, particulate chicken feather filled rubber composites increased in modulus as filler loading increased. This was the same effect reported by Barone and Schmidt, (2005) where feathers was seen to increase the modulus of the composite. Particulate chicken feather (CF) reinforced the rubber matrix better than carbon black (CB) at 8 and 10 ppH only with regards to modulus 100% (M100).

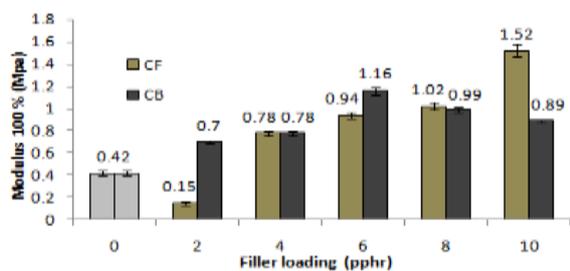


Fig. 4: Modulus 100% values for rubber composites reinforced with varying filler loadings of particulate chicken feather and carbon black

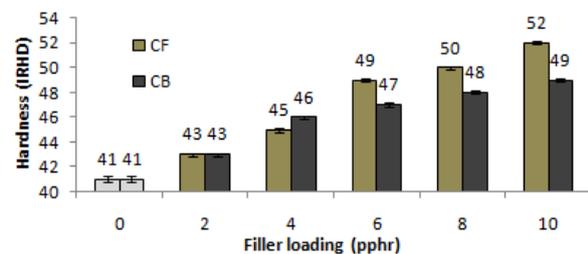


Fig. 5: Hardness values for rubber composites reinforced with varying filler loadings of particulate chicken feather and carbon black

**Shore A Hardness**

The hardness of rubber measures its resistance to surface indentation. Rubber hardness was measured on Shore A Durometer with calibration scale of 0 to 100 (IRHD). Generally, increase in particulate fillers (reinforcing or non-reinforcing) loading on rubber matrix generally leads to increase in the hardness of the resultant composites due to their ability to reduce rubber chain elasticity (Gumel *et al.*, 2013). The extent of hardness of the filler in a matrix is dependent on its stiffness and volume/ loading (Egwaikhide *et al.*, 2007). The results of hardness for carbon black (CB) and particulate chicken feather (CF) reinforced natural rubber composites are shown in Fig. 5.

From Fig. 5, addition of both fillers to the rubber matrix increased its hardness as explained by Gumel *et al.* (2013). The inclusion of particulate fillers into rubber matrix makes the rubber composite to become denser and rigid owing to the reduction of rubber chain elasticity. Increase in hardness results from the hindered rubber chain mobility by the fillers which stiffened the vulcanizates (Motawie *et al.*, 2016). However, carbon black yielded harder rubber vulcanizates at 6, 8 and 10 ppH. This was attributed to the stronger bonds due to surface reactivity of carbon in rubber matrix (Vineetkumar, 2008). Particulate chicken feather (CF) reinforced the rubber matrix better than carbon black (CB) at 4ppH only with regards to hardness.

**Wear test**

The wear is the extent to which a sample is prone to mechanical action such as rubbing, scraping or erosion that gradually removes material from its surface expressed in percentage. The wear percent for carbon black (CB) and particulate chicken feather (CF) reinforced natural rubber composites are shown in Fig. 6.

Wear in natural rubber composite products result in the products' reduction in its usefulness and economic value (Verma *et al.*, 2012). From Fig. 6, the wear percent of the neat rubber vulcanizate generally reduced on addition of both fillers. This was attributed to good dispersion and interfacial adhesion of particles in the matrix as explained by Aguele *et al.* (2014). Particulate chicken feather (CF) reinforced the rubber matrix better than carbon black (CB) at 6, 8 and 10 ppH with regards to wear.

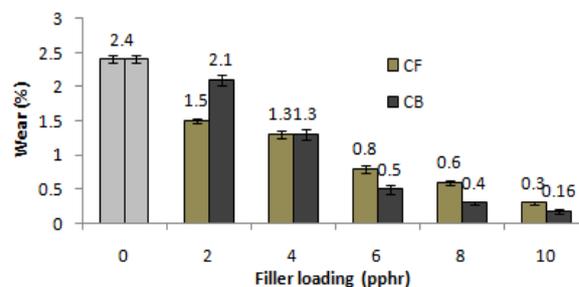


Fig. 6: Wear percent for rubber composites reinforced with varying filler loadings of particulate chicken feather and carbon black

Table 2: TGA and DTG data for unfilled and filled natural rubber nanocomposites

Sample	T5% (°C)	T50% (°C)	Thdr (°C)	Residue (%)
Unfilled NR	200	352	335	3.73
CF (10 ppH)	318	426	395	15.55
CB (10 ppH)	279	328	390	8.77

**Thermal gravimetric analysis (TGA)**

Thermogravimetric Analysis (TGA) and TGA derivative (DTG) profiles are often useful in providing information with regards to decomposition temperatures and in the assessment of the thermal stability of rubber composites (Aleksandra *et al.*, 2014). The TGA and DTG data for carbon black (CB) and particulate chicken feather (CF) reinforced natural rubber composites are shown on Table 2.

The residue left which is the fixed mass at increasing temperature correlates to the inorganic matter present in the nanocomposite. From Table 2, TGA data at 5% (T5%), 50% (T50%) and highest decomposition rate (Thdr) showed higher temperature values for both fillers than neat rubber vulcanizates. This is similar to the case with cellulose particles of all types as reported by Amoke *et al.* (2017). However, particulate chicken feather (CF) gave higher values than carbon black (CB) which corroborates better thermal stability. This was explained by the insulating property of chicken feathers brought about by their inherent hollow structure. Works by Méndez-Hernández *et al.* (2018) and Amieva *et al.* (2015) recorded similar results of better thermal stability for elastomer reinforced with chicken feathers. Also, from the Table 2, the higher residue of 15.55 g for particulate chicken feather (CF) as against 8.77 g for carbon black (CB) reinforced rubber vulcanizates further supports its improvement in thermal stability. Generally, particulate chicken feather (CF) reinforced rubber vulcanizates were thermally more stable than carbon black (CB) reinforced rubber vulcanizates.

**Conclusion**

Application of feathers as reinforcement contributes to value and waste management of the resource. The top-down method was sufficient for the size reduction of feathers to micro – nanoscale as revealed by Dynamic Light Scattering Analysis. Reducing feather fibres to particles proved to improve several physicochemical and thermal properties of natural rubber composites and adequately substitute carbon black as filler in rubber vulcanizates. Suggested applications of the developed feather reinforced rubber vulcanizates are in areas where high thermal stability and moderate strength, elongation, hardness and abrasion resistance are required such as in car brake pads and protective footwear.

**Recommendations**

Surface modification techniques for the particulate feather particles should be further investigated for improved reinforcement abilities to match and possibly outperform

carbon black in natural rubber composites. Other properties should also be analyzed to determine other application areas.

#### Conflict of Interest

Authors have declared that there is no conflict of interest reported in this work.

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