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MORPHOLOGICAL AND MECHANICAL PROPERTIES OF PALM KERNEL

MESOCARP FILLED NATURAL RUBBER

*¹Kevin Shegun Otoikhian and ²Ayo Mark Dada

¹Chemical Engineering Department, Faculty of Engineering, Edo University, Iyamho, Iyamho-Uzairue, Edo State, Nigeria

²Polymer Engineering Department, School of Engineering, Auchi Polytechnic, Auchi, Edo State, Nigeria.

*Corresponding Author: Otoikhian.kevin@edouniversity.edu.ng. https://orcid.org/0000-0001-

5736-8584

ABSTRACT

Morphological and mechanical properties of natural rubber filled with palm kernel mesocarp (PKM) were succinctly investigated in this study. Samples of palm kernel mesocarp were washed, dried, ball milled to fine particle size and used in compounding natural rubber with filler loading ranging from 5 to 30 parts per hundred (pph). Properties investigated were tensile strength, modulus, and elongation at break, hardness, abrasion resistance and compression set. The results revealed that the tensile strength increased from 13.68 to $25.43 N/mm^2$, modulus, from 5.26 to $12.00 N/mm^2$, hardness, from 45.16 to 71.06 Shore A with increase in the filler loadings, while elongation at break, compression set, and abrasion resistance decreased from 633.54 to 441.02 %, 51.48 to 29.03 % and 47.09 to $24.37 mm^2/rev$ respectively with filler loadings from 5 to 30 pph. The scanning electron microscope (SEM) micrographs revealed that the white PKM powdered particles were well dispersed within Natural Rubber (NR) polymer matrix (gray). At higher filler loadings, there was noticeable compaction within the matrices interface, thus contributing to the improved properties The mechanical properties shown by the palm kernel mesocarp filled natural rubber compound demonstrated its compatibility in polymer products manufacture such as oil seal, conveyor belts, tyres, foam, tubes and balloons.

Keywords: Filler, mesocarp, micrograph, palm kernel, rubber, tyres.

INTRODUCTION

Natural rubber is an elastic substance obtained from the latex sap of trees, especially those trees which belong to the genera, Hevea and Ficus. Technically, natural rubber is an elastomer or an

elastic hydrocarbon polymer. It is a vital renewable agricultural resource or commodity that is commercially and widely accepted due to its outstanding properties suitable in the manufacture of a wide range of products [1-2]. Natural rubber on its own does not possess the necessary hardness and modulus required for commercial acceptability. The incorporation of certain compounding ingredient increases these characteristics to the level desired for natural rubber demand [2-4] and enable its use in the manufacture of important commodities such as conveyor belts, tyres, foam, tubes, etc. One of these compounding ingredients that give the natural rubber its outstanding properties is the filler [5-6]. Majority of conventional fillers used in the rubber industries today are silica and carbon black whose relative modulus are relatively low [7-8]. Rubber plays a major role in the socio-economic fabric of many developing countries. Palm Kernel is a tropical tree crop of the tropical rain forest [9]. For optimal growth it requires stable climatic conditions, in particular, with respect to light and moisture supply [10-11].

This study takes an insight into the morphological and mechanical properties of palm kernel mesocarp filled natural rubber. The lignocellulose filler with high strength and modulus properties is suitable for manufacturing composite that reinforce elastomer by increasing the number of chains [12] that shears the load of a broken polymer chain.

EXPERIMENTAL

The materials and equipment used in this study include the following: Natural Rubber (NSR-10) sourced from Rubber Research Institute, Iyanomor; Palm Kernel Filler, sourced from Owan-East Local Government Area of Edo State. Chemical Reagents, such as Mercarptobenzothiazolesulphenamide (MBTS), stearic acid, sulphur, Trimetylquinoline (TMQ), all of analytical grade from Sigma Andriech, Germany, and zinc oxide sourced from the British Drug House (BDH), England.

Some of the equipment used are Monsanto Tensile Tester Model 1/m, manufactured by the British Company Limited, England; Wallace Hardness Tester Model c8007/25 (Elektron Technology Series, UK); Wallace Akron Abrasion Tester (Elektron Technology Series, UK); DuPont Machine, Manufactured by British Company Limited, England; Muffle Furnace METT m-525 (Elektron Technology Series, UK); Two Roll Mill, Manufactured by the British Company Limited, England; and Hydraulic Press (Elektron Technology Series, UK).

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Preparation of Palm Kernel Filler

The mesocarp of the kernel was ground with a milling machine and it was later dissolved in water at a temperature of 100°C. When the mixture was boiling, the particles (filler) were filtered out of the mixture, the kernel was washed and dried using oven. After drying, it was ball milled to reduce the particle size of the filler to mesh of 75 mm.

Characterization of Filler: The fillers were characterized in terms of bulk density, pH and particle size.

Determination of Bulk Density: Bulk density of the various samples was determined by the tapping procedure [13-14]. Accurately weighed samples were poured into a uniform cylinder and were then tapped several times until there was no change in the volume occupied. This volume was then recorded and the bulk density calculated.

Determination of pH: The pH of the palm kernel filler samples was determined using ASTMD 1512 method [13] by immersing 1.0 g samples in 10.0 ml of distilled water in a 15 ml beaker. The mixture was stirred for 15 minutes and the probe of the pH meter was then inserted into the solution to obtain reading directly from the test. It showed that the filler was acidic.

Determination of Particle Size: A sieve was used to determine the particle size of the palm kernel filler samples and was recorded.

Processing of the Composites: This involved the compounding of natural rubber with palm kernel fillers in accordance to the formulation given in Table 1

Ingredients	Parts per hundred (pph) Rubber
Natural Rubber	100
Fillers(palm-kernel)	Variables (5, 10, 15, 20, 25, 30)
Zinc oxide	5.0
Satiric acid	1.5
Sulphur	1.5
MBTS	1.0
TMQ	1.0
Processing oil	5.0

TABLE 1: Formulations for Compounding Nature

A batch factor of two (2) was used

Mixing: The rubber mixes were prepared on a laboratory size two roll mill maintained at 70°C to avoid cross-linking during mixing, after which the rubber compound was stretched out. *Curing:* Curing of the vulcanizates was carried out at 115°C for 8 mins.

Mechanical Properties of the Vulcanizates: The mechanical properties of the vulcanizates were determined using standard rest procedures:

Tensile Property Tests: Tensile properties were determined on a universal tensile tester at a cross speed of $500 \, mm/min$., using dumbbell test piece of dimension $100 \times 20 \times 5 \, mm$. Test samples were tested in the machines giving straight tensile pull, without any bending or twisting. The machine measures both the tensile stress and the tensile strain. The tensile stress is the strength of pull in the area between the notch marks. It is based on original cross sectional area. The tensile strain is a measure of how the test sample has been stretched by the ball.

Hardness Test: Hardness of the sample was determined by adopting the standard dead load method. The standard dead method of measurement covers rubber in the range of 30 to 85 International Rubber Hardness Degrees (IRHD). The test was carried out using the Wallace hardness tester.

Compression Set Test: Compression set evaluates the extent by which the specimen fails to return to its original thickness when subjected to standard compression load for a given period of time at a given temperature [15]. The test samples were cut to standard dimension and compress between parallel steel plates under stress of 2.8 mpa. It was then conditioned for 24 hrs at 70 °C after which the sample was removed and cooled for a period of 30 mins.

Compression Set (%) =
$$\frac{t_0 - t_r}{t_0} \times 100$$

Where, t_0 is the initial thickness and t_r is the recovered thickness of the sample.

Abrasion Resistance: The abrasion was carried out for per 1000 evolution per 1000 and material loss for each run was noted. Wallace Akron Abrasion test was used. The angle between the test sample and the wheel was adjusted to an angle of 15° . The specimen was re-weighted between each test run. The mean of the four revolution of the abrasive wheel was calculated.

 $A brasion \ Resistance = \frac{W eightless \ of \ the \ standard}{W eight \ loss \ of \ the \ sample} \times 100$

Morphological Properties of Composite

Micro-Structure Analysis: Specimen samples, usually nonconductive, were made conductive by introducing about 5 nm gold onto it and were cut into specified dimension using a sputter cutting machine. The samples were place on the column of the Scanning Electron Microscope where the image were focused using navigation camera and transferred to electron mode in accordance to the desired magnification.

RESULTS AND DISCUSSION

Filler Loading (pph) Property (Unit) 5 10 15 20 25 30 Tensile strength(Nmm2) 19.38 24.95 25.03 13.68 21.62 25.43 Tensile modulus (Nmm2) 5.26 8.56 10.50 11.73 11.99 12.00 Elongation at Break (%) 602.20 547.52 518.18 480.09 633.5 441.02 Hardness (Shore A) 54.09 59.04 63.90 67.10 45.16 71.06 Abrasion resistance (mm2rev) 47.09 40.29 37.16 29.41 27.84 24.37 47.42 41.35 Compression Set (%) 51.48 35.00 32.18 29.03

Table 2: Mechanical Test Results











Figure 3: Effects of Elongation Break on Vulcanizate with different Filler Loadings



Figure 4: Effects of Hardness on Vulcanizate with different Filler Loadings



Figure 5: Effects of Abrasion Resistance on Vulcanizate with different Filler Loadings



Figure 6: Effects of Compression Set on Vulcanizate with different Filler Loadings

The reinforcement properties of the rubber composite filled with kernel mesocarp showed a significant rise in the properties of the composite. This observation is noticeable in the results displayed from the various physical tests as shown. In Table 2 and Figures 1-6 above, it was observed that filler loading affects the physical properties of the composition.

The tensile strength increased with increasing filler loading. The result of tensile modulus as presented in Table 2 and Figure 1 shows that a rise in filler loading leads to an increase in the tensile modulus. This was attributed to the fact that there is an excellent adhesion between the filler and the rubber matrix [16]. For elongation at break, the result showed a decreasing trend as filler loading increased. This was because the rubber became stiff as the filler loading increased.

The general trend of increasing hardness with increase in filler loading was observed for filler. This is an expected trend attributed to the fact that as more filler are incorporated in the rubber matrix, the composite becomes more rigid owing to the progressive reduction in elasticity [17]. For abrasion, as the filler loading increases there is a decrease in abrasion resistance of the vulcanizates. This is to say that filler loading does not increase the abrasion resistance index of the vulcanizate. As a filler loading is increased, adhesion strength between the polymer and filler falls. This could partly account for the behaviour of the abrasion property. For compression set as presented in Table 2 and Figure 6, it was observed to take a falling trend as the filler loading increased. As more filler was introduced into the rubber matrix, the more rigid and less susceptible to compression [18]. High values of compression are an indication of less reinforcement.

Morphological Properties Results



Figure 1: Micro-structure of unfilled natural rubber



Figure 2: Micro-structure of PKM (10 pph) filled natural rubber



Figure 3: Micro-structure of PKM (20 pph) filled natural rubber



Figure 4: Micro-structure of PKM (30 pph) filled natural rubber



Figure 5: Micro-structure of PKM (40 pph) filled natural rubber



Figure 6: Micro-structure of PKM (50 pph) filled natural rubber

The micro-structural result obtained revealed that at 1500X magnification, the plate showed clearly pore areas which were relatively lager than the natural rubber matrices. For both filled systems at different loadings (5- 30 phr), the composites showed irregular surface defects and cracks through the unfilled interactions and compaction within the composite material [5]. The morphologies of the fracture surface for both matrices of palm kernel mesocarp filled NR examined by Scanning electron microscope are presented in Figures 4.7 (a – f). The micrographs revealed that the PKM powdered particles (white) were well dispersed within NR polymer matrix (gray) but at higher filler loadings. Compaction within the matrices interface was

observed, indicating an improved properties in the mechanical properties investigated. Most palm kernel mesocarp-fibres were fractured and only a few fibrils were pulled out from the NR matrix which implies that the adhesion between PKM and NR matrix was good [19-20]. In addition, the interface between PKM and NR polymer matrix was very clear with filler loading at 30 - 50 phr with the fibres forming bundles. However, the fracture surface of PKM/NR composite indicated that the interfacial adhesion was still strong and higher crack propagation energy was required to fracture the composite, thus resulting in better and improved mechanical properties.

CONCLUSION

The main aim of this research was to assess the effect of palm kernel mesocarp on the mechanical and morphological properties of filled natural rubber compound. The use of palm kernel mesocarp powdered filler in NR composites has showed that palm kernel mesocarp filler has a great influence on the mechanical and morphological properties of filled NR matrix composites. The result showed that palm kernel mesocarp can serve as reinforcing filler for NR composite. It indicates that mechanical and morphological properties of composites are greatly influenced by filler compatibility and loadings, seen to be of great importance in ensuring that such polymer products meet specification and service requirements.

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