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PREPARATION, CHARACTERIZATION AND PHYSICO-MECHANICAL PROPERTIES OF NATURAL RUBBER VULCANIZATES FILLED WITH CARBONIZED PALM KERNEL SHELL AND CARBON BLACK.

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Abstract

This research work examined how Carbonized Palm Kernel shell (CPKS) filler affects the mechanical properties of natural rubber vulcanizates. The characterization of the filler was determined before applying it on the NR vulcanizates. The filler loading concentrations of CB/CPKS used were labeled as mixes A to F with the composition of 20:0, 20:5, 15:10, 10:15, 5:20, and 0:20. The result obtained on characterization of the filler were bulk density (0.63g/ml), pH (5.7), % of carbon (51.39%), % of nitrogen (1.09%), moisture content (6.33 %), ash content (7.50%) and particle size (5,6,10 and 16) respectively. The results show significant values for all; the moisture and ash content were within the recommended standard of ASTM ($3\text{-}10_{max}$) and (< or =8) respectively. The physico mechanical results obtained on analysis of the vulcanizates produced showed that hardness and young modulus for majority of the blends increase with higher CPKS composition, while ultimate tensile strength decreases with CPKS composition across the filler loading and abrasion resistance were excellent for CB/CPKS compositions. The absolute CPKS loading had the lowest fatique life as the CPKS filler does not deform during the straining of the composites. The value of elongation at break for CB filled vulcanizates were found to be slightly better than CPKS arising from likely partial removal of lignin and hemicellulose of the natural filler through carbonization at 600^0C. This may have induced low resistance to stretching during tensile deformation and improved the elongation at break of the composites. This research showed that CPKS has excellent filler potential as indicated by physico mechanical properties of the vulcanizates.

Keywords: Vulcanizates, Natural Rubber, Physicochemical and Physico-Mechanical Properties, Carbonized Palm Kernel Shell, Carbon Black, Filler.

Introduction

Natural rubber (NR) is one of the main elastomers widely used to prepare many rubber-based products such as tyres, belts, hoses etc. It is not amazing that more than 50% of all chemists and chemical engineers, large number of mechanical engineers, physicists, polymer scientist, technologists, material scientists and textile technologists are involved in research or developmental work with polymers (Ayo et al, 2010). Natural rubber is a renewable agricultural resource that does not naturally possess the necessary hardness and modulus required for its commercial acceptability (Akinlabi *et al.*, 2005). Natural rubbers are frequently reinforced by assimilation of fillers to improve its mechanical properties like: tensile strength, modulus, tear strength, elongation at break, hardness, compression set, rebound resilience and abrasion resistance (Frohlich *et al.*, 2005). The additives which are added to enhance the process ability and properties of rubber vulcanizates are usually sourced from combinations of any of the followings; accelerators, activators, fillers, antioxidants and vulcanizing agents. Fillers play an important function in NR compounding by modifying the physical properties of base polymer. Carbon black and silica are commonly used (Salaech et al, 2012). Fillers are added to rubber either to extend or cheapen the rubber compound, or to add desirable qualities to the final compound and enhance the products service qualities. In rubber industry, fillers that are commonly in use are carbon black, China clay and calcium carbonate while recently nano fillers or nano materials are also used. There is a significant use of carbon black as a reinforcing material and additive in the fields of elastomers and materials manufacture. Over 11 million tonnes of carbon black are used every year in tyre manufacture alone in the world (Kühner and Voll, 1993). The carbon filler in rubber can be over 30% of the material by weight (Kühner and Voll, 1993). The key property of carbon black fillers is the ability to bond with the elastomer component, hence providing mechanical strength as well as imparting durability to the materials. The production process of carbon black requires a partial combustion of fossil-origin hydrocarbon fuels (such as heavy oil, coal tar and natural gas etc.) under reduced oxygen conditions. The production is not only energy intensive, but also unsustainable in the long-term as the source is exhaustible. Due to the increasing demand for non-renewable materials in industrial and manufacturing processes coupled with the need to reduce the release of fossil-origin carbon dioxide and the widespread recognition that the adoption of the circular economy model is important for the long-term development of the society. There is a major need to search and develop the potential of alternatives and waste materials to be used for NR compounding. The use of bio-based fillers would further help to reduce the dependence upon fossil fuel and move towards a sustainable material for rubber filler production. Previous works have successfully recovered carbon black from end-of-life tyres and reapplied as a new type of rubber filler and related materials. In this study, Carbonized Palm Kernel Shell (CPKS) is being considered as alternative feedstock for carbon black-like materials as reinforcing filler in rubber. It is also widely known that palm kernel shell can be used as a bio-fuel and is also a commercial source of charcoal and activated carbon. The activated carbon made from palm kernel shell is considered to be superior to those obtained from other feedstock due to its highly microporous and complementary mesoporous structure. This study has focused on the development of novel carbonaceous rubber fillers produced by the pyrolysis of palm kernel shell. The aim of this work is to study physico-mechanical properties of carbonized palm kernel shell blended with carbon black filled natural rubber vulcanizates while the specific objective is to produce carbon-based fillers from feedstock through the application of conventional pyrolysis to convert into char fillers and further carry out proximate analysis.

Materials and Methods

Apparatus and Equipment

The equipment and apparatus used for this study include: weighing balance RS232, model WT2203GH, Saumya Two roll mill (DTRM-50) for compounding rubber, Saumya Compression moulding machine 50 TONS (PID528) for vulcanization, Saumya Universal tensile machine (UTM192-2L) for testing tensile properties, Rex durometer (OS-2H) for testing hardness, Din abrasion tester (FE05000) for testing wear resistance, 250ml reagent bottle, Stop Watch: 31305 model, Carbolite furnace, model Cw 1100, Desiccator.Product number-Z553808, Oven, model DHG – 9101, Measuring cylinder: SPG1000 mL graduated, Thermometer .Made in Nigeria.

Methods

Carbonization

Palm Kernel Shells (PKS) were obtained from Apomu, Osun State and washed to remove accompanying dirt, thereafter, sun dried for 2 days. The PKS was pulverized to particulate size, weighed and recorded. Carbonization was done using a modified method of Emmanuel *et al*, (2017). The dried sample was then carbonized for 1 hour at 500-600^{0}C using the muffle furnace. The sample was removed from the furnace and placed into a bowl containing water for quenching and allowed to cool. Then, the shell was drained, dried, weighed and recorded.

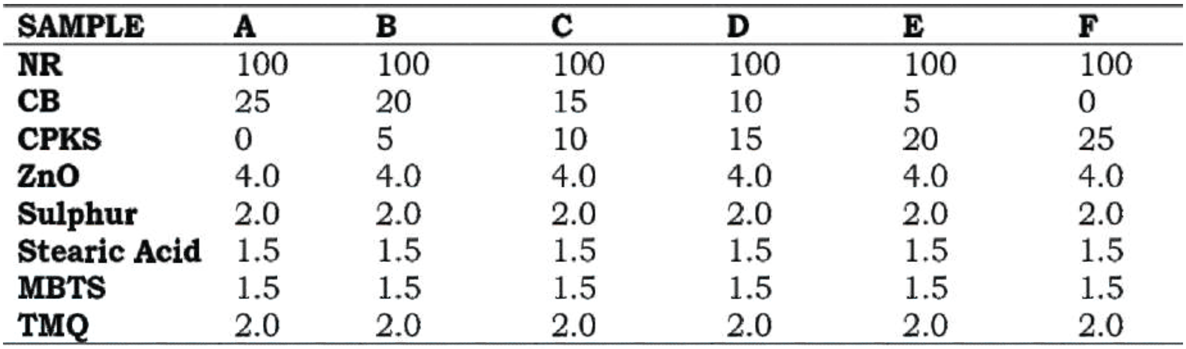
Chemical Activation

The carbonized palm kernel shell (CPKS) particle was activated using a modified method of Emmanuel *et al*, (2017).The sample was soaked in H_3PO_4 (0.1M) for 24 hours. The activated carbon is dried in oven to obtain the initial mass recorded. The activated sample is then washed with distill water and KOH (0.1M) to neutralize the material being activated to pH 7 and finally sun dried for 2-3 hours followed by oven drying for 1-2 hours at about 170^{0}C. The activated particle is weighed and recorded.

Preparation of Recipe

The formulation used for the rubbers compounding are presented table 1below. All measurements were carried out in part per hundred of rubber (Phr).

Table 1: Recipe for Carbon Black/CPKS Filled NR Vulcanizates



SAMPLE	A	B	C	D	E	F
NR	100	100	100	100	100	100
CB	25	20	15	10	5	0
CPKS	0	5	10	15	20	25
ZnO	4.0	4.0	4.0	4.0	4.0	4.0
Sulphur	2.0	2.0	2.0	2.0	2.0	2.0
Stearic Acid	1.5	1.5	1.5	1.5	1.5	1.5
MBTS	1.5	1.5	1.5	1.5	1.5	1.5
TMQ	2.0	2.0	2.0	2.0	2.0	2.0

NR = Natural Rubber, CB = Carbon black, MBTS = 2, 2'-Dithiobisbenzothiazole, TMQ = 1,2-Dihydro-2,2,4-trimethylquinoline

Compounding and Mastication Process

The compounding of the polymer was carried out using the two-roll-mill (DTRM-150). The mastication of the rubber was carried out first before the compounding where the rubber was milled continuously to make it more elastic and softer for easy incorporation of ingredients and shaping process.

Characterization of Carbonized Palm Kernel Shell Powder (CPKS).

pH

The pH of the Palm Kernel Shell powder was determined in accordance with ASTMD 1512-05, (2012) method by immersing 1.0g sample in 20.0cm^3 of water in a beaker. The mixture was stirred for 15minutes and the pH meter was then inserted into the mixture to obtain the readings directly.

Particle Size

The Palm Kernel Shell powder was sieved to determine particle size of CPKS using different sieve mesh sizes. This is characterized using standard ASTM method.

Bulk Density

The Bulk density was determined by the tapping procedure described by (Ahmedna *et al*, 1997). **Moisture Content**

The moisture content of the sample was determined by using the method prescribed by ASTM D 1509 (1995).

Ash Content

The ash content of the sample was determined by using the method prescribed by ASTM Standard D1506-99 (2013).

%Carbon

The % C content was evaluated as follows

$$\% \text{ of Carbon} = \frac{100 - \% \text{ Ash}}{1.80.}$$

Characterization of Vulcanizate Properties

Tensile Properties

The tensile mechanical properties of the vulcanizate was carried out using Saumya universal tensile machine (UTM192-2L model) which determined the stress-strain behavior of the blends (Malomo *et al*, 2019).

Young Modulus

This was obtained as the slope of the stress-strain graph of the various samples (Raji, 2015).

Ultimate Tensile Strength

The ultimate tensile strength was calculated by dividing the maximum load carried on the specimen by the original cross-sectional area of the in mm^2 (Raji, 2015)

Hardness

This was done using a rex durometer (OS-2H). The sample was placed on a metallic base with the indentor pin of the durometer very close to it. The load of the durometer was pressed downwards so that the indentor pin could penetrate the sample. The measure of the resistance of the sample to indentation was observed on the display screen and the value was recorded. This was done thrice per sample and the average value taken (Raji, 2015).

Rubber Fatique

Rubber fatigue test was carried out using rubber fatique tester of model ZME-7003 (oscillation of 2000) using a method of ASTM Committee E0.06 (2013).

Abrasion Resistance

The ASTM D1650 was used in determining the ability of the material to resist wear when in contact with abrasive surface.

Physicochemical Properties of Carbonized Palm Kernel Shell

The moisture content, ash content, pH, bulk density, % of carbon and % of nitrogen are the physicochemical properties carried out on carbonized Palm Kernel Shell. The values obtained for moisture content of carbonized palm kernel shell (6.33 %) were within the recommended standard of ASTM ($3\text{-}10_{max}$), Table 2. The values recorded in this study indicated that CPKS had adsorption capacity within the material due to the fact that higher moisture content tends to favour higher or greater adsorption capacity. The result of this study is similar to value of 4% obtained for CPKS by Emmanuel *et al,* (2017) thereby making CPKS possess high adsorption capacity since high moisture content enhances high adsorption (Balakrishnan and

Results and Discussion

Table 2: Physicochemical properties of Carbonized Palm Kernel Shell

	Moisture (%)	Ash (%)	Bulk density (g/ml)	Carbon (%)	Nitrogen (%)	pH	Particle size (mm)
CPKS	6.33	7.50	0.63	51.39	1.09	5.7	4.00,3.35, 2.00, 1.18
ASTM Standard	3-10 Max	< or = 8 Max	0.36-0.74	-	-	-	5-50

CPKS – Carbonized Palm Kernel Shell

Satyawali, 2007). The ash content of 7.50% was recorded for carbonized Palm Kernel Shells and was within the ASTM required standard (< or =8). Bulk density reflects the filler's ability to function for structure support. The bulk density of 0.63g/ml was obtained for carbonized palm kernel shell which is within the standard required by regulatory body (ASTM 0.36-0.74). The result of this sample in the present study showed an increase implying greater mechanical strength thereby improving dimensional stability (Balakrishnan and Satyawali, 2007). The particle size obtained for CPKS were 4.00, 3.35, 2.00 and 1.18mm of sieve opening. The values correspond to 5,6,10 and 16 sieves number respectively. The results obtained were within the standard value of 5-50 sieve size (Table 2). Smaller particles sizes have the ability to wet rubber surface more and therefore greater reinforcement. The density is influenced by the particle size and structure of the fibre and the lower the particle size, the lower the density and therefore the better the filler-matrix interaction (Momoh *et al*, 2017).

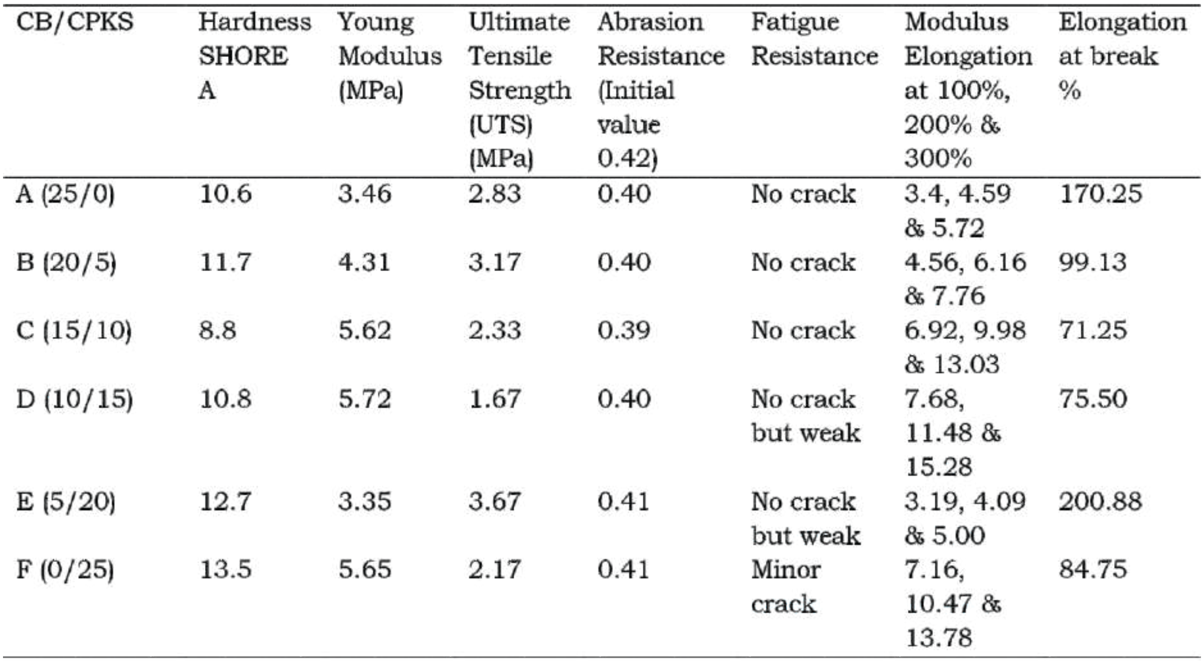
The carbonized palm kernel shells had nitrogen percentage of 1.09%, Table 2. This result is due to carbonization process at 600°C. This value of 1.09% was found to be much higher than that of Ndubuisi *et al*, (2016) who reported nitrogen content of 0.48% for palm kernel shell. The two ranges will however promote filler-matrix interactions. The percentage carbon content of 51.39 was obtained for carbonized palm kernel shell, Table 2. The higher values recorded were as a result of release of volatile matter in the samples and this showed that CPKS possesses good precursor in material reinforcement and this was in line with report of Andas *et al*, (2017) that the higher the value of carbon content the greater will be the reinforcement effect of the filler. The acidic pH 5.7 was recorded in this study for carbonized palm kernel shells, Table 2. The pH of the samples indicated acidic properties which were ascribed to presence of soluble acid radicals in the filler. Meanwhile, acidity many not enhance the cure time of vulcalnizates compounded because acidity of the filler tends to slow the cure rate and hence reduce the crosslink density (Malomo *et al*, 2018).

Hardness

The result recorded for hardness in varying carbon black/carbonized palm kernel shell filled vulcanizes for various compositions (A25/0, B20/5, C15/10, D10/15, E5/20, and F0/25) for carbon black/carbonized palm kernel shell are 10.6, 11.7, 8.8, 10.8, 12.7 and 13.5 respectively as shown on Table 3 and Figure 1. The hardness of a particular materials is the measurement of materials resistance to indentation. This hardness can be improved with fillers; this is as a result of

Mechanical Properties of Carbon Black/Carbonized Palm Kernel Shell Filled NR Vulcarizated

Table 3: **Mechanical Properties of the Carbon Black/Carbonized Palm Kernel Shell (CB/CPKS) filled Vulcanizates**



CB/CPKS	Hardness SHORE A	Young Modulus (MPa)	Ultimate Tensile Strength (UTS) (MPa)	Abrasion Resistance (Initial value 0.42)	Fatigue Resistance	Modulus Elongation at 100%, 200% & 300%	Elongation at break %
A (25/0)	10.6	3.46	2.83	0.40	No crack	3.4, 4.59 & 5.72	170.25
B (20/5)	11.7	4.31	3.17	0.40	No crack	4.56, 6.16 & 7.76	99.13
C (15/10)	8.8	5.62	2.33	0.39	No crack	6.92, 9.98 & 13.03	71.25
D (10/15)	10.8	5.72	1.67	0.40	No crack but weak	7.68, 11.48 & 15.28	75.50
E (5/20)	12.7	3.35	3.67	0.41	No crack but weak	3.19, 4.09 & 5.00	200.88
F (0/25)	13.5	5.65	2.17	0.41	Minor crack	7.16, 10.47 & 13.78	84.75

reinforcing properties of fillers through filler-material interactions (Mwaikambo and Ansell, 2001). The present results show that the hardness for majority of the blends increases with higher CPKS composition; this indicates an improvement in resistance to indentation. However, the values of hardness decreased as the CPKS composition decreases from sample F to C and then increased again at sample B before slight decrease at sample A. The blend (Sample F) having only NR and CPKS was observed to have the highest value of hardness (13.7) across A-F composition and higher value when compared to sample A (10.6) consist of only NR and CB. Sample C and D having almost the same composition of CB to CPKS had an intermediate hardness across the six blends. With the values obtained, it shows that CPKS possesses better reinforcement and strength imparting properties than CB. During preparation of CPKS, some organic matter such as wax, adhesive pectin and lignin were oxidized from the fillers (PKS). The observation in the present study is similar to the work of Mwaikambo and Ansell, (2001) where the removal of these constituents in the material filler changed the physicochemical properties of the cellulose. The PKS went through pulverization and carbonization which increased the surface area of the material.

Young Modulus

Young modulus is the resistance to deformation of the materials. In this study, the carbon black/carbonized palm kernel shells had values of 3.46, 4.31, 5.62, 5.72, 3.35 and 5.65MPa Table 3 and figure 2 for samples 25/0, 20/5, 15/10, 10/15, 5/20, 0/25. The effect of CPKS also reflected, as the value of modulus for the blend with sample D (CB/CPKS, 10/15) having the highest value modulus of (5.72MPa) followed by sample F

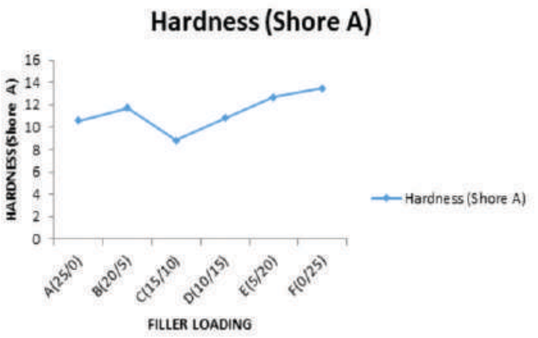


Fig 1: Graph of Hardness against Filler Loading Vulcanizates.

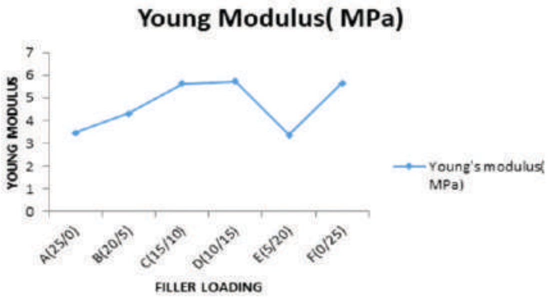


Fig 2: Graph of Young Modulus against Filler Loading Vulcanizates.

(CB/CPKS, 0/15) having the highest CPKS composition across the blends with value modulus of 5.65MPa. The value of modulus for sample C (CB/CPKS, 15/10) with 5.62MPa was close to that of sample D and F.

Ultimate Tensile Strength (UTS)

The carbon black/carbonized palm kernel shell had ultimate tensile strength of 2.83, 3.17, 2.33, 1.67, 3.67 and 2.17MPa for sample 25/0, 20/5, 15/10, 10/15, 5/20, 0/25 The results obtained show decrease in CPKS composition across sample A-F (Table 3 and Figure 3). This indicates that majority of the blends having higher CPKS to CB composition ratio showed low values of UTS. The value obtained for UTS decreases as the CPKS composition increases from sample B to sample D and then increases again at sample C before reducing again at sample F. Sample E with the composition ratio of (5CB: 20CPKS) recorded the highest Value of 3.67MPa across the blend A to F. By comparing samples, A and F (CB25, CPKS 0 And CBO, CPKS 25), it was observed that sample A produced the highest value of 2.83MPa with sample F giving 2.17MPa. Sample B, C and D with comparatively higher values of CPKS recorded low UTS. This enhancement in UTS of CB filled rubber vulcanizate is due to the fact that when carbon black filler enters the rubber matrix; the elasticity of the rubber chain is reduced resulting to a more rigid vulcanizate thereby increasing the UTS. This reinforcement effect comes from the filler and filler rubber interactions comprising chemical and physical types. The reinforcing property of CB in this study is further confirmed by the work of Frohlich *et al*, (2005).

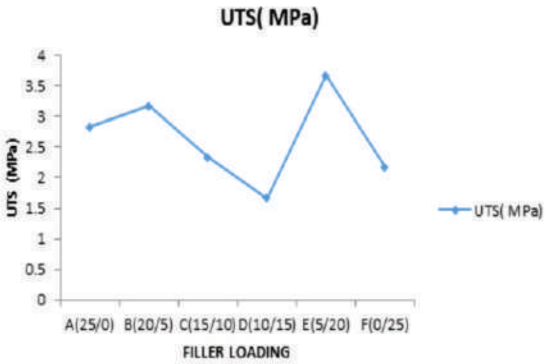


Fig 3: Graph of Ultimate Tensile Strength against Filler Loading Vulcanizates.

Abrasion Resistance

The result of abrasion resistance of the vulcanizates is presented in Table 3. The measure of the resistance of a material to scrapping, rubbing or scratching is known as the material's abrasion resistance. The result presented in table 3 and Figure 4 shows that blend across sample A to F virtually showed 95.24% resistance to wear. The highest abrasion resistance being 97.62% (0.1 wear) for sample E and F. The results obtained for abrasion resistance for all the samples were excellent. The results revealed the production of well compounded and blended polymer chain indicating compatibility of the filler with the base polymer.

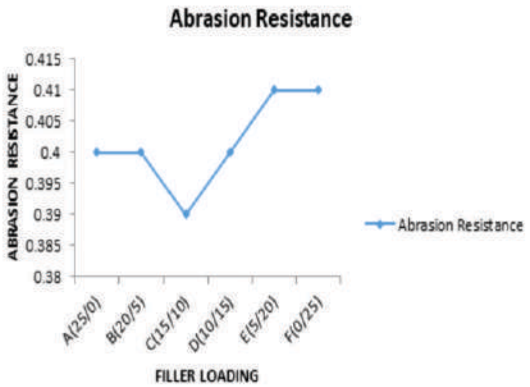


Fig 4: Graph of Abrasion Resistance against Filler Loading Vulcanizates.

Fatique Resistance

The fatique resistance of the vulcanizates is presented in the Table 3. The results showed excellent resistance to fatique for most samples except at higher loadings of CPKS where minor cracks and weaknesses in vulcanizate structure were observed. It was observed that the vulcanizates were slightly prone to fatique as the CB content decreased across the blends. For instance, (25CB, 0CPKS), (20CB, 5CPKS) and (15CB, 10CPKS) blends show complete fatique resistance, on the other hand at (10CB, 15CPKS) and (5CB, 20CPKS) blends vulcanizate were slightly prone to fatique and weakness. Similar observations were recorded at the 0CB, 25CPKS filler loading thereby showing that CB had improved resistance to fatique characteristics compared to CPKS. According to Payne *et al,* (1972) the composite with highest PKS loading had the lowest fatique life as the PKS filler does not deform during the straining of the composites.

Modulus at 100%, 200% and 300% Elongation

The modulus at 100%, 200% and 300% elongation percentage was presented in the Table 3 and Figure 5 for CB/CPKS blending. The result obtained across A-F recorded an increase in modulus at each percentage (100%, 200% and 300%) as the value of CPKS composition/loading increases. The result indicates the higher loading of CPKS to CB increases modulus at percentage elongations under study. The higher the loading of CPKS filler, the greater the modulus. From the result obtained, it suggests that increasing CPKS filler in the vulcanizates was found to enhance the modulus, this implies improved enhancement. Therefore, CPKS composition has better reinforcing properties compared to the CB filler loading. The filler loading composition in the trend being E < A < B < C < F < D (increasing order).

Elongation at break (EB)

The elongation at break (EB) for CB/CPKS vulcanizate are shown in Table 3 and presented in Figure 5. The elongation at break across sample A-F decreased as CPKS to CB filler loading composition increases. It decreases from sample A to sample C and gradually increased at sample D to sample E then reduced at sample F. Sample E (5CB, 25 CPKS) with highest elongation at break of 200% followed by sample A and B (25CB, 0CPKS and 20CB, 5CPKS) with 170.25% and 99.13% respectively. With this result CB has relatively better impact on NR vulcanizate compared to CPKS. The slight advantage of CB over the CPKS here may be as a result of partial removal of lignin and hemicellulose of the natural filler added in the rubber matrix resulting in the low resistance to stretching during tensile deformation, improving the elongation at break of the composites as proved by Dhanalakshmi *et al.* (2015).

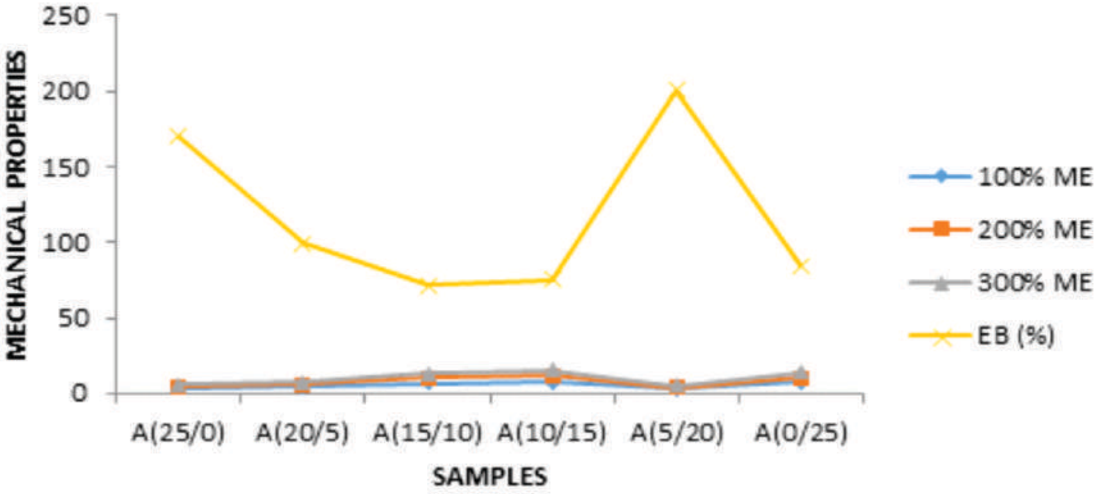


Fig 5: Graph of %Modulus Elongation and Elongation at Break against Filler Loading Vulcanizates.

Conclusion

The research work had examined the physicochemical properties of carbonized palm kernel shell blended with carbon black filled on natural rubber vulcanizates. It was observed that the physicochemical properties were within ASTM recommended standards and the physico-mechanical properties like hardness, young modulus, and ultimate tensile strength were appreciably high with CPKS introduction. The work showed that CPKS has great potential as filler in NR technology.

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