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Studies on the Physicochemical and Physico-Mechanical Properties of Activated Palm Kernel Shell blended with Carbon Black filled NR Vulcanizates.

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Abstract- Palm kernel shell was activated using chemical activation of H_3PO_4 and KOH. Various amounts of activated palm kernel shell (APKS) couple with carbon black (CB) and other conventional ingredients were used to produce natural rubber vulcanizates (NR vulcanizates). The NR vulcanizates were compounded on a two-row mill and tested for its physico-mechanical properties. The results for characterization of physicochemical properties carried out on APKS were ash content (2.06%), moisture content (8.06%), %carbon (54.41%), particle size (4.00, 3.35, 2.00, 1.18mm), bulk density (0.62g/ml) and pH (5.3).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 filler loading concentrations CB/APKS were labeled as mixes 1 to 7. The composition of CB/APKS filler loading ratios were 30:0, 25:5, 20:10, 15:15, 10:20, 5:25, and 0:30 samples 1,2,3,4,5,6 and 7 respectively. Results obtained showed that CB/APKS filled vulcanizates exhibited improvement in the physico-mechanical properties investigated. The results obtained for CB/APKS across the samples filler loading shows that CB composition possess higher UTS, EB and rubber fatigue test while APKS filler loading composition exhibited higher hardness and young modulus. Abrasion resistance was excellent for both CB and APKS filler loading composition.

Keywords- Activated Palm Kernel Shell, filler, carbon black, Chemical Activation, Natural Rubber.

1 INTRODUCTION

Natural Rubber (NR) is a polymer composed of isoprene monomers (2-methyl-1, 3-butadiene). It possesses ability to return to its original shape after being stretched or deformed. The demand for polymers in many applications has experienced a steady growth over the years in the developed countries of Europe, America and Asia. The demand and volume of polymer used is significantly more than ceramic or metals. This has made polymer more readily important and of great economical values. The excellent physical properties of natural rubber especially the high mechanical strength, low heat build-up, excellent flexibility, resistance to impact and tear has made it a material with success and renewability (Daniel *et al*, 2009). Natural Rubbers is 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 reported by Frohlich *et al*., 2005. The applications and usefulness of NR in its raw state may not be good enough. Therefore, there is the need for addition of additives which help to enhance the properties (Ayo *et al*, 2011). The use of filler plays a dominant role in NR vulcanizate and modifies the physical properties of base polymer.

A lot of research is ongoing today in order to find alternatives (local source) to carbon black as a filler. Great advances are being made and the use of biomass of agricultural origin (by-products) such as groundnut shell, rice husks, rubber seed shell etc. Egwakhide *et al*. (2007) investigated the effect of coconut fibre filler on the rheological, physico-mechanical and swelling properties of NR vulcanizates in diesel, kerosene and toluene.

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The results showed that resistance to swelling of NR compound is dependent on amount of filler loading. The higher the filler content, the lower the equilibrium sorption values obtained. Ahmad, (2012) studied the effect of oil palm empty fruit bunch (OPEFB) micro filler on free initiation and propagation in silicone rubber with different weight percentages (wt%) of filler being 0wt% and 1wt% respectively. It was found that OPEFB decreased propagation of electrical treeing development. For tree inception study, the addition of 1wt% of OPEFB increased the tree inception voltage of silicone rubber. Cuttlebone- a biomass was studied as reinforcing filler for NR was also studied by Sirilux *et al*, (2008).

There has been significant shift in industrial societies through recognition of the finite nature of materials and resources. Hence, growing focus on the treatment and disposal of waste material is becoming a priority. A variety of legislation on waste management has been approved by the European Union in recent times. The treatment and disposal of waste materials is becoming a problem of increasing urgency in industrialized societies (McAllister, 2015; Strange, 2002). There is no doubt that the concern about environmental issues is gradually increasing. At the same time, environmental issues have been constantly broadened with concepts, such as sustainable development, which implies not only ecological, but also economic and social responsibilities. In this study, Activated Palm Kernel Shell (APKS) is being considered as alternative feedstock for carbon black-like materials as reinforcing filler in rubber. Palm kernel shell, a cellulosic and lingo cellulosic waste, is available in large quantities. It has potential to be used as a low-cost parent material for rubber fillers. The production of a carbon black substitute from a biodegradable waste would be beneficial as it reduces the demand on non-renewable carbon black feedstock such as fuel oils or natural gas and also helps to combat global warming by converting the biodegradable waste material into solid char, stabilizing the carbon and preventing its release as carbon dioxide (CO_2) or methane (CH_4).

Carbon black is a material which is used mostly as reinforcing filler in rubber and plastic production. The manufacture is energy-consuming, contributes significantly to global CO_2 emissions and uses non-renewable feedstock making it unsustainable. The importation of carbon black is very expensive. It is also a product of petroleum, a source which is non-renewable giving the increasing pressure against the continued use of non-renewable sources of feedstock. The aim of this work is to study physicochemical and physico-mechanical properties of activated palm kernel shell blended with carbon black filled natural rubber vulcanizates while the specific objective is to develop a screening tool to better enable selection of the most suitable materials as carbon black replacement fillers in the rubber system.

2 Materials and Methods

The following equipment were used during the research. The equipment and apparatus used during characterization of physicochemical properties include: 250ml reagent bottle, Stop Watch: model 31305, Thermometer, Desiccator: Product number-Z553808, Oven, model DHG – 910. Flat bottom glass dish, measuring cylinder: SPG1000 mL graduated, weighing balance RS232, model WT2203GH.

The equipment and apparatus used during mechanical properties include: 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, Din abrasion tester (FE05000) for testing wear resistance, Rex durometer (OS-2H) for testing hardness. Rex Guage Company, USA.

2.1 Chemical Activation

The Palm Kernel Shells were obtained from Apomu, Osun State. The shells were washed to remove accompanying dirt and thereafter, sun dried for 2 days. The PKS was pulverized to particulate size, weighed and recorded. Activation was done using a modified method of Emmanuel *et al*, (2017). The shell particle was activated using H_3PO_4 and KOH.

2.2 Preparation Of Recipe For Compounding

The formulation used for compounding in this research is presented in Table 1, measurements were carried out using part per hundred of rubber (Phr).

2.3 Compounding and Mastication

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.

2.4 Characterization of Activated Palm Kernel Shell Powder (APKS).

2.4.1 pH

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

2.4.2 Particle Size

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

2.4.3 Bulk Density

The Bulk density was determined by the tapping procedure described by (Ahmedna *et al*, 1997). Accurately weighed 2.0 g sample was transferred into a cylinder of uniform cross-sectional area and then tapped several times until there was no change in volume occupied by the material. The volume was then recorded and the bulk density calculated as follows:

$$Bulk\ density = \frac{Mass\ of\ Sample}{Volume\ of\ Sample} \quad (1)$$

Table 1. Recipe for CB/APKS Compounding

Sample	A	B	C	D	E	F	G
NR	100	100	100	100	100	100	100
CB	30	25	20	15	10	5	0
APKS	0	5	10	15	20	25	30
ZnO	4.0	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	2.0
Stearic Acid	1.5	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	1.5
TMQ	2.0	2.0	2.0	2.0	2.0	2.0	2.0

NR = Natural Rubber CB = Carbon black APKS = Activated Palm Kernel Shell
MBTS = 2, 2- Dibenzothiazole disulphide TMQ = 1, 2-Dihydro-2, 2, 4-trimethylquinoline

2.4.4 Moisture Content

The moisture content of the sample was determined by using the method prescribed by ASTM D 1509 (1995). A quantity (2.0g) of the sample was weighed and its initial weight recorded. The sample was then oven dried to a constant weight at a temperature of 70°C. The sample was removed from the oven and allowed to cool. The cooled sample was then removed and weighed again, and the weight recorded as the final weight of the sample.

$$Moisture\ content\ (\%) = \frac{(\text{Initial} - \text{Final})\text{weight of sample}}{Initial\ weight\ of\ sample} \times 100 \quad (2)$$

2.4.5 Ash Content

The ash content of the sample was determined by using the method prescribed by ASTM Standard D1506-99 (2013). 2.0 g of sample was weighed into a crucible which is made up of platinum or ceramic. It is important to know the weight of the empty crucible before taking the weight of the sample. It was transferred into a muffle furnace and ashed at 600°C for 3 hours. Then removed and allowed to cool in a desiccator. The final weight of the sample was taken with the aid of a Mettler-Toledo analytical balance.

%Ash=

$$\frac{Weight(g)\ of\ (empty\ crucible + Ash) - empty\ crucible}{Weight\ of\ empty\ crucible\ (g)} \times 100 \quad (3)$$

2.4.6 %Carbon

The % Carbon content was evaluated as follows:

$$\%\ of\ Carbon = \frac{100 - \%Ash}{1.80} \quad (4)$$

2.5 Mechanical Properties

2.5.1 Tensile Properties

The tensile mechanical properties of the vulcanizate were carried out using a method described by Malomo *et al*, (2019). 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. The sample was fixed to the sample holders, one pulling the sample up and the other pulling it down. As the sample was being stretched by the pulling action of the sample holders, the graphical result containing parameters like yield load, elongation at break, tensile strength at yield load, breaking and load and so on were shown on the system.

2.5.2 Young Modulus

This was obtained as the slope of the stress- strain graph of the various samples (Raji, 2015). The loads were converted to stress by dividing the by the area. The elongations were also converted to strain by subtracting the original length from the elongations and the result divided by the original length.

2.5.3 Ultimate Tensile Strength

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

2.5.4 Hardness

This was determined using a method described by Raji, 2015. 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 downward 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 three times per sample and the average value was taken.

2.5.5 Rubber Fatigue

Rubber fatigue test was carried out using a method of ASTM Committee E08.06 (2013). Rubber fatigue test is carried out using rubber fatigue tester or machine with model ZME-7003 and oscillation 2000. It is performed with a sample loaded into a fatigue tester or fatigue test machine and loaded using the pre-determined test stress, then unloaded to either zero load or an opposite load. This cycle of loading and unloading is then repeated until the end of the test is reached. The test may be run to a pre-determined number of cycles.

2.5.6 Abrasion Resistance

The ASTM D1650 was used in determining the ability of the material to resist wear when in contact with abrasive surface. Abrasion test is done using Din Abrasion Tester (Model FE 05000). The sample cut out to a diameter of 16mm, then inserted into the sample holder and the test begins to run. Before starting the wear test, the sample was weighted. Finally, on completion of the wear test, the sample was weighted again to get the weight reduction.

3 Results and Discussion

3.1 Physicochemical Properties of Activated Palm Kernel Shell

The physicochemical properties carried out on Activated Palm Kernel Shells (Table 2) were moisture content, ash content, pH, bulk density, % of carbon, % of nitrogen and particle size. The result for ash content recorded was 2.06% which is within the ASTM required standard (< or =8) Table 2. The result obtained for moisture contents of activated palm kernel shells was 8.06%. The result obtained falls within ASTM recommended standard of 3-10_{max} (Table 2).

According to Emmanuel *et al*, (2017) higher moisture content tends to favour higher adsorption capacity within the material. Bulk density reflects the filler's ability to function as structure support. The APKS has bulk density of 0.62g/ml. This is within the ASTM standard recommended value of < or =8 (Table 2). With this result, APKS may possess high mechanical strength as high bulk density showed an increased greater mechanical strength (Balakrishnan and Satyawali, 2007). The particle size obtained for APKS were 4.00, 3.35, 2.00 and 1.18mm sieve opening. 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. The lower the particle size the lower the density and therefore the better the filler-matrix interactions (Momoh *et al*, 2017).

The activated palm kernel shell had nitrogen percentages of 0.56%. This is similar to work of Ndubuisi *et al*, (2016) who reported nitrogen content of 0.48% for carbonized palm kernel shells as these ranges will however promote filler-matrix interactions. The acidic pH of 5.3 was recorded in this study for APKS. The pH of the sample was acidic and this may be due to acidification process through chemical activation. Meanwhile, acidity many not enhance the cure time of vulcanizates compounded because acidity of the filler tends to slow the cure rate and hence reduce the crosslink density (Malomo *et al*, 2018). The percentage carbon content obtained for APKS was 54.41% (Table 2). The higher the value of percentage carbon the greater will be the reinforcement effect of the filler. The result of this study is related to carbon content of 56.97% reported by Ndubuisi *et al*. (2016).

3.2 Mechanical Properties of the Vulcanizates

3.2.1 Hardness

The result recorded for hardness in varying carbon black/activated palm kernel shell filled NR vulcanizes is presented in Table 3 and Figure 1. In this study, the various values of hardness of 8.3, 9.7, 13.2, 14.2, 14.6, 16.2 and 9.0 (Shore A) were obtained for the sample composition of 30/0, 25/5, 20/10, 15/15, 10/20, 5/25, and 0/30 (CB/APKS) loaded with carbon black/activated palm kernel shells. The hardness of materials is a measure of material's resistance to indentation. This hardness can be improved with fillers; this is as a result of reinforcing properties of fillers through filler-material interactions (Mwaikambo and Ansell, 2001). The trend of the result across sample 1to7 (Table 3) revealed that majority of blends having APKS composition tend to possess high values of hardness. It was also observed that for blends of (CB/APKS) composition having higher APKS composition showed higher hardness. For sample 1 (CB 30, APKS 0), hardness value was 8.3 Shore A, sample 7 (CB 0, APKS 30) hardness value was 9. Shore A. Also, comparing sample 2 (CB 25, APKS 5) and sample 6 (CB 5, APKS 25), hardness value was 9.7 and 16.2 Shore A respectively. Considering, sample 3 and 4 with (CB 20, APKS 10) and (CB 10 and APKS 20) having 13.2 and 14.6 Shore A hardness value while sample 4(CB 15, APKS 15) of intermediate composition with 14.12 Shore A. The hardness obtained for CB/APKS across the samples showed that APKS possesses better reinforcement and strength imparting properties than CB. The PKS went through chemical activation which increases the surface area of the material. Increased surface area improves the filler polymer matrix leading to increasing surface roughness. The surface roughness in turn improves the rubber - filler adhesion resulting in higher tensile strength and hardness compared to those of lower or no APKS composition. It was also found that activation of the filler improved the reinforced mechanical properties as found in the works of Dand *et al*. (2017).

Table 2. Physicochemical Properties of Activated Palm Kernel Shell

Material	Parameters Characterized						
	Moisture Content (%)	Ash Content (%)	Bulk Density (g/ml)	Carbon (%)	Nitrogen (%)	pH	Particle Size (mm)
APKS	8.06	2.06	0.62	54.41	0.56	5.3	4.00, 3.35, 2.00, 1.18
ASTM Standard	3-10 Max	<=8 Max	0.36-0.74	-	-	-	5-50

APKS – Activated Palm Kernel Shell

Table 3: Mechanical Properties of the Carbon Black/Activated Palm Kernel Shell (CB/APKS) Filled Nr Vulcanizates

Samples CB/APKS	Hardness (Shore A)	Young's Modulus (MPA)	Ultimate Tensile strength (UTS) (MPA)	Abrasion Resistance (Initial Value =0.45)	Fatigue Resistance	Modulus elongation at 100%, 200% & 300%	Elongation at Break%
1(30/0)	8.3	4.01	3.50	0.44	No crack	4.08, 5.42, 6.76	137.88
2(25/5)	9.7	5.36	2.83	0.43	No crack	6.19, 21.88, 37. 57	87.38
3(20/10)	13.2	4.32	2.17	0.44	minor crack and weaker	3.90, 5.28, 6.67	96.38
4(15/15)	14.2	6.16	4.33	0.43	minor crack and weaker	7.09, 9.92, 12,75	97.38
5(10/20)	14.6	3.37	1.55	0.44	minor crack and weaker	3.64, 4.73, 5.84	100.88
6(5/25)	16.2	5.80	1.83	0.44	minor crack and weaker	7.62, 11.32, 15.02	68.38
7(0/30)	9.0	4.31	0.20	0.43	minor crack and weaker	0.64, 0.98, 1.32	79.00

3.2.2 Young Modulus

In this study, values of 4.01, 5.36, 4.32, 6.16, 3.37, 5.8 and 4.31MPa were obtained for young modulus by varying ratios of CB/APKS with 30/0, 25/5, 20/10, 15/15, 10/20, 5/25, and 0/30 Table 3 and Figure 1. The results obtained showed increase in young modulus for the majority of blends having high APKS filler loading composition. For instance, the composition of APKS in sample 7 and 6 (0/30, CB/APKS and 5/25, CB/APKS) where high APKS to CB filler loading is used, recorded high young modulus values of 4.31MPa and 5.8MPa compared to composition of high CB filler loading composition in sample 1 and 2 (30/0, CB/APKS and 25/5, CB/APKS) having 4.09MPa and 5.36MPa modulus respectively.

3.2.3 Ultimate Tensile Strength (UTS)

The carbon black/activated palm kernel shell vulcanizates recorded ultimate tensile strength of 3.50, 2.83, 2.17, 4.33, 1.55, 1.83 and 0.20MPa, for samples 30/0, 25/5, 20/10, 15/15, 10/20, 5/25, and 0/30 respectively. Across sample 1 to 7 (Table 3 and figure 2) filler loading, the trend observed was that the majority of the blends having APKS composition tend to have low UTS. For examples it was observed that blend of CB/APKS, 30/0 (sample 1) had higher value of 3.50Mpa of UTS compared to the blend of CB/APKS, 0/30 (sample 7) with lowest and least value of 0.20MPa. Sample 4 (15CB, 15APKS) is having highest value of 4.33MPa, sample 2 and 6 (25CB, 5APKS and 5CB, 25APKS) with value of 2.83MPa and 1.83MPa while that of sample 3 and 5 (10CB, 20APKS and 20CB, 10APKS) with 2.17 MPa and 1.55 MPa respectively. The results from this study showed that CB filler loading composition resulted in high UTS values when compared with APKS filler loading composition across the samples.

3.2.4 Abrasion Resistance

The result of abrasion resistance is presented in Table 3 and Figure 2 below. The measure of the resistance of a material to rubbing or scratching is known as the material's abrasion resistance. The result presented on table 3 shows that for blends across samples 1 to 7 abrasion resistance for all the samples were excellent. The results pointed to the production of well compounded and cross - linked polymer chains. Across sample 1to7 (Table 3) majority of the blends showed excellent abrasion resistance of 97.78%. This could be ascribed to the excellent wear resistance properties of NR compared with the reinforcing effect of the materials used as fillers.

3.2.5 Fatigue Resistance

The fatigue resistance of the vulcanizates is presented in the Tables 3. The results showed excellent resistance to fatigue for most samples except at higher loading of APKS where minor cracks and weaknesses in vulcanizate structure were observed. It was observed that the vulcanizates were slightly prone to fatigue as the CB content decreases across the blends. For instance, at the (30CB, 0APKS), blend recorded high fatigue resistance. It was observed that sample 3 to 7 of CB/APKS filler loading vulcanizate were slightly prone to fatigue and weakness. This could be due to the fact that CB has improved resistance to fatigue characteristics compared to APKS. According to Payne *et al.* (1972) the composite with highest PKS loading had the lowest fatigue life as the PKS filler does not deform during the straining of the composites.

3.2.6 Modulus at 100%, 200% and 300% Elongation

The modulus at 100%, 200% and 300% percentage elongation are presented in the Table 3 and Figure 3 for CB/APKS compounding. The results obtained across sample 1 to 7 showed no agreement with CB or APKS composition because neither of the filler loading recorded an outstanding improvement over the other. From the result obtained it was observed that as the APKS filler loading increases there is rise and fall in the value of modulus at each percentage (100%, 200% and 300%) and vice versa, Also as the loading of CB to APKS loading increase there is rise and fall in the value of modulus at each percentage obtained and vice versa. For example, sample 2 (25 CB, 5 APKS) the values obtained were 6.19%, 21,8% and 37.57% respectively while that of sample 3 (20 CB, 10 APKS) recorded were 3.90%, 5.28% and 6.67%, showing increase of APKS loading. For increase CB, considering sample 6 (5 CB, 10 APKS) and sample 5 (10 CB, 20 APKS) with 7.62%, 11.32% and 15.02%; 3.64%, 4.73% and 5.84% for modulus at 100%, 200% and 300% elongation respectively. The result obtained may be due to method of preparation and mixing of the blends.

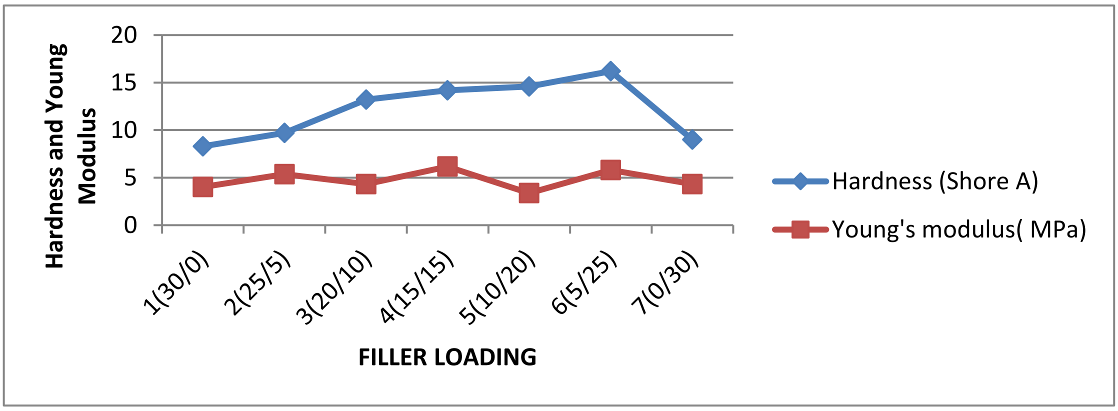

Fig. 1: Graph of Hardness and Young Modulus against Filler Loading Vulcanizates.

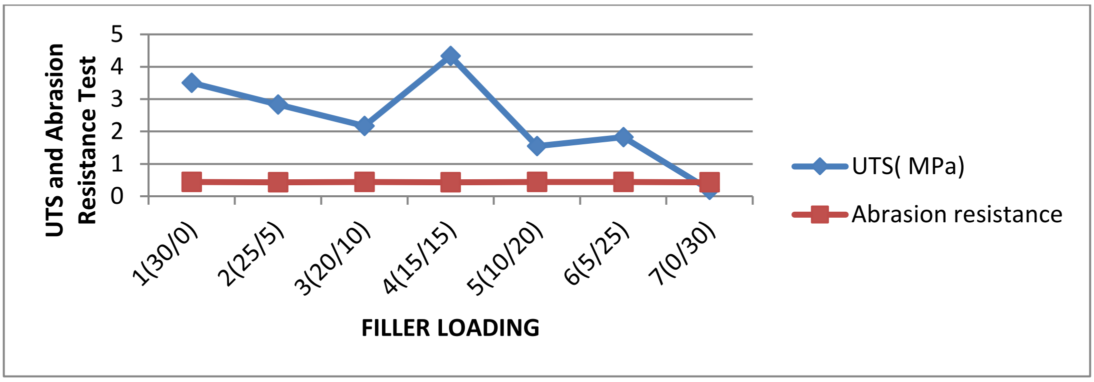

Fig. 2: Graph of Ultimate Tensile Strength and Abrasion resistance against Filler Loading Vulcanizates.

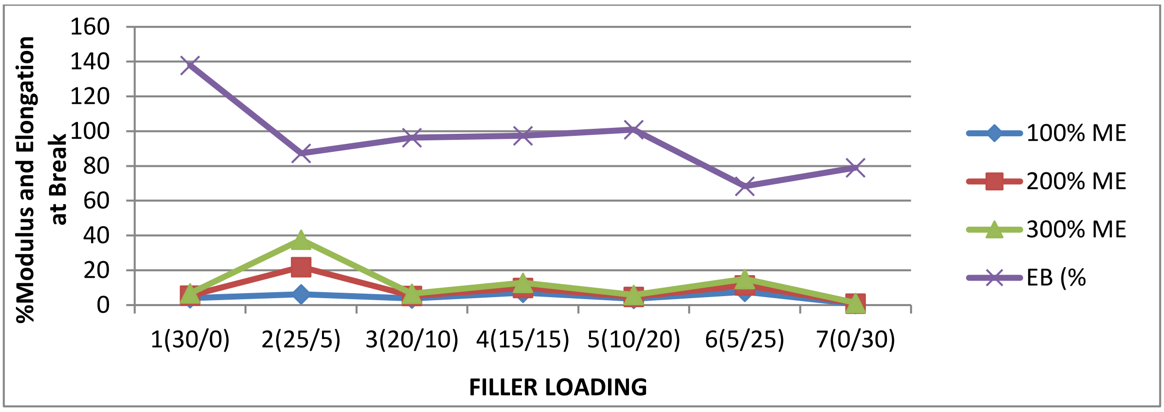

Fig. 3: Graph of % Modulus and Elongation at Break against Filler Loading Vulcanizates.

3.2.7 Elongation at break (EB)

The elongation at break (EB) for APKS vulcanizates is shown in Table 3 and Figure 3. The EB across samples 2-7 showed increase in the elongation as the APKS loading increases. Sample1 (30CB, 0APKS) had the highest value of elongation at break of 137.88%. Apart from sample 1 with highest value, there is steady increase in EB from sample 2, 3, 4 and 5 and subsequently decrease in sample 6 and rise again in sample 7. The higher EB due to CB alone may be due to the fact that chemical activation only may not have the potential ability to partial removal of lignin and hemicellulose polymer matrix of material which likely may result to low resistance to stretching during tensile deformation reported by Dhanalakshmi *et al.* (2015). The decreasing in elongation at breaking as regards APKS to CB loading has been explained in the terms of the filler to the polymer phase leading to the stiffening of the polymer chain and hence resistance to stretch of the polymer when the strain is applied as reported by Jorts *et al*. (2005).

4 Conclusion

This work has shown that the physicochemical properties test on activated palm kernel shell fall within the recommended ASTM standards. Results of the physico-mechanical properties exhibited higher UTS, EB and rubber fatigue test for high CB filler loading composition to APKS while high APKS filler loading composition to CB exhibited higher hardness and young modulus across the CB/APKS filler loadings. Abrasion resistance was excellent for CB to APKS filler loadings. This research showed that higher CB loading exhibited higher mechanical strength to APKS loading; this may be due to the chemical activation of APKS which may not have the potential ability for partial or whole removal of lignin and hemicellulose polymer matrix of palm kernel shell material. Chemical activation only may pose adherence of the filler to the polymer phase leading to the stiffening of the polymer chain and resistance to stretch. The investigation shows that APKS has prospect as filler in natural rubber compounding.

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