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PHYSICO-MECHANICAL PROPERTIES OF NATURAL RUBBER FILLED WITH MERCERIZED SAWDUST POWDER

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ABSTRACT

Assessment of mercerized sawdust as filler in natural rubber vulcanizates was studied. Samples of sawdust was sourced from a wood sawmill, washed off dirt and was mercerized using a solution of sodium hydroxide at a concentration of 25%. The dried sawdust was ground to fine particles and sieved through a mesh size of 75 µm and was used in compounding natural rubber using varying loading levels of 10 phr, 20 phr, 30 phr, 40 phr and 50 phr. The physico-mechanical properties of the mercerized sawdust powder filled vulcanizates presented a better tensile strength than the untreated sawdust powder in all the filler loading at 10 phr (17.94 mpa) to 50 phr (29.05mpa), modulus at 10 phr (10.09%) to 50 phr (21.85%) and from the results obtained, mercerized sawdust when compared with the untreated sawdust had higher values for tensile test which shows that chemical modification helps to improve the properties in natural fibres for a better interaction between fibres and rubber matrix.

Keywords: Loading, natural rubber, sawdust, vulcanizates.

INTRODUCTION

It is a known fact that pure gum rubber is relatively soft and susceptible to degradation by ozone, oxidation and heat. These deficiencies in physical and chemical properties of unreinforced natural rubber made it of very little value in polymer product industries. Therefore, there is the need to incorporate additives such as fillers, cross-linking agents, antioxidants, peptizing agents, softners, accelerators, activators, abrasives, flame proffers and tack producers.

Fillers are one and the most frequently used additives in the processing of polymers. Fillers in rubber compounds play a major role in the physico-mechanical properties and exercise control over cost of the products [1]. It is a well-established fact that carbon black is one of the most classical reinforcing fillers, especially for the rubber industry. However, carbon black is expensive and petroleum from which it is derived is non-renewable [2].

Research and developmental efforts are being carried out with a view of modifying and using natural fibres and other agricultural wastes or by-products such as rice husk, rubber seed, groundnut shell, etc. In rubber compounding [3] on the other hand, lack of good interfacial adhesion and poor resistance to moisture absorption make the use of natural fibres reinforced composites less attractive. Cellulosic fibres are hygroscopic and moisture absorption can result in the swelling of the fibres which may lead to micro-cracking of the composites and degradation of mechanical properties [4].

Chemical treatment of cellulosic fibres by mercerization is a method of reacting the hydroxyl groups -OH of the fibre constituent of the cell wall with sodium hydroxide solution thereby substituting the hydroxyl groups with sodium ethoxyl groups and thus making it hydrophobic. Sawdust, a by-product of wood processing is generally regarded as a waste.In developing country like Nigeria, proper utilization of sawmills waste has not been given due attention which makes sawdust to constitute environmental problem as they form refuseheap in areas where they are not disposed.

This work determines the physico-mechanical properties of natural rubber filled with mercerized sawdust powder as well as accessing its reinforcing potentials as fillers in natural rubber composites.

MATERIAL AND METHODS

Natural rubber conforming to the standard African Rubber grade 3 (SAR3) was obtained from the Rubber Research Institute Iyanomoh, Benin City, Nigeria. Sawdust was obtained from a wood workshop in Auchi, Nigeria. The other reagents ingredients (zinc oxide, stearic acid, Trimethylquinoline, (TMQ), processing oil etc.) were obtained from commercial sources and used as required.

The equipment used during this study is of world class standard and made in England. They include:

a) Monsanto tensile tester model 1/m was used for tensile test properties
b) Wallace hardness tester model c8007/25 was used for hardness test
c) Wallace akron abrasion tester was used for abrasion test

d) Compression set test was carried out by the laboratory press at a pressure of 150Kg/cm2 and at a temperature of 140°C for about 20 minutes for each of the formulation.

Sawdust was collected from a wood shop at Auchi, Nigeria, and thoroughly washed to remove sand and other impurities. It was milled and sieved using a 75µm screen gauze

Mercerization Process: 200g of powdered sawdust (PSD) was soaked in 25% sodium hydroxide solution for 1 hour at room temperature. The solution was filtered and thoroughly washed with distilled water, dried at room temperature for 48hours followed by oven drying at 70°C for 2 hours.

$$\text{Fibre –OH +NaOH} \longrightarrow \text{Fibre- ONa+ } H_2O$$

Characterization

Moisture content was determined for the untreated and mercerized samples by weighing 5 g of dried various samples into a petric dish. The samples were placed in and oven maintained at temperature of 125 °C for an hour. The various samples were then removed from the oven and allowed to cool at room temperature in a dedicator. Triplicate determinations were carried out and the average change in weight was calculated as a percentage of the moisture content [5].

The pH of the slurry of the untreated and mercerized sawdust was prepared by mixing 5g the samples into 50ml of boiling distilled water. The mixture was allowed to cool to room temperature; the p^H meter was used to measure the acidity/alkalinity of the cooled mixture [6]

The bulk density of the various samples was determined by the tapping procedure [7]. Accurately weighed samples were poured into a uniform measuring cylinder of cross sectional area and were then tapped several times until there was no change in volume occupied the volume was then recorded and the bulk density calculated.

The ash content of the untreated and mercerized sawdust was determined;using gravimetric method.In this method, air dried samples were measured into a weighed crucible and its content was placed into an electrical controlled muffle furnace maintained at a temperature of 125°C for 4 hours. Thereafter, the crucible was removed from the furnace and allowed to cool in a desiccator. The ash content was calculated using the average of triplicate determination [8].

PREPARATION OF THE VULCANIZATES

The formulation of the mixes was based on ASTM 3184 [9]as shown below;

Table.1

Ingredients	Parts for hundred (PPR),
Natural Rubber	100
Filler (TSD)	10/20/30/40/50
Filler (USD)	10/20/30/40/50
Zinc oxide	5.0
Stearic acid	2.5
Sulphur	1.5
MBTS (Mecaptobenothiosulphide)	1.5
TMTD (Tetramethylthiurumdisulphide)	3.5
Processing oil	5.0

A batch factor of four was used to multiply the weight of the ingredients in pphr. The efficient vulcanization system was used.

MIXING PROCEDURE

The rubber mixes were prepared on a laboratory size two roll mill according to the mixing cycle shown in table 2. It was maintained at 70 °C to avoid cross linking during mixing, after which, the rubber composite was stretched out. Mixing followed ASMD 3184 standard.

Table 2: Mixing Steps and Mixing Time

Mixing Step	Time (minutes)
Natural Rubber	5
Addition of Stearic acid	1
Addition of Zinc oxide	1
Addition of Filler	10
Addition of MBTS	1
Addition of TMTD	1
Processing of oil	1
Addition of supur	2
Total	22

DETERMINATION OF THE CURE CHARACTERISTICS

The cure characteristics were determined using the Mosanto Rheometer, MDR 2000 model at 150 °C. The cure time predicted by the Mosanto rheographs was used as a guide to obtain vulcanizates for the test specimen.

COMPOUNDING COMPOSITE CURING

The curing of the test pieces was done using compression moulding machine.The curing was carried out at 130 °C for 15 minutes [10].

DETERMINATION OF VULCANIZATE PROPERTIES

The tensile strength, modulus and elongation at break were measured using a Mosantoinstrontensometer in accordance with ASTMD 1412 method [11], ASTMD 1412 [12] Dumb bell test pieces of dimension (45x5x2mm) were used.

The hardness of the vulcanizates was measured with a Wallace hardness tester model C8007/25 in accordance with ASTMD 1415 [12].

Abrasion resistance measurement was based on Din 1504649 Akron to B5 903 part 49 method [13]. Compression set test measurement was based on ASTM 385 [14].

RESULTS AND DISCUSSION

Result of fillers characterization

The physical properties of the mercerized sawdust and the untreated sawdust are shown in Table 3. The result shows that the moisture content of the treated (mercerized) and the untreated sawdust are 3.23% and 5.5% respectively with the mercerized lower which is as a result of the chemical treatment modification. The value of the moisture content of the treated sawdust shows that the effect of moisture at this level on the properties of the vulcanizate is negligible and could be discountenanced. The moisture content of the filler is often used to predict the degree of defect arising from shrinkage during curing particulary for product processed at elevated temperature [4].

The result of the pH for both samples that is, the treated and the untreated sawdust are 8.72 and 5.86 respectively with the treated having an alkaline pH. This is an indication that the treated fibre when used as fillers in compounding, the rate of cure of vulcanizates will be expected to be high. This will enhance time of cure and little amount of accelerators may be needed [15]

The bulk density of the mercerized and the untreated sawdust are 0.40g/cm^3 and 0.65g/cm^3 respectively. The bulk density is mainly influenced by the particle size, the smaller the particle size the lower the bulk density and therefore the better the interaction between the polymer matrix and the reinforcing fibre [7]. From the result in table 3 the bulk density of the treated is lower than of that the untreated which means that the treated sawdust has a better reinforcing potentials.

Weight loss on ignition is a measure of the carbon content loss during combustion and measures the effectiveness of the filler. The higher the values, the greater the reinforcing potential. The result of weight losses, on ignition at 125 ^{o}C in Table 3 are 88.60% and 76.60% for treated sawdust and untreated sawdust which is an indication that treated sawdust is more reinforcing than that of untreated sawdust.

Table 3: Characteristics of untreated sawdust and treated sawdust

PARAMETERS	**USD**	**MSD**
Moisture content (%) w	5.53	3.23
p^{H}of slurry	5.86	8.72
Bulk density (g/cm^{3})	0.65	0.40
Ash content (%)wt	23.40	11.40
Wt loss on Ignition (%) at 125^{0}c	76.60	88.60

Key: USD -Untreated sawdust ; USD- Mercerized sawdust

Mechanical Properties

The physico mechanical properties of the natural rubber filled with treated sawdust powder and untreated sawdust powder are shown in Table 4. The results show that there is an improvement in the tensile properties of the treated over the untreated vulcanizates. In all filled system, the tensile strength increased with increasing filler content

The tensile strength and modulus at 100% obtained were less for USD compared to TSD. This indicates that TSD had effect on the vulcanizates. The marked increasing filler loading for the treated sawdust could be as a result of the superior surface area and loss on ignition with bonding quality between the filled and the rubber matrix of the treated over the untreated sawdust. These suggest a better polymer filler interaction and reinforcement.

The results in Table 4 also show the value of elongation at break as a function of filler loading for the treated/untreated sawdust. The values in the elongation at break decrease with increase in filler content of the mixes for all the fillers. The decrease in elongation at break with increasing filler loading may be due to the adherence of the filler to the polymer matrix leading to stiffening of the polymer chain and hence resistance to stretch when a strain is applied [16]. The elongation at break (EAB) for the treated or mercerized sawdust filled vulcanization in all the loadings are lower than the untreated showing a superior service performance.

The increasing filler loading may be due to the adherence of the filler to the polymer matrix leading to stiffening of the polymer chain and hence resistance to stretch when a strain in applied (16). The elongation at break (EAB) for the vulcanizates in all the loadings are lower than the untreated showing a superior service performance. The hardness values for TSD and USD filled vulcanizates increase with increasing filler loading with the TSD values higher than the USD values. This is an expected trend. This is an indication that as more filler get into the rubber, the elasticity of the rubber chain is reduced resulting in a more rigid vulcanizated [15].

The compression set results in table 4, show that as the filler loading increases the compression set of the filled vulcanizates decreases. However, the vulcanizates filled with TSD have lower compression. The observation may not be unconnected with the amount of filler incorporated into the matrix the degree of dispersion of the filler and particles size. As the filler loading increases, it can be expected that more and more filler particles and aggregates will not be dispersed and method sufficiently by the rubber matrix. [17]. The abrasion resistance results shown in Table 4, decreases marginally with the increasing filler loading with a higher resistance of the treated sawdust. This may be due to the fact that as the filler loading increases, the strength of adhesion between the matrix and filler become very strong [18]. It also indicates that the abrasion resistance of vulcanizates filled with treated sawdust has a better resistance to surface scratch than the untreated sawdust.

Table 4: PHYSICO-MECHANICAL PROPERTIES OF UNTREATED SAWDUST AND MERCERIZED SAWDUST

	FILLER LOADING (PPHR)				
PROPERTIES	10	20	30	40	50
Tensile strength	(15.35)	(18.89)	(19.04)	(23.83)	(21.64)
(Mpa)	[17.94]	[22.45]	[26.15]	[28.42]	[29.05]
Modulus at	(8.31)	(11.25)	(11.98)	(15.63)	(16.05)
(100%)	[10.09]	[13.94]	[15.75]	[21.29]	[21.85]
Elongation at	(540.28)	(486.15)	(461.46)	(398.04)	(341.76)
break (%)	[505.26]	[435.38]	[401.29]	[335.58]	[303.23]
Hardness	(47.43)	(53.39)	(55.48)	(56.36)	(59.25)
(IRHD)	[50.05]	[53.94]	[56.65]	[61.28]	[63.45]
Compression	(21.23)	(18.06)	(17.92)	(14.72)	(12.03)
set (%)	[18.35]	[15.28]	[13.09]	[11.05]	[9.35]
Abrasion	(28.02)	(23.86)	(19.44)	(19.05)	(16.41)
resistance (%)	[31.32]	[27.28]	[25.53]	[23.85]	[20.35]

KEY: Untreated sawdust (USD); Mercerized sawdust [MSD]

CONCLUSION

This study investigated the effect of mercerization of sawdust on the fibre properties and its reinforced natural rubber composites properties. The mechanical properties of natural rubber vulcanizates were enhanced with increasing mercerized filler content, this result show that mercerized saw dust (MSD) is potential reinforcing filler for natural rubber composites

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