

Properties of *in situ* Prepared Various Ebonite Natural Rubbers.

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ABSTRACT

Preparation of various samples of ebonite vulcanizates and their physico-mechanical properties have been investigated using standard methods. This work explores the production of ebonite dust and its rubber vulcanizates, characterization and analysis of the prepared ebonite rubbers. Five different ebonite rubbers – labeled A, B, C, D, and E with sulphur content in parts per hundred grams of rubber (Phr) of 32, 34, 36, 38 and 40 parts respectively were prepared. Physico-mechanical properties were carried out on the prepared rubbers vis-a-viz: tensile strength, hardness and abrasion resistance. The tensile strength (MPa) for sample A, B, C, D and E were 5.6, 3.5, 4.7, 1.7 and 2.0 MPa respectively while the abrasion (% mass loss) were 8.49, 4.24, 2.59, 1.08 and 1.05 % respectively and the hardness (IRHD) being 63, 64, 65, 70 and 82 (IRHD). The results showed that the preparation of ebonite from natural rubber as a base polymer is achievable considering the results of characterization obtained.

Keywords: compounding, natural rubber, ebonite dust, vulcanization, ebonite material.

Introduction.

Vulcanization is the conversion of rubber molecules into a network by formation of cross links using vulcanizing agents [1]. These agents can include sulphur, peroxide, sulphurnating agents and high energy radiation [2]. Vulcanization is the process of converting the gum-elastic raw material into hardened rubber with elastic properties. The extent of the reaction depends on the amount of sulphur used, temperature and duration of heating [3], as the crude rubber is intimately mixed with about 3% ground sulphur, accelerator and activator and then heated to about [4] 150⁰C (for tyres, it is 153 ⁰C). The accelerators increase the rate of the vulcanization process while the activator

forms a complex with the accelerator [4]. Common accelerators in use include mercaptobenzothiozole, diphenyl guanidine, zinc diethyl dithiocarbamate, zinc salt of mercaptobenzothiazole while the activator system commonly in use is the zinc oxide stearic acid system [4].

Natural rubber by nature at molecular state contains unsaturation (residual double bonds) and this makes it susceptible to attack by oxygen, ozone and light. Natural rubber has some inherent properties like poor oil resistance, solubility in aromatic, aliphatic and halogenated hydrocarbons [5]. It has limited resistance to oxidising acids and mineral acids even though it is found to be resistant to many inorganic chemicals. In

view of these limitations, natural rubber is vulcanized to improve its level of resistance to general agents of degradation [6]. Ebonite was a brand name for very hard rubber first produced by Charles Goodyear by vulcanizing rubber for prolonged periods. It contains about 30-40% sulphur and its initial use was meant for artificial substitute for ebony wood. Ebonite being another class of interesting rubber products is used in mechanical applications such as in water – meter components, pipe stems, fountain pens, piston rings, textile machinery accessories and surgical equipment [6]. Chemical applications include battery boxes, pumps for chemicals, tank linings, buckets and roller coverings. The electrical applications include electrical insulating components such as plugs, sockets, inductance coil and covering for electrical tools [6]. Vulcanized ebonite is machinable and many engineering products can be made adopting regular engineering processes like turning, grinding, boring and drilling. It cannot however be welded for making joints [6].

In view of the wide domestic and industrial applications of ebonite, this work involves the formulation, compounding and characterization of newly prepared ebonite vulcanizates with the expectations of finding uses for the prepared rubber materials.

Experimental.

Materials

Natural rubber conforming to Technically Specified Rubber (TSR) 10, but usually denoted as Standard African Rubber (SAR) grade 10 was used and obtained from Rubber Research Institute of Nigeria. The properties of this TSR are as presented in Table 1 [7] for the preparation of ebonite. The chemicals used include, high aromatic white spirit, xylol mercaptan (XM) peptizer,

stearic acid, magnesium oxide, diphenyl guanidine (DPG), sulphur and Polymerised 2,2, 4-trimethyl – 1, 2-dihydroquinoline (TMQ) and anti oxidant which are all industrial grades.

Table 1: Typical characteristics of the Crumb rubber

Parameter	Values
Dirt content (DC) (%)	0.17
Volatile Matter (VM) (%)	0.81
Ash content (AC) (%)	0.36
Plasticity Retention Index (PRI) (%)	92.20

Compounding and production of Ebonite material

The detailed process used in the production of ebonite material is given below.

Compounding and production of ebonite dust filler In the production of ebonite dust, five formulations as shown in Table 2 were developed and labeled A, B, C, D and E.

Table 2 Formulations for production of five samples of ebonite dust.

Ingredients (Phr)	A	B	C	D	E
Natural Rubber (NR)	100	100	100	100	100
Stearic Acid	2	2	2	2	2
Magnesium oxide	5	5	5	5	5
Diphenyl guanidine	6	6	6	6	6
Tetramethylquinoline	0.67	0.67	0.67	0.67	0.67
Sulphur	32	34	36	38	40

The process involves mastication of NR in a two roll mill for molecular weight reduction. Thereafter, the compounding ingredients were added in the order; Stearic acid, MgO, TMQ, DPG and Sulphur. The compounded samples were used one after the other in a compression moulding machine for nine hours to produce a hard material which was

crushed in a grinding machine to obtain ebonite dust. The machine was set at 150⁰C for purpose of curing the vulcanizate material. The ebonite dust produced in this stage served as the filler for the subsequent process where ebonite material was produced

Compounding and production of ebonite material

For the production of the ebonite vulcanizates, five formulations were also developed as presented in Table 3.

Table 3: Formulation for the production of five samples of ebonite vulcanizates

Ingredient (Phr)	A	B	C	D	E
Natural Rubber	100	100	100	100	100
Stearic Acid	2	2	2	2	2
Magnesium Oxide	5	5	5	5	5
Diphenyl granidine	6	6	6	6	6
Tetramethylquinoline	0.67	0.67	0.67	0.67	0.67
Ebonite dust (ED)	32	34	36	38	40
Sulphur	32	34	36	38	40

For each sample, the NR was masticated in a two roll mill for easy incorporation of compounding ingredients. The ingredients were added in the order stearic acid, MgO, TMQ, DPG, ED and Sulphur. Cure characteristics were carried out for all the five samples in an oscillating Disc Rheometer (ODR) machine. Thereafter the curing of the samples at 150⁰C for one hour each were carried out in the hydraulic press to facilitate the action of sulphur in cross linking the polymer chains. After curing the machine was switched off and the mould opened to remove the cured ebonite vulcanizate.

Determination of Physico-mechanical Properties of Ebonite Vulcanizate [8].

Tensile Properties.

The tensile tests were carried out using Zwick/Roell Tensometer on a dumb bell shape samples at room temperature (27⁰C). The size of the dumbbell shaped samples were 30.00mm and area of 15.00mm². The load cell for the machine was 1KN with a cross head speed of 100mm/min.

Abrasion resistance

The Wallace Abrader model-Wallace was used for determination of abrasion resistance [8].

Hardness

All hardness tests readings were determined using the pocket-Durometer Hardness Tester ZHT2093. This was done by making indentations on samples and the values of hardness were read as the area of indentations formed [9].

Results and Discussion.

The effect of Sulphur loading on tensile strength, abrasion resistance and hardness are presented on Table 4

Table 4: Results of Characterisation of the Ebonite Samples

Sample Code	Sulphur content %	Tensile Strength (MPa)	Abrasion Resistance (% wt loss)	Hardness (IRHD)
A	32	1.7	8.49	63
B	34	2.0	4.24	64
C	36	3.5	2.59	65
D	38	4.7	1.08	70
E	40	5.6	1.05	82

Note: Each of the value on Table 3 above is mean of three replicate readings.

Tensile Strength

The result for tensile strength shows a progressive increase from sample A to E with sample A having value of 1.7 MPa while sample E had a value of 5.6 MPa representing over 200 % increase.

This increase as shown on Fig1 may be attributed to the greater cross linking density arising from several sulphur linkages formed in the rubber molecular network. The tensile strength of the material is expected to get to a maximum at a certain level of sulphur loading arising from maximization of cross linking of chemically accessible double bonds.

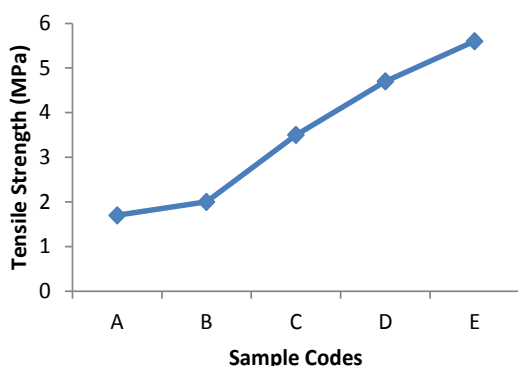


Figure 1: Variation in Tensile properties for the five ebonite samples

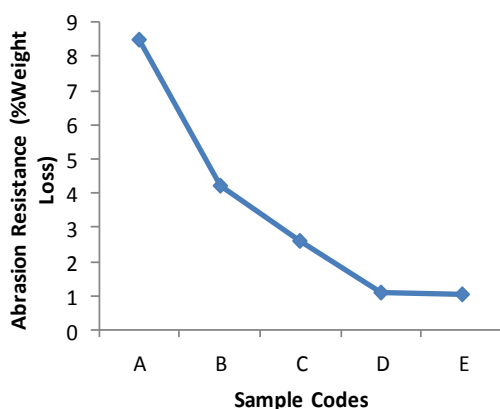


Figure 2: Variation in Abrasion resistance for the five ebonite samples

Abrasion Resistance

The extent of abrasion resistance is determined by weight loss of material under investigation and the results show that as the sulphur loading increases, the value reduced, as can be seen in Figure 2. The reduction in value being equivalent to the increasing resistance to surface wearing is indicative of increased intermolecular forces binding the rubber molecules together. This is thought to arise from the increased sulphidic linkages most of which may be mono sulphides in nature with three dimensional bonding directions [10].

Hardness

The international hardness degree number is the measure of hardness taken as the resistance of the material to indentation under standard specific loading and conditions. The values recorded for samples A, B, C, D and E was 63, 64, 65, 70 and 82 IRHD respectively. These values as presented in figure 3 shows that sample A had least value of hardness while sample E had the highest value. The hardness of samples A B and C are essentially the same while that of D and E are distant apart. Samples D and E are however much harder than the conventional networks known for natural rubber vulcanizates [11]. The high value of sample E is in conformity with the observation in tensile strength and resistance to surface wear. Sample D and E were found to exhibit the ebonite properties which could be useful sources of raw materials for hard rubber products.

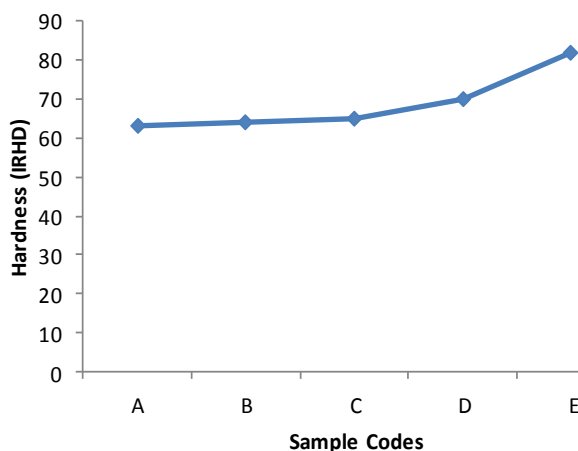


Figure 3: Variation in Hardness for the five ebonite samples

Conclusion

After investigations of the physico-mechanical properties and the cure characteristics of the ebonite vulcanizates, it was found that they compared favourably with those quoted in literature [12]. The work produced information on the production of different grades of the material suitable for several industrial applications where mechanical wear is inevitable. The material produced can also be used for caging of car batteries as it can resist the acidic environment in service conditions. The investigation also shows that straight compounding and vulcanization of NR to make ebonite material, irrespective of % Sulphur applied is not technically achievable. The initial production of ebonite dust which serves as filler for the subsequent compounding and curing of ebonite samples was found to be compatible. The physico-mechanical properties of the ebonite samples A – E were found to be several folds higher than the conventional NR networks. The formation of several mono

sulphidic linkages of sulphur atoms in the molecular networks is hereby adduced for the results obtained.

Recommendation

Further works on the degradability of the ebonite rubbers in different environment is suggested in order to ascertain the level of resistance to chemicals, radiation and ozone. Of equal importance is the need to do a detailed analysis to determine nature and density of cross links formed

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