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Preparation and Properties of NR Based Ebonite Rubber Suitable for Use as Engineering Material

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Abstract

The 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, production of ebonite vulcanizates and investigation of the characterization of the ebonite. Five different ebonite materials-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 respectively were produced. The physico-mechanical properties carried out were 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 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.The results show that the preparation of ebonite from natural rubber as a base polymer is feasible considering the results of characterization obtained.

Keywords: Compounding; Natural rubber; Ebonite dust; Vulcanization; Ebonite material

Abbreviations: IRHD: International Rubber Hardness Degrees; TSR: Technically Specified Rubber; SAR: Standard African Rubber; ODR: Oscillating Disc Rheometer.

Introduction

Vulcanization is the conversion of rubber molecules into a network by formation of cross links by use of vulcanizing agents. These agents include sulphur, peroxide, sulphating agents and high energy radiation. Since vulcanization is the process of converting the gum-elastic raw material into the rubber-elastic end product, the ultimate properties like hardness and elasticity depend on the course of vulcanization. During this process, rubber is hardened with sulphur or sulphur compounds. The extent of the reaction depends on the amount of sulphur used, temperature and duration of heating [1-3].

During vulcanization, the crude rubber is intimately mixed with about 3% ground sulphur, accelerator and activator and then heated to about 150^0C (for tyres it is 153^0C). The accelerators increase the rate of the vulcanization process while the activator forms a complex with the accelerator-sulphur system to enhance the efficiency of the accelerator. Common accelerators in use include mercaptobenzothiozole, diphenyl guanidine, zinc diethyl dithiocarbamate, zinc salt of mercaptobenzothiazole. The activator system commonly in use is the zinc oxide stearic acid system. Natural rubber by nature at molecular state contains unsaturation (residual double bonds and this makes it susceptible to attack by oxygen, ozone and light. It is also not oil resistant and is swollen in aromatic, aliphatic and halogenated hydrocarbons. 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 [4]. At a higher level of sulphur loading during vulcanization under well monitored conditions, a unique product, the ebonite is produced .Initially 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. 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. 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.

In view of the wide domestic and industrial applications of ebonite, this work involves the formulation, compounding and characterization of ebonite vulcanizates. From the results obtained it should be possible to suggest the uses of the various formulations based on their property vis-a-vis service condition when in use. The work is a clear deviation from the extensive researches in NR technology which had hitherto focused on conventional sulphidic linkages.

Experimental

Materials

Natural rubber conforming to Technically Specified Rubber (TSR) 10, but usually denoted as Standard African Rubber (SAR) grade 10 was obtained from Rubber Research Institute of Nigeria. The properties of this rubber are presented on Table 1 [5]. The chemicals used include, high aromatic white spirit, xylyl mercaptan (XM) peptizer, stearic acid, magnesium oxide, diphenyl guanidine (DPG), sulphur and Polymerised 2,2, 4-trimelthyl -1, 2-dihydroquinoline (TMQ) and anti-oxidant which are all industrial grades. Ebonite dust a pre-cursor to ebonite production was produced in first compounding process.

Apparatus and equipment

The following equipment and apparatus of The Rubber Research Institute, Nigeria were used for the work.

i. Compression moulding machine: TECHNO LOIRE PLC 50T-3P. Made in England.
ii. Banbury Pullen 2-Roll Mill. BR-1600. Made in England.
iii. Wallace Abrader. Model-Wallace Ref A2. Made in England.
iv. Pocket Durometer Hardness Tester. ZHT 2093. Made in England
v. Rheometer: ALPHA TECHNOLOGIES. Oscillating Disc Rheometer ODR 2000 MODEL. Made in France.
vi. Oven: GRIFFEN 300FC. Isuzu Seisakusho Co. LTD Tokyo Japan.
vii. Metrolac – zeal. SPGRT6628. Made in England
viii. Zwick/Roell tensometer: 300 series electromechanical test machines. Made in England.
ix. Metal Metrolac jar: Made in Nigeria.
x. Measuring cylinder: SPG1000 mL graduated. Made in England
xi. Plastic bowl: Made in Nigeria
xii. Weighing balance: Sartorius Ag. Gottingen. BP 1215. Made in England
xiii. Flat bottom glass dish
xiv. Stop watch: 31305 models. Made in China
xv. Wallace Plastimeter: P12E. S/no C97008/28. Made in England.
xvi. Grinder – Apex grinder. Mesh 100.
xvii. Micrometre Screw gauge. Model 196A6Z. Made in Germany.
xviii. Microtome (Punch): Model dumbbells punch C88036.
xix. Scissors
xx. Unglazed non-gummed acid free cigarette paper: King size Rizla, Made in England
xxi. Desiccator. Product number-Z553808. Made in England.
xxii. Petri dish
xxiii. Conical flask: Pyrex SPG 1000ml graduated. Made in England
xxiv. Hot plate. Clifton hot plate. HP1.30E. Made in China.

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

Table 1: Typical characteristics of the Crumb rubber.

Compounding and production of Ebonite material

The production of ebonite material requires the initial production of ebonite dust as a pre-cursor. The ebonite dust is therefore latter used in as filler in the proportion similar to carbon black. For the production of ebonite material, therefore three major materials are required; crumb rubber, ebonite dust and compounding ingredients. The detailed process used in the production of ebonite material is given in 5.2.1. And 5.2.2

Compounding and production of ebonite dust filler: In the production of ebonite dust, five formulations as shown on Table 2.0 were developed labeled and labeled A, B, C, D and E.

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

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

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 150^{0}C 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.

Ingredient (Phr)	A	B	C	D	E	
Natural Rubber (NR)		100	100	100	100	100
Strearic Acid		2	2	2	2	2
Magnessium Oxide (MgO)		5	5	5	5	5
Diphenyl granidine (DPG)		6	6	6	6	6
Tetramethylquinoline(TMQ)		0.67	0.67	0.67	0.67	0.67
Ebonite dust (ED)		32	34	36	38	40
Sulphur	32	34	36	38	40	

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

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 150^{0}C for one hour each was carried out in the compression moulding machine. 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

Tensile properties: The tensile tests were carried out using Zwick/Roell Tensometer on a dumb bell shape samples at room temperature (30^{0}C).The size of the dumbed bell shaped samples were 30.00mm and area of 15.00mm^2 The load cell for the machine was 1KN with a cross head speed of 100mm/min [6].

Abrasion resistance: The Wallace Abrader model- Wallace Ref 2 was used for determination of abrasion resistance [6].

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 [7].

Results and Discussion

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

Sample code	Sulphur content %	Tensile Strength (MPa)	Abrasion Resistance (% wt loss)	Hardness (IRDH)
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

Table 4: Results of Characterisation of the Ebonite Samples.
Note: Each of the value on Table 4 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 5.6MPa representing over 200% increase.

This increase as shown on Figure 1 may be attributed to the greater cross linking density arising from several sulphur linkages formed in the rubber molecular network [8].

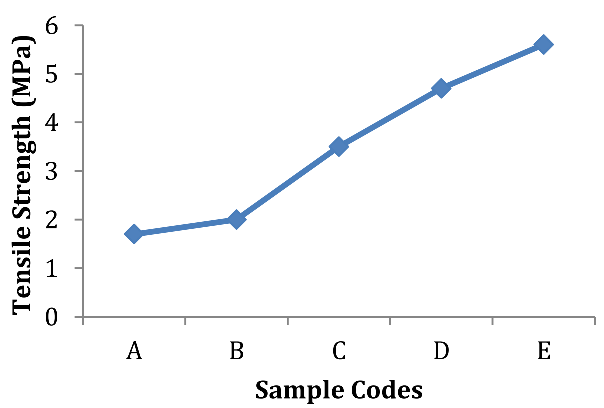

Figure 1: Variation in Tensile properties for the five ebonite samples.

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, 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 sulphide in nature with three dimensional bonding directions [8].

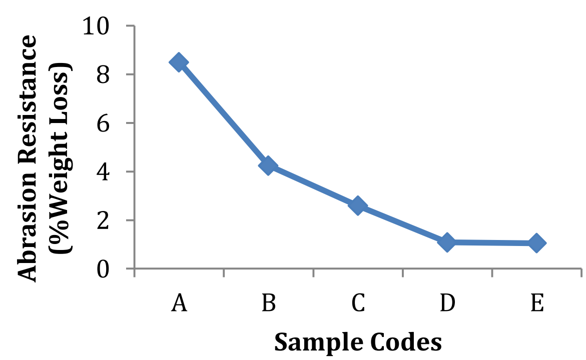

Figure 2: Variation in Abrasion resistance for the five ebonite samples.

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 respectively. These values as presented on Figure 3 shows that sample A had least value of hardness while sample E had the highest value. The harness 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 [9]. 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 to useful sources of raw materials for hard rubber products.

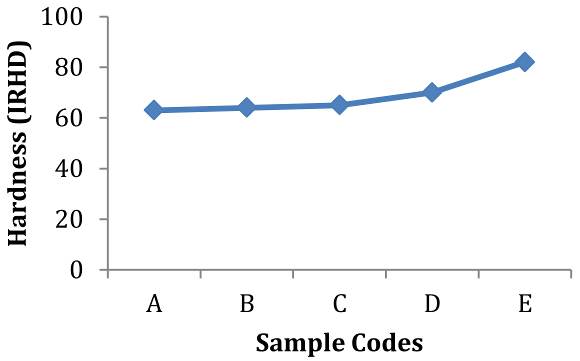

Figure 3: Variation in Hardness for the five ebonite samples.

Conclusion

After investigations of the physico-mechanical properties and the cure characteristics of the ebonite vulcanizates, it was found that the compare favorably with those quoted in literature [10] although this work produced information on the production of different grades of the material suitable for several industrial applications where mechanical wear and chemical attack are inevitable. The material produced can 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 feasible. 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.

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