Effect of Carbonization Temperature of Filler on the Tensile Properties of Natural Rubber Compounds Filled with Cassava (*Manihot esculenta*) Peel Carbon.

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ABSTRACT

Samples of cassava peels were carbonized at varying temperatures (100, 200, 300, 400, and 500°C) for forty-five mins each and sieved through 150µm screen. The portion of the cassava peel carbon that passed through the screen was characterized in terms of loss on ignition, surface area, moisture content, pH, bulk density, and metal content and used in compounding natural rubber. The characterization shows that the pH, conductivity, loss on ignition, and surface area increases as the heating temperature increases, unlike the bulk density which decreases. The compound mixes were cured using semi-efficient vulcanization system. Cure characteristics and tensile properties of the vulcanisates were measured using N330 carbon-black filled natural rubber vulcanisate as a standard. The results of the cure characteristics showed that the cure times, scorch times and the torque gradually increased, with increasing filler content for cassava peel carbon-filled natural rubber, with filler obtained at carbonizing temperature of 300°C tending to show optimum cure indices. The physico-mechanical properties of the vulcanisates increase with filler loading. The reinforcing potential of the carbonized cassava peel carbon was found to increase markedly for the filler obtained at the temperature range 300°C and then decrease with further increase in temperature.

(Keywords: cassava peel carbon, natural rubber, physic-mechanical properties, vulcanisates)

INTRODUCTION

One of the most important additives and second largest following the base polymer in rubber compounding is the filler. Fillers improve processability; physiomechanical properties such as tensile properties hardness; flex fatigue; tear and abrasion resistances; and may cheapen the final product. They achieve performance enhancement by forming strong chemical bonds with the rubber, that is, strong filler elastomer interactions. The fillers used in rubber compounding can be classified into reinforcing and non-reinforcing types. Reinforcing filler is one which increases the tensile strength, hardness and abrasion resistance of the rubber article. The finer the particles size the more reinforcing the filler is. Examples of reinforcing filler include; carbon black and silica. Non-reinforcing fillers cause reduction in strength properties but may increase hardness and modulus of the rubber product. They are usually applied as diluents or extenders to generally reduce cost, examples include the whitings ($CaCO_3$) and China clay [1-3].

The use of carbon from agricultural by-products (maize cob, bamboo culms, groundnut husk, cassava peel, cocoa pod husk, plantain peel, rubber seed shell, pineapple leaves, etc.) to produce vulcanisate materials is gaining attention over the past few years, because of availability of materials [3, 8].

Cassava peel is an agricultural by-product of the cassava plant. Cassava is the third largest source of carbohydrates for human food in the world, with Africa its largest center of production. World production of cassava root was estimated to be 184 million tonnes in 2002. The majority of production is in Africa where 99.1 million tonnes were grown. 51.5 million tonnes were grown in Asia and 33.2 million tonnes in Latin America and the Caribbean. Nigeria is the world's largest producer of cassava [4]. The economic importance of the cassava has largely focused on the cassava tubers with little or no attention paid to the potential usefulness of its by-product. While significant progress has been made in the development and utilization of modified agricultural by-product in water and wastewater treatment, [4, 5] little information exists on the potential for the application of these by-product as filler in the processing of polymers [6, 7].

The temperature at which carbonization of an agricultural by products is carried out could affect the characteristics of the carbon obtained and therefore, the Rheology/physico-mechanical properties of the rubber vulcanisates. It is in view of these that this present research work was undertaken, with an objective to explore the effect of carbonization temperature of cassava peel on the tensile properties of the natural rubber compounds.

MATERIALS AND METHODS

Cassava peels were obtained from a garri processing house at New Benin; Benin City, Nigeria. Natural Rubber crumb (NSR 10) used for the study was obtained from the Rubber Research Institute of Nigeria, Iyanomon, Nigeria. All the other reagents used were of commercial grade, while the industrial grade carbon black (N330) filler was obtained from the Port Harcourt Refinery and Petrochemical Company, Port Harcourt, Nigeria.

Preparation of the Cassava Peel Carbon

The cassava peels were separated from the tuber crumbs, air-dried and reduced to small sizes. Five samples of 1kg each were weighed and heated to temperatures: 100, 200, 300, 400, and 500°C at forty-five minutes each, using a Muffle furnace. The carbonized peels were then milled to fine powder, and sieved through a mesh size of 150µm. The carbon particles that passed through the screen were collected, characterized and used for compounding.

Characterization of the Cassava Peel Carbon and the N330 Carbon Black

The cassava peel carbon (CPC) and the N330 carbon black were characterized as follows: loss of ignition was determined gravimetrically [9], moisture content was determined by method described in [10], the bulk density was determined according to the method described by [11], the pH was determined using ASTM D 1512, 1983 [11], iodine adsorption number method was used for surface area measurement as described by [12], and conductivity was determined using the conductivity meter. Results are as presented in Table 2.

PREPARATION OF THE NATURAL RUBBER VULCANISATE

The formulation of mixes is shown in Table 1. Natural rubber was masticated on the mill for five minutes followed by addition of the ingredients. A semi-efficient vulcanization system was chosen. The Vulcanisate materials were prepared in a laboratory two-roll mill (160 x 320mm) maintained at a temperature below 80°C.

Table 1: Formulation for Compounding Natural Rubber.

Ingredient	Parts per Hundred Rubber
Natural rubber	100
Filler (CPC/CB)	20/40/60
Zinc oxide	5.0
Stearic acid	2.5
Sulphur	1.5
MBTS	1.5
TMTD	3.5
Processing oil	5.0

The recipe for compounding of the natural rubber (NSR 10) with CPC for each sample

CURE CHARACTRISTICS OF NATURAL RUBBER COMPOUNDS

The cure characteristics were measured using the Mosanto Rheometer, MDR 2000 model. The cure times predicted by the Mosanto rheographs were used as guide to obtain vulcanisates for the test specimens.

DETERMINATION OF VULCANISATE PROPERTIES

Compression molding process was used for curing. This was done using the laboratory press at a pressure of 150Kg/cm^2 and at a temperature of 140^{o}C. The tensile strength, modulus and elongation at break were measured using a Monsanto instron tensometer in accordance with ASTM D412-87 method A, 1983 [13]. Dumbbell test pieces of dimension (45 x 5 x 2mm) were used.

Table 2: Characterization of the Cassava Peel Carbon.

Temperature of Carbonization	pH of Slurry at 28° C	Conductivity mho/cm	Iodine Adsorption number (mg/g)	Bulk density g/ml	Loss ignition %
100°C	3.48	32.95	32.10	0.81	4.82
200°C	4.62	40.42	43.86	0.64	38.30
300°C	6.85	101.32	66.11	0.54	67.25
400°C	7.35	153.18	56.24	0.44	75.12
500°C	8.50	181.20	40.61	0.40	78.90
Carbon black	6.25	200.90	82.61	-	91.40

RESULTS AND DISCUSSION

Characteristics of the Cassava Peel Carbon

The characteristics of the cassava peel carbon (CPC) are given in Table 2, using the characteristics of N330 carbon black as a standard. The trend of the pH of the cassava peel carbon as a function of the carbonizing temperature given in Table 2 varied over a range of 6.51-8.10. The results show a progressive Increase in pH with increase carbonization temperature. The pH of the carbon black is 6.50, which is lower to that of the cassava peel carbon at 500°C.

The bulk density of the CPC samples given in the Table 2 varied from between 0.585- 0.411 g/ml. It shows that at high temperature, the bulk density reduces showing that the interstitial spaces (micropores) in the carbon residue are opened and thus resulting in easy compaction and interaction with the polymer matrix.

Cure Characteristics

The cure characteristics of CPC filled natural rubber systems being evaluated are given in Table 3. The results showed that the scorch, and cure times gradually decreased with increasing filler content for CPC-filled natural rubber, while the torque increases. The trend observed in the cure characteristics may be attributed to differences in the filler properties. However, in the present study the most probable factors to account for the observed cure enhancement are surface area, moisture content, and pH.

Tensile Properties of the Natural Rubber Vulcanisates

The tensile properties of the CPC-filled natural rubber vulcanisate with N330 carbon black as standard are shown in Table 4.

A gradual increase in tensile strength as well as modulus with the weight fraction of filler is noticed up till 300°C. It clearly indicates that as the loading increases there is a progressive increase in tensile strength and the modulus for both the N330 carbon black and CPC-filled natural rubber vulcanisates. In all filled systems, tensile strength and modulus at 100% increase with increasing filler type and content.

The results show that the filler loading at which maximum values of the tensile strength and modulus at 100% obtained were less at above filler type 300. The modulus data showed decrease as filler loading increase above 300. From the table, the values of the tensile strength and modulus at 100% for N330 carbon black shows that as the filler loading increases the tensile strength and modulus also increases and more higher than that of CPC–filled Vulcanisates, suggesting that these could be as a result of the higher surface area and loss on ignition of the N330 carbon black.

The values of Elongation at break (EB) decreases with increase in filler type and content of the mixes for all the fillers below 300 and also for N330 carbon black. Decrease in elongation at break has been explained in terms of adherence of the filler to the polymer phase leading to the stiffening of the polymer chain and hence resistance to stretch when strain is applied [3, 8]. However, the values of the EB of the vulcanisates are quite close and comparable to that of N330 carbon black particularly at lower loading and at 300.

Table 3: Cure Characteristics of the Natural Rubber Filled with Cassava Peel Carbon in Comparison with Carbon Black N330.

Parameters	Filler loading	F100	F200	F300	F400	F500	N330
Scorch time(s)	20	66	52	44	42	40	94
	40	70	56	50	48	42	72
	60	80	64	52	50	47	68
Cure time(s)	20	98	210	190	180	165	72
	40	106	192	189	160	140	90
	60	126	227	200	190	170	82
Tmax-Tmin (kg-cm)	20	7.01	9.03	9.78	10.00	10.25	41.04
	40	7.23	11.75	10.06	10.48	10.95	41.39
	60	8.39	12.50	13.89	14.05	14.35	15.86

Table 4: Tensile Properties of the Natural Rubber Vulcanisates.

Properties	Filler Loading	0°C	100°C	200°C	300°C	400°C	500°C	Carbon Black
Tensile Strength (MPa)	20 Phr	5.20	6.12	6.66	8.15	7.65	7.19	24.63
	40	6.78	8.89	9.10	9.40	8.02	7.68	28.15
	60	7.85	8.94	9.53	10.73	9.50	8.11	30.00
Elongation At beak (%)	20Phr	460.00	420.00	402.50	340.00	385.00	320.00	465.00
	40	448.20	415.00	384.00	355.10	361.00	331.00	324.00
	60	422.10	400.15	354.10	333.20	340.40	358.50	302.00
Modulus (%)	20Phr	3.30	3.58	5.63	5.96	4.11	4.03	6.50
	40	3.54	3.66	6.82	7.45	7.24	6.91	7.78
	60	4.05	4.39	6.98	7.68	7.43	7.22	8.50

CONCLUSION

The results indicate that physico-mechanical properties of vulcanisates are greatly influenced by filler carbonizing temperature and loading; and are therefore significant factors in determining the use in rubber compounding.

The vulcanisates exhibit high quality characteristics at filler type 300-500 (that is at filler carbonized at 300 -500°C) and with 60phr loading. It is concluded that for high quality vulcanisate using cassava peel as the reinforcing filler, carbonization should be done at 300°C for 45 mins.

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