Effect of Filler Carbonizing Temperature on the Mechanical Properties of Natural Rubber Composites

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Abstract: The effect of carbonization temperature on the mechanical properties of groundnut shell filled natural rubber composite was studied. Samples of groundnut shell were carbonized at varying temperature (100,200,300,400,500,600,700 ^{o}C) for three hours each and they were ground and passed through 150μm sieve. Another sample was ground without carbonization and filtered as well. The compound mixes were cured using efficient vulcanization system. Mechanical properties of the composites were measured as a function of filler type and loading in comparison with N330 carbon black filled natural rubber composite. It was found that some mechanical properties such as tensile strength, modulus, hardness and abrasion resistance increase with filler carbonization temperatures and loading while other properties such as compression set, Flex fatigue and elongation decreases with filler carbonization temperature and loading. The percentage swelling in Benzene, Toluene and Xylene also decreases with carbonization.

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INTRODUCTION

Raw Rubber, in it original state is not used for any Engineering and domestic applications. The manufacture of rubber products involves the addition of many ingredients called additives into the rubber. This is to allow the rubber compounds to be satisfactorily processed and when vulcanized improved the application properties of the rubber products. The additives used in rubber compounding include: vulcanizing agents, accelerators, activators/retarders , antidegradant, fillers, plasticizers and other ancillary ingredients. One of the most important additives and second largest in the rubber compounding is the filler. Fillers improve physico-mechanical properties such as tensile strength, modulus, hardness, flex fatigue, tear and abrasion resistance. They also reduce the cost of the final products. They achieve performance enhancement by forming strong chemical bonds with the rubber, that is, strong filler elastomer interactions.

In the rubber industry, fillers that are commonly in use are carbon black, china clay, calcium carbonate and aluminium hydroxide. Carbon black which is petroleum derived is the most widely consumed. However, the unstable high price of crude oil has led to the search for fillers from agricultural by-products that can either replace carbon black or blend with it to a reasonable level without adverse effect on the desired properties.

The purpose of this study, therefore, is to evaluate the effect of carbonization temperature on the filler properties of groundnut shell, the physico-mechanical properties and equilibrium swelling of groundnut shell filled natural rubber composite.

EXPERIMENTAL

Materials

Natural Rubber crumb (NSR 10) used in this study was obtained from Rubber Research Institute of Nigeria, Iyanomon, Benin city, Edo State.

Groundnut shells used in this study were obtained from Auchi, in Edo State, Nigeria. All the other additives used in compounding were of industrial grade.

Preparation of Groundnut shell filler and carbonization

The groundnut shell were washed in water and dried in air to remove sand particles and moisture. After drying, portion of the groundnut shell were milled to fine powder and sieved through a mesh size of 150μm. The fine particles that passed through the mesh were collected and characterized. The remaining part were weighed and carbonized at temperatures varying from 100, 200, 300, 400, 500, 600 and 700^{o}C, for three hours (Ishak and Baker, 1995). The carbonized shells were then milled to fine powder and sieve through the 150μm sieve mesh. The fine powder that passed through were collected and characterized.

Characteristics of the Groundnut shell filler

The various groundnut shell powder were characterized in terms of pH, Bulk density, moisture content, Iodine adsorption number and loss on ignition. The pH of the carbonized and uncarbonized samples were determined using ASTMD 1512 method, the bulk density were determined by the tamping procedure

(Ahmedna et al, 1997), the moisture content were determined by adopting the method described in ASTMD 1509, Iodine adsorption number (surface area) were determined using ASTMD 1510(1983), while loss on ignition was determined using procedure described in ASTMD 7348.Results are as presented in Table 1.

Compounding and Curing

The Formulation shown in Table 2 was used to compound the natural rubber. Mixing was carried out on a laboratory size (160 x 320mm) two-roll mixing mill maintained at a temperature below 80°C.

Compression molding was the process used for curing. This was done using the laboratory press at a pressure of $150 Kg/cm^2$ and at a temperature of 140^oC for about 20 minutes for each of the formulation.

Nation of the Physico – mechanical properties

The tensile properties were determined on a Monsanto tensile tester model 110 in accordance with ASTM D412-87 method A using dumbbell specimens that were punched from the molded sheet with die C. For each composite, three specimens were tested and average values reported.

Sorption Experiment

The equilibrium swelling of the compounds in Benzene, Toluene and Xylene were determined by using the method described in ASTM D3010.

$\% \text{ Swelling} = (W_2 – W_1/ W_1) \text{ X } 100$

Where W_1 and W_2 are the initial weight and weight of the swollen sample respectively.

RESULTS AND DISCUSSION

Characteristics of the Filler

The physical properties of the groundnut shell filler are shown in Table 1; the results show that the pH increases from acidity to alkalinity as the carbonization temperatures increases. The possible reason for this is that as the residual materials are being lost on combustion the metal content activity increases leading to the alkalinity.

As can be seen from the table, the moisture content decreases from 1.97% to 0.01 as the carbonization temperatures increases. The lower the moisture content, the lower the degree of defect arising from shrinkage during curing at elevated temperature.

The iodine adsorption number as shown in Table 1 reveals a progressive increase as the carbonization temperatures increases. The iodine adsorption number elicits the surface area of the material. An increase in the surface activity results in higher modulus at higher strain, higher abrasion resistance and lower hysteresis (Blow, 1971). The bulk density decreases with carbonization temperature. The density is influenced by the particle size and structure of the fibre and the lower the particle size, the lower the density and therefore, the better the interaction between the polymer matrix and the reinforcing fibre.

The results of the mechanical properties of the composite are shown in Table 2. In all the composite, tensile strength, modulus at 100% and hardness increases with increasing temperatures of carbonization. This is as a result of the increase in surface area which leads to more interaction between the filler and the rubber matrix.

From the results in the table, it is seen that the values of elongation at break decreases with increasing carbonization temperature. The decrease in elongation at break has to do with adherence of the filler to the polymer phase leading to the stiffening of the polymer chain. The stiffening of the chain lead to high resistance to stretch when strain is applied (Gent and Campion, 1992).

The abrasion resistance shows a progressive increase with carbonization temperatures of the fillers. This observation may be as a result of the degree of dispersion of the fillers.

The compression set results as presented in Table 2 shows a progressive decrease with the carbonization temperatures up to 500oC before it start rising again. The compression set is affected by the affinity of the rubber for the filler surface and primarily a function of surface energy of the filler (Nemour, 1986).

Table 1: Characteristics of the powdered filler

Temperature Of carbonization	**0**	**100**	**200**	**300**	**400**	**500**	**600**	**700**	**CB(N300)**
Loss on ignition (%),	6.00	6.80	28.30	57.20	65.40	74.00	77.00	82.00	-
Moisture content (%)	1.97	0.95	0.42	0.12	0.08	0.04	0.01	0.01	0.90
pH of slurry at 28^oC	4.70	5.60	5.90	7.10	7.60	8.00	8.20	8.40	6.00
Iodine adsorption Number (mg/g)	32.50	52.00	53.50	56.72	57.20	60.00	57.00	62.10	82.60
Bulk density(g/ml)	0.79	0.71	0.64	0.68	0.62	0.58	0.60	0.61	-

Mechanical Properties of the Composites

Table 2:**MECHANICAL PROPERTIES OF THE COMPOSITE**

Properties	Filler Loading	0^{o}C	100^{o}C	200^{o}C	300^{o}C	400^{o}C	500^{o}C	600^{o}C	700^{o}C	Carbon Black
Tensile strength	30 Phr	5.20	6.00	6.50	7.00	7.65	7.89	7.70	7.96	24.63
(MPa)	50Phr	5.45	7.14	7.30	7.83	9.00	10.62	10.20	10.98	30.00
Elongation	30Phr	490.00	480.00	472.50	440.00	485.00	380.00	375.00	450.00	465.00
At beak (%)	50Phr	486.40	430.00	380.00	350.10	360.00	311.00	366.20	468.20	324.00
Modulus	30Phr	1.20	1.42	1.41	1.92	2.40	2.80	3.20	3.10	5.33
(%)	50Phr	1.28	1.46	2.00	1.85	2.60	2.90	3.60	3.48	6.78
Flex fat-	30Phr	8910	8675	6672	5860	5700	5720	4770	3890	3804
Igue kc	50Phr	8060	7360	6330	5360	4867	4890	7000	4720	4280
Hardness	30Phr	37.00	41.10	50.00	52.10	55.60	56.00	57.80	59.00	58.60
IR HD	50Phr	38.68	55.60	58.00	59.20	61.20	62.10	60.00	59.30	63.00
Abrasion	30Phr	17.00	20.00	22.50	25.00	28.10	32.20	35.40	35.10	40.60
Resistance	50Phr	19.22	20.80	23.60	27.00	31.80	40.20	42.50	38.60	42.20
Compression	30Phr	27.00	23.41	19.00	17.00	15.20	13.20	13.50	14.20	16.80
Set %	50Phr	22.50	15.22	14.20	11.10	10.60	9.80	10.00	10.21	8.98

TABLE 3: % SWELLING OF THE COMPOSITES.

Carbonization temperature	Benzene (%)	Toluene (%)	Xylene (%)
0^{0}c	279	395	395
100^{0}c	273	320	325
200^{0}c	245	316	320
300^{0}c	235	308	310
400^{0}c	230	300	300
500^{0}c	225	289	295
600^{0}c	225	285	293
700^{0}c	214	280	285

In the solvent resistant test result in Table 3, the effect of Benzene on the test samples was the least. In all the three solvents, the percentage swelling decreases with carbonization temperature. This may be as a result of the increase activities of the fillers which increases the crosslink density.

CONCLUSION

The main aim of this work is to find out the effects of filler carbonization temperatures on the mechanical properties of natural rubber composites and also to compare the results with that of N330 carbon black filled natural rubber composite. The preliminary results show that carbonized groundnut shell has the potential semi-reinforcing effect on the rubber composites.

The results indicates that mechanical properties of composites are largely influenced by the carbonization temperature and load of the filler; and are therefore significant factors in determining the filler type to use and the application of the composite. It also shows that the mechanical properties of the carbonized groundnut shell filled composites are quite comparable to that of N330 carbon black filled composite.

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