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Mechanical properties of natural rubber filled with carbonized dikanut shell (*Irvingia wombolu*) and carbonized rubber seed shell (*Hevea brasiliensis*)

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ABSTRACT

Samples of dikanut shell and rubber seed shell were carbonized at varying temperatures (100, 200, 300, 400, 500, 600, 700 and 800°C) for three hours each and sieved through a 100µm screen. The portion of the dikanut shell and rubber seed shell carbon that was passed through the screen was characterized and used in compounding natural rubber. The mechanical properties of the DNSC-filled vulcanizates present better tensile strength, modulus at 100%, abrasion resistance, compression set and hardness as carbonization temperature increases while the RSSC-filled vulcanizates exhibits better elongation at break and flex fatigue resistance.

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KEYWORDS

Dikanut;
Rubber seed;
Carbonization;
Vulcanizate;
Shell;
Cross-linking.

INTRODUCTION

The search for means and methods of improving the properties and processing of rubber products dates back to over a century ago^[1]. One way of achieving these extensions of service life of these rubber products is the incorporation of additives into the polymer matrix^[2]. The use of agricultural by-products as fillers for renewable polymer additives tried by several authors is drastically taken its position in the polymer industry^[1]. Fillers, as one of the major additives used in natural rubber compounds are either used raw or modified^[2]. These fillers function to modify the physical and to some extent, the chemical properties of the vulcanizates^[3]. In this research

work, the dikanut shell and rubber seed shell were carbonized at various temperatures to see the effects of carbonization temperature on the filler properties and on the Mechanical properties of the vulcanizates^[3].

OBJECTIVE

- To investigate the effect of carbonized dikanut and carbonized rubber seed shells on the mechanical properties of the rubber vulcanizates.
- Combating agricultural by-products as a route to renewable polymer additives.
- To compare the reinforcing potentials of the fillers used.

EXPERIMENTAL

Materials and method

Nigeria standard rubber of grade NSR-10 and rubber seed shells were obtained from Rubber Research Institute, Iyanomoh, Benin City. The dikanut shell were obtained from Leventis farms, Agenebode, Edo State while Zinc oxide, processing oil, Tetramethyl thiuram disulphide (TMTD), Mercarpto benzothiazole sulphenamide (MBTS), Stearic acid, Sulphur, Trimethylquinoline (TMQ), Sodium sulphate (99.5%), Analytical grade potassium iodide and N330 carbon black produced by British Drug House (BDH) were obtained from Rovet Chemicals, Benin City.

Apparatus

- Monsanto Tensile tester model 1/m was used to test the tensile properties^[4].
- Wallace Hardness tester model c8007/25 was used for hardness test^[5].
- Wallace Akron abrasion tester was used for abrasion test^[19].
- Du Pont machine was used for flex fatigue test^[6].
- Muffle Furnace METTm-525 was used for the carbonization of the dikanut shell and rubber seed shell^[5].
- Two Roll Mill was used in mixing the rubber composite^[8].
- Hydraulic press was used for curing the rubber composites^[8].

Filler carbonization

The dikanut shell and rubber seed shells were washed in water and dried in air to removed sand particles and moisture respectively. After drying, half of the dikanut shell and rubber seed shell were milled to fine powder as well as the carbonized portion, and sieved through a mesh size of 100 μ m^[14]. The fine particles that passed through were collected and used for compounding. The fillers were carbonized at varying temperatures from 100, 200, 300, 400, 500, 600, 700 and 800°C^[7,10].

Processing of the composites

The formulation used for the compounding of the

natural rubber (NSR 10) with the dikanut shell and rubber seed shell fillers is shown in TABLE 1.

A batch factor of six was used to multiply the weight of the ingredients in parts per hundred of the rubber^[3,8].

TABLE 1 : Formulation for compounding natural rubber.

Ingredient	Parts per hundred rubber
Natural rubber	100
Fillers (DNS/RSS)	40
Zinc Oxide	5.0
Stearic acid	2.5
Sulphur	1.5
MBTS	1.5
TMTD	3.5
Processing Oil	5.0

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 follows ASTM D 3184-80 Standard^[8].

TABLE 2 : Mixing steps and mixing time

Mixing steps	Time (minutes)
Natural rubber mastication	5
Addition of Stearic acid	1
Addition of Zinc Oxide	1
Addition of filler	10
Addition of MBTS	1
Addition of TMTD	1
Processing Oil	1
Addition of Sulphur	2
Total	22

Compounded composite curing

The curing of test pieces was done in a compression moulding machine. The curing was carried out at 130°C for 25 minutes^[3].

Mechanical properties of the vulcanizates

The mechanical properties carried out include; tensile properties,

Hardness test, Compression set, Flex fatigue and Abrasion resistance respectively^[15].

RESULTS

TABLE 3 : Mechanical properties of the vulcanizate

Properties	N330 carbon	Carbonization Temperature (°c) at 40phr Filler Loading								
		Uncarbonized	100	200	300	400	500	600	700	800
Tensile strength (Mpa)	29.20	(6.00) [5.80]	(7.10) [6.20]	(8.00) [7.35]	(8.20) [7.46]	(10.40) [8.10]	(12.11) [9.20]	(12.80) [11.40]	(12.45) [11.00]	(11.90) [10.98]
Modulus at 100%	6.30	(1.25) [1.20]	(1.70) [1.63]	(2.50) [2.34]	(2.58) [2.49]	(2.70) [2.65]	(2.74) [2.89]	(3.60) [3.50]	(3.48) [3.40]	(3.28) [3.10]
Elongation at break (%)	324.10	(489.40) [500.00]	(468.50) [485.00]	(450.40) [474.20]	(438.65) [450.10]	(420.50) [432.10]	(379.10) [386.07]	(365.20) [324.10]	(372.10) [375.00]	(456.20) [476.00]
Flex fatigue (kcx10 ³)	4.58	(8.89) [9.20]	(8.20) [8.70]	(7.30) [7.42]	(6.45) [6.75]	(6.20) [6.40]	(5.00) [5.90]	(5.00) [5.88]	(5.86) [6.00]	(6.40) [7.22]
Hardness (IRHD)	58.60	(40.90) [40.00]	(43.60) [42.00]	(44.20) [43.02]	(50.94) [50.00]	(51.20) [50.40]	(54.10) [53.60]	(58.00) [57.80]	(57.85) [57.40]	(57.60) [57.20]
Abrasion Resistance (%)	40.60	(19.90) [19.00]	(20.00) [19.20]	(21.40) [20.00]	(24.80) [24.10]	(28.00) [27.25]	(39.80) [34.00]	(41.20) [41.00]	(40.00) [39.20]	(37.25) [35.60]
Compression set (%)	8.61	(19.35) [19.20]	(19.80) [19.00]	(16.80) [16.40]	(14.40) [14.20]	(12.10) [12.00]	(11.65) [10.40]	(8.80) [8.70]	(9.00) [8.95]	(10.10) [9.01]

Key : Dikanut shell Carbon (), Rubber seed shell Carbon []

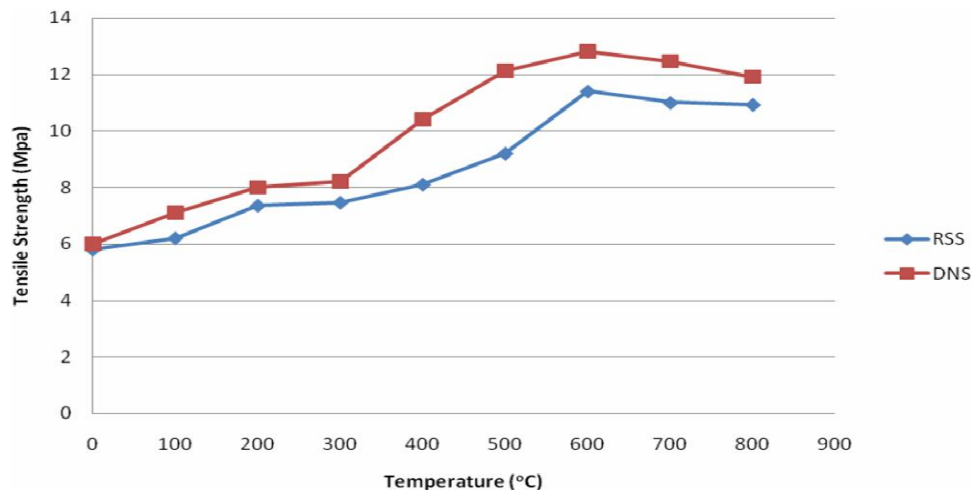


Figure 1 : Tensile strength of DNSC-filled and RSSC-filled vulcanizate

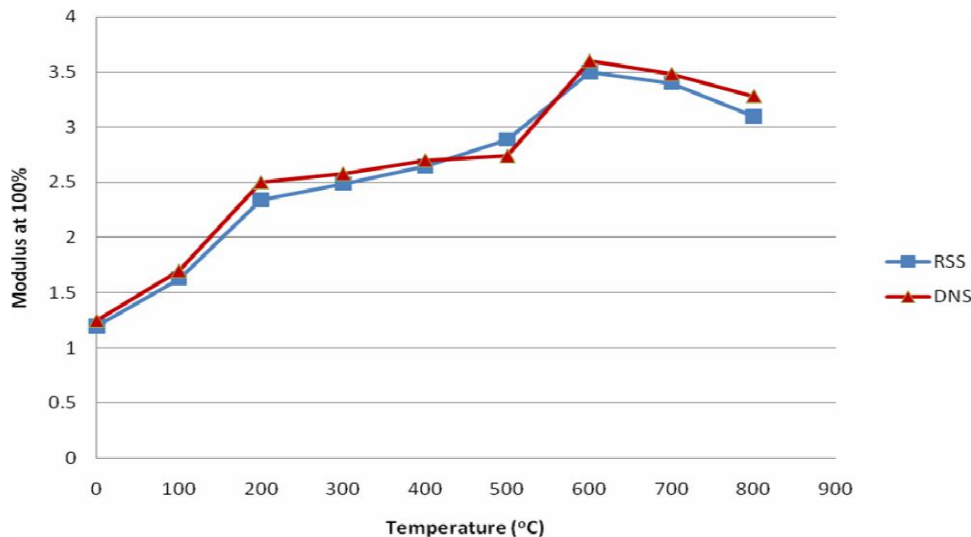


Figure 2 : Modulus at 100% of DNSC-filled and RSSC-filled vulcanizate

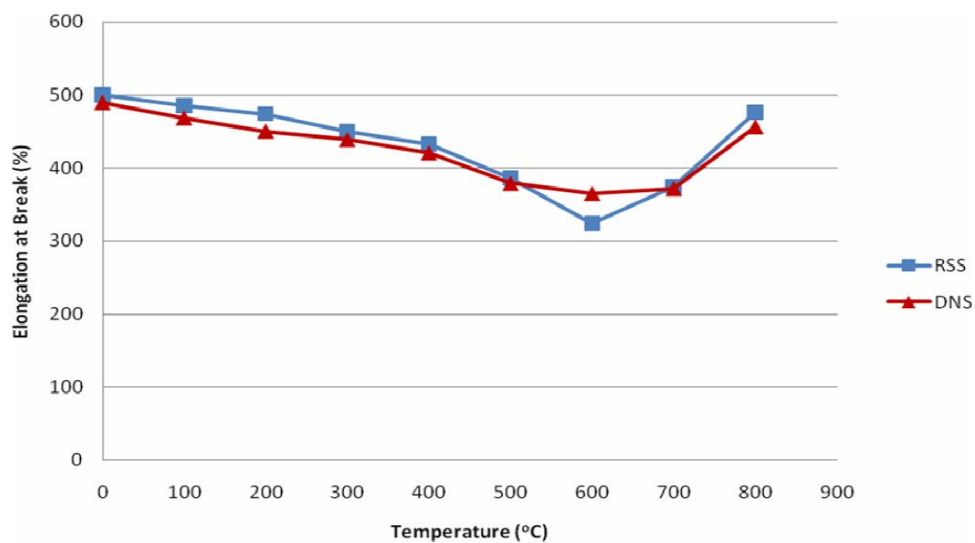


Figure 3 : Elongation at break of DNSC-filled and RSSC-filled vulcanizate

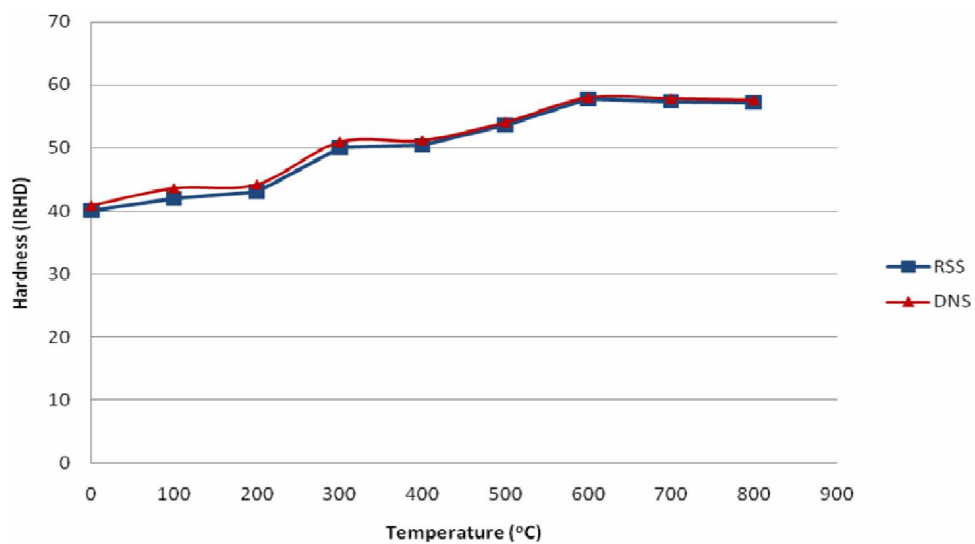


Figure 4 : Hardness of DNSC -filled and RSSC-filled vulcanizate

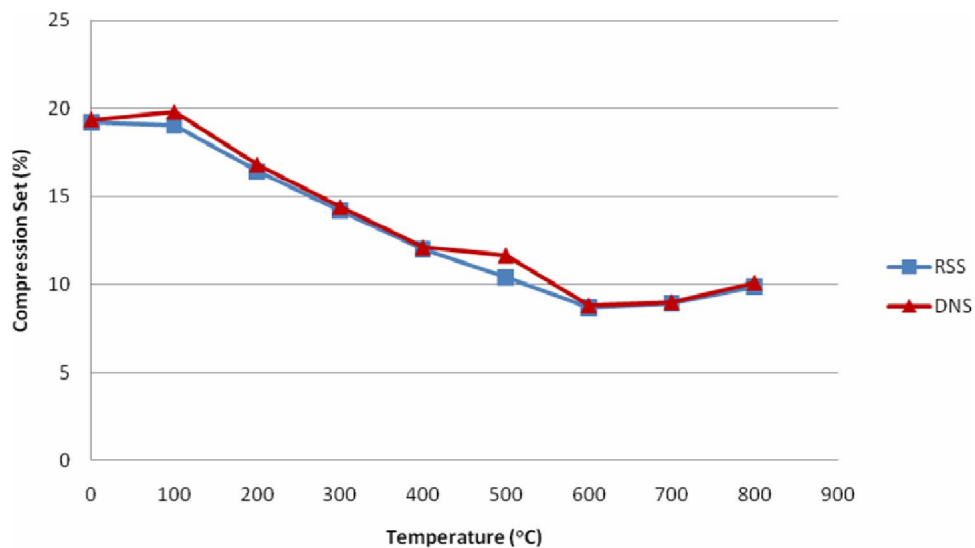


Figure 5 : Compression set of DNSC-filled and RSSC-filled vulcanizate

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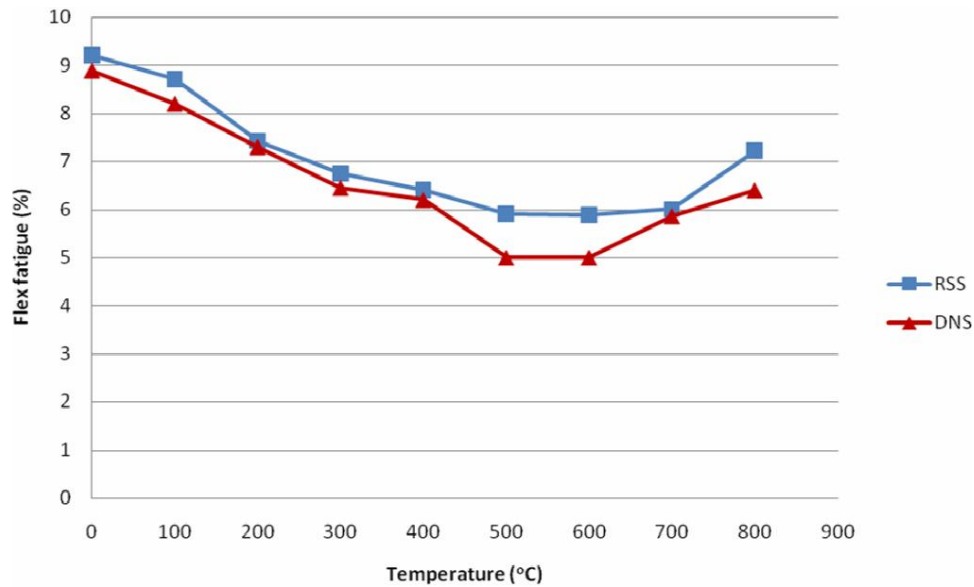


Figure 6 : Flex fatigue of DNSC -filled and RSSC-filled vulcanizate

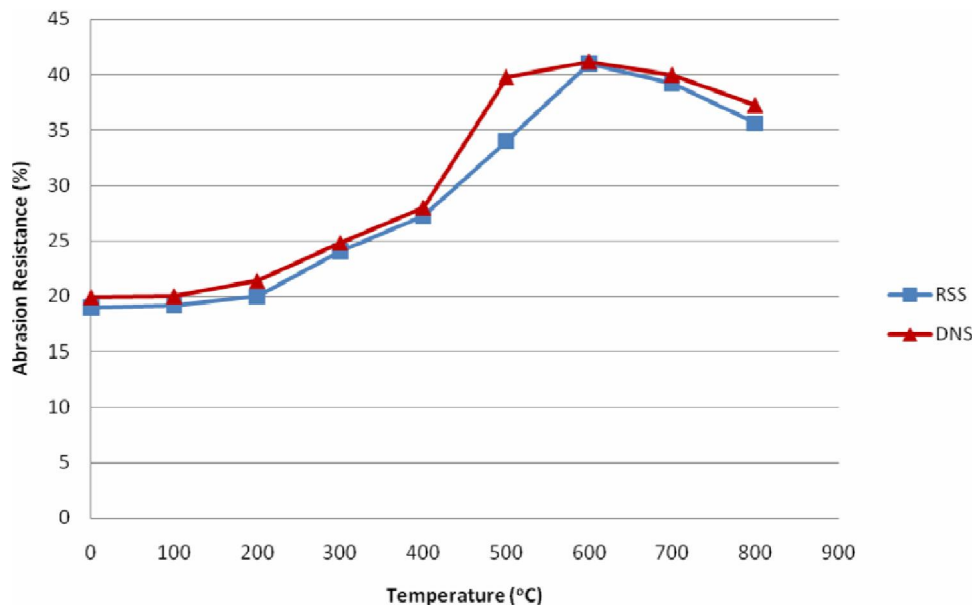


Figure 7 : Abrasion resistance of DNSC-filled and RSSC-filled vulcanizate

DISCUSSION

Natural fibres are derived from lignocelluloses containing strongly polarized hydroxyl group, hence hydrophilic in nature. Most fibres contain cellulose, lignin, water-soluble compounds, waxes, hemicelluloses etc., where lignin, hemicelluloses and celluloses are the major constituents^[9]. The hydrophilic nature of fibres causes the fibre to swell considerably and ultimately rotten through fungi attack. The major causes of this biodegradation are the presence of hemicelluloses in

natural fibres, whereas lignin is prone to Ultra-violet degradation but thermally stable^[9]. Carbonization is aimed at curbing this short coming posed by natural fibres for use in rubber vulcanizates.

In all filled system as shown in Figure 1 and Figure 2, a gradual increase in tensile strength as well as modulus with carbonization temperature of the filler is noticed up to 600°C. It clearly indicates that as the temperature increases, there is a noticeable increase in tensile strength and modulus for both RSSC-filled and DNSC-filled vulcanizates^[7,13]. It may be mentioned here that both tensile strength and modulus are important for recommend-

ing any vulcanizate for structural applications.

The increase in tensile strength and modulus is as a result of high surface area of DNSC compared to RSSC, suggest better polymer filler interaction and hence enhanced better tensile properties for the DNSC-filled Vulcanizate than the RSSC-filled. The factors that affect the reinforcing potentials of fillers include filler dispersions, surface area, surface reactivity, bonding capacity (quality), particle size between the filled and the elastomeric matrix^[10].

The modulus data in TABLE 3 and Figure 2 showed increase with filler carbonization temperature for DNSC and RSSC up to 600°C before showing a marked decrease with temperature above 600°C. The fact that DNSC has a higher modulus than RSSC suggest that fillers are more reinforcing when properly adhered into the polymer matrix^[18].

The results in TABLE 3 and Figure 3 shows that elongation at break (EAB) decrease with increasing filler carbonization temperature of the mixes for all the fillers below 600°C, above which there was a gradual rise in the value of EAB. A decrease in EAB 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 the strain is applied. DNSC-filled vulcanizates has less values of EAB as temperature increases compared to RSSC-filled^[13].

The hardness of DNSC-filled and RSSC-filled vulcanizates increased with increasing carbonization temperature. However, above 600°C the trend took a different direction as a result of the filler characteristics. This result is expected because as more filler particles get into the rubber, the elasticity of the rubber chain is reduced, resulting in more rigid vulcanizates. The hardness results of DNSC-filled vulcanizates are higher than those of RSSC-filled as shown in TABLE 3 and Figure 4 because of the more adherence of DNSC carbon to rubber matrix^[7,13].

The results of compression set in TABLE 3 and Figure 5 show that as filler carbonization temperature increases, the compression of filled vulcanizates decreases for both DNSC-Filled and RSSC-Filled vulcanizates up to 600°C before it start rising again. This observation is connected with the degree of filler dispersion and its particle size which may have enhanced the DNSC-filled vulcanizates^[7,9]. However, at 400°C,

600°C and 700°C, the value of compression set of both the DNSC-filled and RSSC-filled were relatively close when compared^[20].

The values of flex fatigue decreases with increasing carbonization temperature of the mixes for all the fillers below 600°C before it starts rising again presented in TABLE 3 shown in Figure 6. A decrease in flex fatigue 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^[13,14,18].

The abrasion resistance of a solid body is defined as its ability to withstand the progressive removal of the material from its surface as a result of the mechanical action of rubbing, scraping or erosive nature^[18,19]. The trend of abrasion resistance with carbonization temperature of filler presented in TABLE 3 and Figure 7 show a regular pattern of increase with increasing the filler carbonization temperature for both DNSC-filled and RSSC-filled vulcanizates. This indicates that filler carbonization temperature is a function of the measured parameter attributed to the degree of dispersion of the fillers. However the abrasion resistance of the vulcanizates decreases above 600°C, hence DNSC-filled has better abrasion resistance when compared with RSSC-filled vulcanizates^[9,10].

CONCLUSION

The main aim of this research work is to examine how the filler carbonization temperature of the dikanut shell and rubber seed shell may influence its characteristics properties and hence the mechanical properties of natural rubber vulcanizates^[12]. The initial result shows that DNSC is more reinforcing filler for natural rubber compound than RSSC. The reinforcing potential of DNSC is best compared to that of N330 carbon black in some measured properties. The results indicate that mechanical properties of the vulcanizates are greatly influenced by filler carbonization temperature and are therefore significant factors in determining the application in rubber compounding^[12,13]. The result also predicts the potential applications of DNSC as low cost filler when compared with RSSC in natural rubber products. The vulcanizates exhibit high quality characteristics at filler carbonization temperature (600°C). This in-

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dicate that for high quality vulcanizates using both the dikanut shell and rubber seeds shell as reinforcing fillers, carbonization should be done at 600°C for 3 hours^[7].

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