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PHYSICO-MECHANICAL PROPERTIES OF RUBBER SEED SHELL CARBON – FILLED NATURAL RUBBER COMPOUNDS

Samples of rubber seed shells were carbonized at varying temperatures (100, 200, 300, 400, 500, 600, 700 and 800 °C) for three hours each and sieved through a 150 μm screen. The portion of the rubber seed shell 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 the surface area increases with the increases of the heating temperature, unlike the bulk density which decreases. The compound mixes were cured using the efficient vulcanization system. Cure characteristics of compounds and physico-mechanical properties of the vulcanisates were measured as a function of filler loading along with that of N330 carbon-black filled natural rubbers. The results showed that the cure times, scorch times and the torque gradually increased, with increasing the filler content for rubber seed shell carbon-filled natural rubber, with the filler obtained at carbonizing temperature of 600 °C tending to show optimum cure indices. The physico-mechanical properties of the vulcanisates increase with filler loading. The reinforcing potential of the carbonized rubber seed shell carbon was found to increase markedly for the filler obtained at the temperature range of 500-600 °C and then decreases with further increase in temperature.

Key words: rubber seed shell carbon; natural rubber; physico-mechanical properties; vulcanisates.

Fillers in rubber compounds play a major role in physico-mechanical properties and exercise control over cost of the products [1]. It is well established that carbon black is one of the most important classical reinforcing fillers, especially for the rubber industry. However, carbon black is expensive and petroleum from which it is derived is non renewable [2]. Therefore, considerable research and development efforts are being carried out to investigate the possibility of replacing this filler with renewable raw materials as fillers [3].

The use of carbon from agricultural by-products (maize cob groundnut husk, cassava peel, cocoa pod husk, plantain peel, rubber seed shell, etc.) for producing vulcanisate materials that are competitive with synthetic composites has been gaining attention in the last decade because of availability of materials, easy processing, low cost, high volume applications and less abrasive to equipment [3-4].

Agricultural residues as by-products and co-products of agriculture and processing of agricultural products represent a large feedstock of underutilized resources which can be used directly or converted by fairly simple chemical processes into higher value added materials.

Rubber seed shell is an agricultural by-product of the rubber tree. The economic importance of the rubber tree has largely focused on the rubber latex 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, [5-6] there is little information on the potential for the application of these by-product as an extender and/or filler in the processing of polymers [7-8].

Previous studies [5-8] reveal that the temperature at which carbonization of agricultural by-products is carried out could affect the characteristics of the carbon obtained and therefore, the physico-mecha-

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nical properties of the rubber vulcanisates. However, the present research work was undertaken, with an objective to explore the influence of carbonizing temperature of the rubber seed shell on the physico-mechanical properties of carbon and on the rubber reinforcement potential of carbon along with the commercial grade N330 carbon. The N330 carbon black was chosen because of its availability and quality of final products.

MATERIALS AND METHODS

Rubber seeds were obtained from the Rubber Research Institute of Nigeria, Iyanomon, Benin City, Nigeria. Natural rubber used for the study was procured from the Famad Rubber Factory, Benin City, Nigeria. All other reagents used were of commercial grade, while the industrial grade carbon black (N330) filler was obtained from the Warri Refinery and Petrochemical Compony, Warri, Nigeria.

Preparation of the rubber seed shell carbon

The rubber shells were separated from the seeds, air-dried and reduced to small sizes. Eight samples of 1 kg each were weighed and heated to temperatures: 100, 200, 300, 400, 500, 600,700, and 800 °C and then next three hours using the METM-525 Muffle furnace. The carbonized shells were then milled to fine powder and sieved through a mesh size of 150 μm. The carbon particles that passed through the screen were collected, characterized and used for compounding.

The characterization of the rubber seed shell carbon and the N330 carbon black

The rubber seed shell carbon (RSSC) and the N330 carbon black were characterized as follows: loss of ignition was determined gravimetrically [9], the moisture content was determined by the method described in ASTM D 1509 [10], the bulk density was determined according to the method described in ASTM D [11], the pH was determined by using ASTM D 1512 method [12], the method used for the surface area measurement of the fillers is the iodine adsorption number [13], the conductivity was determined by using the pH-conductivity meter. The calcium and magnesium contents of RSSC were determined by complexometric titration, while the sodium and potassium contents were determined by flame photometry and are given in Table 1.

Preparation of the natural rubber vulcanisate

The formulation of mixes is shown in Table 2. Natural rubber was masticated on the mill for five minutes followed by addition of the ingredients. An efficient vulcanization system was chosen. The Vulcanisate materials were prepared in a laboratory two-roll mill (160×320mm) maintained at the temperature below 80 °C through an attached water cooling system.

Table 1. Mineral content of the rubber seed shell carbon

Element	Content, %
Magnesium	0.673
Sodium	0.014
Potassium	0.01
Calcium	0.32

Table 2. Formulation for compounding natural rubber; the recipe for compounding of the natural rubber (NSR 5) with RSSC for each sample

Ingredient	Phr
Natural rubber	100
Filler (RSSC/CB)	20/40/60
Stearic acid	4.0
Zinc oxide	2.0
ZMBT	3.5
Processing oil	2.0
Sulphur	0.5

Cure charactristics of natural rubber compounds

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

Determination of vulcanisate properties

The curing of test pieces was done by compression molding. The curing was carried out at 140 °C. The tensile strength, modulus and elongation at break were measured by using a Monsanto instron tensometer in accordance with ASTM D412-87 method A [14]. Dumbbell test pieces of dimension (45 mm×5 mm×2 mm) were used. Compression set of vulcanisates were determined by using the method described in ASTM D385 [15].

The hardness of the rubber vulcanisate was determined using the Wallace hardness tester model C8007/25 in accordance with ASTM 1415 [16]. The abrasion resistance measurement was based on DIN to ISO 4649 Akron to BS 903 Part 49 method C [17]. The measurement of the flex fatigue was carried out in accordance with the procedure described in ASTM D430 [18], using the Du Pont model C82075.

RESULTS AND DISCUSSION

Characteristics of the rubber seed shell

The characteristics of the rubber seed shell carbon, as well as the characteristics of N330 carbon black, are given in Table 3. The trend of pH of the rubber seed shell carbon as a function of the carbonizing temperature given in Table 3 varied over a range of 4.79-8.77. The results show a progressive increase in pH with the increase of carbonization temperature. The possible reason for the trend in the pH of carbon could be the result of the metal content of RSSC presence of which was shielded by the influence of inorganic volatiles present as the carbonization was being done at lower temperature. However, pH at acidity level tends to slow the cure rate and hence reduce the cross link density which informs the choice of fast accelerator and activators in the mixing formulation. The pH of the carbon black is 6.50, which is of close value to that of the rubber seed shell carbon at 600 °C.

Table 3 shows also that the electrical conductivity of RSSC increases with the increase of temperature. When the temperature reaches 600 °C, the resistance in RSSC becomes very small, meaning good conductivity, while when above 600 °C the conductivity descended slightly. The reason for this is probably that the volatiles in RSSC completely released at this temperature. The Conductivity of the carbon black N330 from the table is quite superior to that of the rubber seed shell carbon, suggesting a very low resistance and very good conductivity which could be the result of the particle size and the conducting power of its ions in the solution. High conductivity in N330 based elastomer composites is primarily due to its structure and the ability had by N330 to retain that structure even when dispersed in elastomer matrix.

The bulk density of the RSSC samples given in Table 3 varied between 0.611-0.785 g/ml. Bulk density is principally influenced by the particle size and structure of the fiber and the lower the particle size, the lower is the bulk density and therefore there is a better the interaction between the polymer matrix and a reinforcing fiber, which will thus enhance the vulcanisate processing and improve the quality of the final product as desirable properties for fibers include excellent tensile strength and modulus, high durability, low bulk density, good moldability and recyclability [19]. Table 3 shows that at high temperature, the bulk density reduces showing that the interstitial spaces (micro pores) in the carbon residue is opened and thus resulting in easy compaction and interaction with the polymer matrix.

The iodine adsorption number from the table reveals that the amount of iodine adsorbed per 100 gram of the material increases with the increase of the filler carbonizing temperature. One important application of iodine adsorption number is that it elicits the surface area of the material and indicates the macrostructure of the filler and reflects its reaction and adsorption abilities [19]. Like wood fiber, at high temperature, all kinds of porosities will form inside RSSC, which bring RSSC a certain specific surface area, reaction and adsorption capacity. The maximum iodine adsorption number (66.75mg/100g) represents the adsorbed iodine which is formed when the carbonizing temperature reaches 600 °C; eliciting the fact that the maximum surface area occurs at this temperature; the surface area value is much smaller when carbonizing at low temperature (200 °C) due to the low porosity resulting from incomplete carbonization. At higher temperature (>600 °C), the porosity reduces too and the reason might be that some cavities have been burned and the surface area corresponding re-

Table 3. Characterization of the rubber seed shell carbon

Temperature °C	100	200	300	400	500	600	700	800	CB (N330)
Yield, %	92.9	70.9	40.7	25.9	23.0	22.4	17.5	16.8	NA
pH of slurry at 28 °C	4.79±0.021	5.17±0.020	5.30±0.022	5.75±0.030	6.16±0.021	6.36±0.020	7.68±0.023	8.77±0.0225	6.50
Conductivity $\Omega^{-1}m^{-1}$	31.90	43.40	181.40	173.50	135.80	261.0	226.0	245.0	288.50
Bulkdensity g/ml	0.755±0.0214	0.646±0.020	0.785±0.015	0.678±0.012	0.635±0.010	0.691±0.0214	0.667±0.001	0.611±0.056	ND
Iodine adsorption number mg/100g	20.12±0.060	36.32±0.057	47.20±0.058	50.34±0.060	61.24±0.062	66.75±0.063	65.11±0.062	57.24±0.060	80.78
Loss on ignition, %	7.1	29.1	59.3	74.1	77.0	77.6	82.5	83.2	92.85

duced. So, when the heating temperature reaches 800 °C, the surface area value is small too.

It can be seen from Table 3 that the loss on ignition percentage increases from 7.1 to 83.2% with the increase of the heating temperature of the rubber seed shell, the loss on ignition percentage increased rapidly with the increase of the temperature up to 800 °C. This might be caused by almost completed volatilization of the volatile matter at the temperature above 600 °C. The loss on ignition of N330 carbon black is 92.85% from the Table 3, which is higher than that of the rubber seed shell carbon suggesting a high amount of carbon present and hence better reinforcement of natural rubber than RSSC.

Cure characteristics

The data on the processing characteristics of RSSC filled natural rubber systems being evaluated are given in Table 4. The analysis of the Monsanto rheograph facilitates the determination of various cure related parameters. From the rheographs there is the evidence of torque increase with increasing filler loading and filler type as with N330 carbon black as seen in figure 3. Torque values provide direct information on the extent of cross linking in the rubber compounds. The results showed that cure times, the scorch and the maximum torque gradually increased with increasing filler content for RSSC-filled natural rubber, as can be seen in Figures 1-3. However, for CB-filled natural rubber, the scorch and the cure times decreased while the maximum torque increased with increasing filler content. The trend observed in the cure characteristics may be attributed to differences in the filler properties. The cure enhancement of the vulcanisates can be associated with the filler related parameters such as surface area, surface reactivity, particle size, and moisture content. In general, a faster cure rate is obtained with filler having a higher pH and high moisture content [20]. There are many factors that affect the cure time of rubber compounds. The temperature, a curing system and thickness are the most important factors that affect cure

Table 4. Cure characteristics of the natural rubber filled with rubber seed shell carbon in comparison with carbon black N330

Parameter	Filler loading	F100	F200	F300	F400	F500	F600	F700	F800	N330
	20	36	22	14	12	14	16	10	11	34
Scorch time, s	40	40	26	20	18	22	25	12	16	42
	60	50	34	22	24	27	29	22	20	38
	20	68	180	180	185	185	200	185	180	42
Cure time, s	40	76	162	179	190	210	255	210	200	60
	60	96	197	200	200	240	285	220	210	52
	20	4.01	6.03	6.78	7.00	7.25	7.55	4.91	4.75	11.04
Tmax-Tmin (kg cm)	40	4.23	8.75	7.06	7.48	7.95	8.00	5.39	5.25	11.39
	60	5.39	9.50	10.89	11.05	11.35	11.50	7.48	6.03	12.86
	20	3.13	0.63	0.60	0.58	0.58	0.54	0.57	0.59	12.5
Cure rate index, %	40	2.78	0.74	0.63	0.58	0.53	0.43	0.51	0.54	5.56
	60	2.17	0.61	0.56	0.57	0.47	0.39	0.51	0.53	7.14

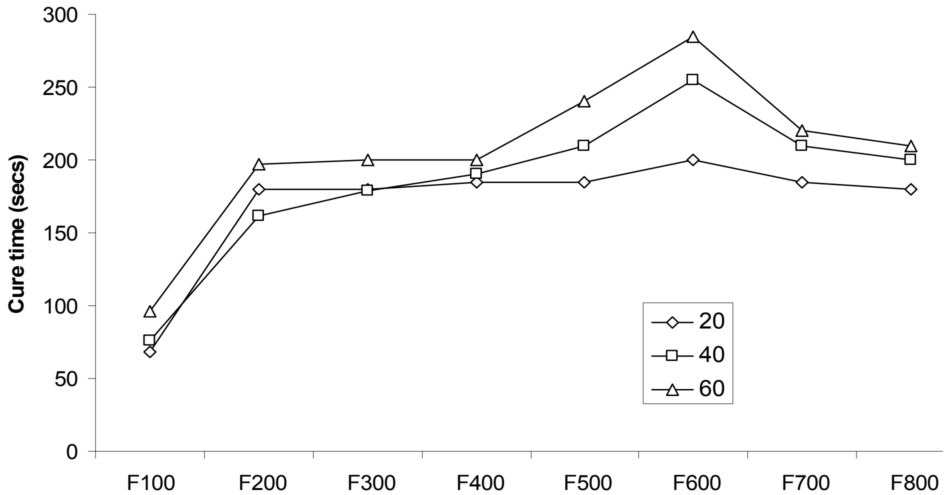

Figure 1. Variation of cure time with filler loading and filler type.

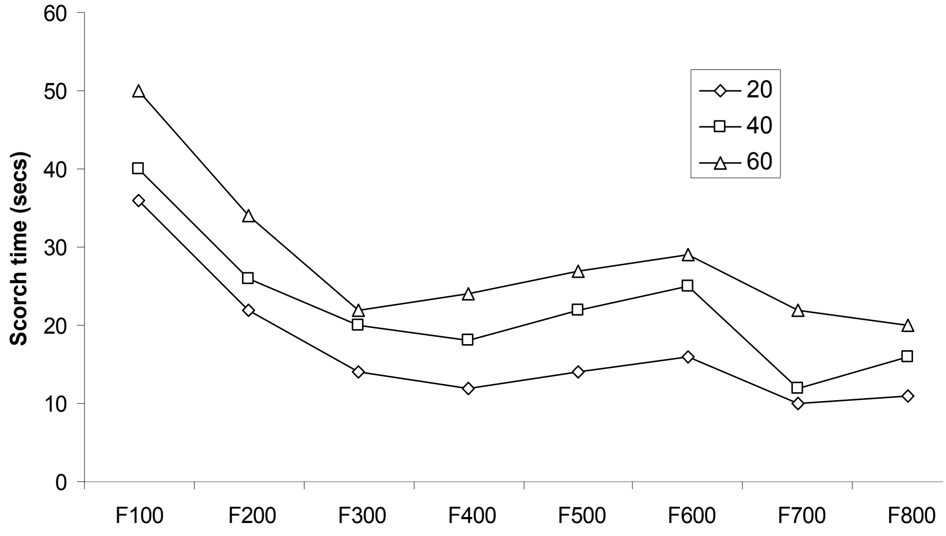

Figure 2. Variation of scorch time with filler loading and filler type.

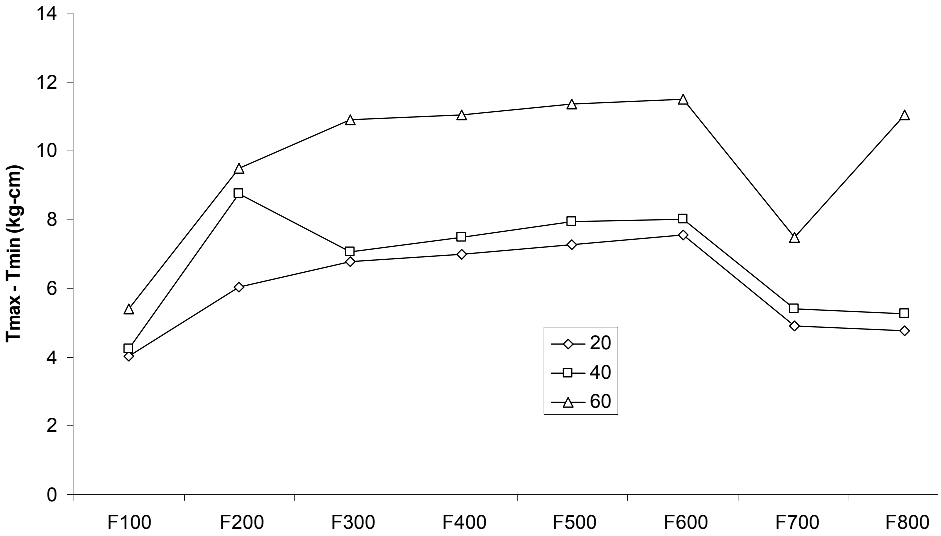

Figure 3. Variation of change in torque with filler loading and filler type.

time. It has been reported that cure rate is directly related to the humidity and water content of the compound mix [21]. However, in the present study the most probable factors to be taken into account for the observed cure enhancement are surface area, moisture content, and pH. The marked increment in the torques observed for the compound mix shows that the presence of the filler in the rubber matrix has reduced the mobility of the macromolecular chains of the rubbers.

Physico-mechanical properties of the natural rubber vulcanisates

The mechanical properties of the RSSC-filled natural rubber vulcanisate and N330 carbon black are shown in Table 5. A gradual increase in tensile strength, as well as modulus with the weight fraction of the filler is noticed up to 600 °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 RSSC-filled natural rubber vulcanisates. It may be mentioned here that both tensile strength and modulus are important for recommending any vulcanisate as a candidate for structural applications. In all filled systems, tensile strength and modulus at 100% increase with increasing the filler type and content. The iodine adsorption value of carbon black N330 from Table 3 shows that the N330 carbon black is higher in terms of the surface area than RSSC, suggesting more polymer- filler interaction and hence enhanced tensile properties for the CB-filled Vulcanisate than the RSSC-filled product.

Table 5. Mechanical properties of the natural rubber vulcanisates

Property	Filler loading	F100	F200	F300	F400	F500	F600	F700	F800	CB (N330)
Tensile strength	20phr	5.41	6.13	6.50	7.01	7.65	7.98	7.77	6.19	21.60
MPa	40	6.14	7.22	7.33	7.89	9.11	10.82	10.00	8.26	29.00
	60	6.90	8.80	9.32	10.40	11.23	14.55	13.98	10.40	32.08
Modulus at 100%	20phr	1.40	1.40	1.94	2.38	2.81	3.27	3.20	2.39	4.33
	40	1.44	2.03	1.72	2.64	2.89	3.60	3.41	3.28	6.30
	60	1.62	2.22	2.44	3.27	4.33	5.96	5.11	3.00	8.43
Elongation at	20phr	560.02	513.20	513.10	510.19	410.10	362.01	375.12	458.13	525.02
break, %	40	481.04	476.07	448.10	490.11	381.07	301.04	366.08	476.07	324.07
	60	430.05	381.01	349.03	354.14	311.10	263.05	275.11	490.11	251.10
Flex fatigue	20phr	nf	nf	nf	nf	nf	nf	3.11	3.36	-
($K_c \times 10^3$)	40	nf	nf	nf	8.70	7.86	6.31	7.00	7.22	
	60	8.72	7.36	6.67	6.33	5.86	3.36	4.72	6.89	
Hardness	20	35.22	37.30	32.00	34.24	37.22	41.10	41.30	38.56	45.20
IRHD	40	41.00	41.02	49.00	50.00	50.10	55.60	55.50	42.40	58.60
	60	43.01	53.00	56.50	57.89	59.11	61.33	60.02	51.05	60.50
Abrasion	20phr	17.02	20.03	21.17	24.09	27.18	35.24	35.11	31.21	39.54
resistance	40	19.22	20.12	23.41	27.01	33.00	42.70	39.10	34.68	40.60
	60	21.01	25.21	27.01	39.08	40.96	44.41	40.45	38.50	41.22
Compression set	20phr	27.10	23.41	19.20	17.11	15.22	13.02	13.75	15.10	16.34
%	40	19.20	15.22	13.02	11.55	10.02	8.64	8.98	10.42	8.61
	60	16.10	12.76	10.25	9.61	8.66	6.30	7.34	10.21	6.25

The results show that maximum values of tensile strength and modulus at 100% are obtained for filler type F600 at the loading of 60 phr. The factors that affect the reinforcing potential of the fillers include filler dispersions, surface area, surface reactivity, particle size and bonding quality between the filled and elastomers matrix. The modulus data showed a decrease with the increase of the filler loading above F600. According to Table 5, the values of the tensile strength and modulus at 100% for N330 carbon black show that as the filler loading increases the tensile strength and modulus also increase suggesting that these could be the result of the high surface area and loss on ignition of the N330 carbon black.

The values of elongation at break (EB) decrease with the increase in filler type and content of the mixes for all the fillers below F600 and also for N330 carbon black. A 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 the strain is applied [4,7].

The results in Table 5 show that an increase in filler loading of the vulcanisates increases its hardness value. 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 vulcanisate. Hardness increases with the increase of the filler loading for N330 carbon black vulcanisate.

The compression set results in Table 5 show that as the filler type and loading increases, the compression of filled vulcanisates decreases for both the RSSC-filled and the N330 carbon black (CB)-filled vulcanisates. The observation may not be unconnected with the amount of the filler incorporated into the matrix, the degree of dispersion of the filler and its particles size which may have enhanced the CB-filled vulcanisates.

The abrasions resistance of a solid body is defined as its ability to withstand the progressive removal of the material from its surface as the result of a mechanical action of the rubbing, scrapping or erosive nature. The trend of abrasion resistance with loading is presented in Table 5; it shows a regular pattern of the increase with increasing the filler type and loading for both RSSC -filled and CB-filled vulcanisates. This indicates that filler loading is a function of the measured parameter. This observation may therefore be attributed to the degree of dispersion of the fillers.

The values of flex fatigue decreases with the increase in filler type and content of the mixes for all the fillers below F600. 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.

CONCLUSION

The main aim of this work is to examine how the filler carbonizing temperature of the rubber seed shell may influence its characteristics properties and hence the mechanical properties of natural rubber vulcanisates and the possibility of a comparative study of the mechanical properties of the vulcanisates with that of carbon black. The preliminary results show that carbonized rubber seed shell is potential reinforcing filler for natural rubber compounds. The results indicate that mechanical properties of vulcanisates are greatly influenced by filler carbonizing temperature and loading and are therefore significant factors in determining the application in rubber compounding. The vulcanisates exhibit high quality characteristics at filler type F600 (that is at filler carbonized at 600 °C) and with 60 phr loading. The conclusion is that for high quality vulcanisate using Rubber seed shell as the reinforcing filler, carbonization should be done at 600 °C for 3 h. It also shows that mechanical properties of the RSSC vulcanisates are quite comparable to those of CB vulcanisates, particularly in terms of hardness, compression set and abrasion resistance.

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NAUČNI RAD

FIZIČKO-MEHANIČKA SVOJSTVA KAUČUKOVIH SMESA NA BAZI PRIRODNOG KAUČUKA I ČAĐI DOBIJENIH OD LJUSKI SEMENA KAUČUKOVCA

Uzorci ljuski semena kaučukovca su karbonizovani na različitim temperaturama (100, 200, 300, 400, 500, 600, 700 i 800 °C) u vremenu od 3 sata i prosejani kroz sito od 150 μm. Deo aktivne čađi dobijene iz ljuske semena kaučukovca koji je prošao kroz sito je okarakterisan preko vrednosti gubitka žarenjem, specifične površine, sadržaja vlage, pH, gustine i sadržaja metala, i iskorišćen za dobijanje smesa na osnovu prirodnog kaučuka. Karakterizacija je pokazala da vrednosti pH, provodljivosti, gubitka žarenjem i specifične površine rastu sa povećanjem temperature dobijanja, dok vrednost gustine opada. Smese su podvrgnute vulkanizaciji primenom efikasnog umrežavajućeg sistema. Karakteristike umrežavanja i fizičko-mehanička svojstva vulkanizata praćena su u zavisnosti od količine punioca uporedo sa vulkanizatima na osnovu prirodnog kaučuka i čađi tipa N330. Rezultati su pokazali da vreme vulkanizacije, skorč vremena i obrtni moment postepeno rastu sa povećanjem količine punila, pri čemu punilo dobijeno karbonizacijom na 600 °C daje optimalne indekse vulkanizacije. Fizičko-mehanička svojstva vulkanizata se povećavaju sa povećanjem količine punila. Ustanovljeno je da sposobnost ojačanja za čađi dobijene od ljuski semenki kaučukovca znatno raste ako je to punilo dobijeno u opsegu temperatura 500-600 °C i da opada sa daljim povećanjem temperature dobijanja.

Ključne reči: čađ ljuski semena kaučukovca; prirodni kaučuk; fizičko-mehaničke osobine; vulkanizati.