

## The study of nanostructure and phase composition of silicon-carbon fillers produced from the rice hulls for elastomers

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The outcomes of numerous experiments on studying the dynamics of changes in macro- and microstructures and phase transformations of solid products from the rice hulls pyrolysis depending on temperature and applying them in production of various rubbers have indicated that the produced silicon-carbon nanocomposites are perspective fillers for elastomers.

### 1. INTRODUCTION

The employment of small-size functional objects called nanoparticles, nanostructures, nanoclusters, etc. is the basic tendency in the development of the state-of-the-art technology. In recent years, the interest in them has greatly grown, which arose from the prospects as to their wide common use when producing advanced materials with the unique properties: mechanical, optical, magnetic and some others.

By reason of nanoparticles' size control and reproduction, the following two trends [1] were outlined in regard to their preparation and application:

- Nanoobjects synthesis in small numbers (mainly for preparing sensory materials and nano-electronic devices);
- Synthesis of nanomaterials applied in large volumes (fillers, composites, highly dispersed sorbents, etc.).

Within the bounds of the latter trend, preparation of nanostructure carbon- and siliceous materials, the field of application which is greatly multipurpose, deserves special attention. However, the large-scale application is hampered by

costs of their production. The problem comes from the raw material base expansion, advancement of techniques for synthesis of nanomaterials under consideration, etc.

In this regard, a number of investigations were outlined aimed at preparing carbon nanostructures from the materials of vegetable origin [2, 3]. The large-volume rice-production waste – the rice hulls (RH) – appears to be greatly perspective raw stuff in this context.

As it was indicated previously [4, 5], the rice hulls represent a complex aggregate of organic and mineral components. Determination of the quantitative chemical group composition indicated that in the rice hulls of the Kyzylorda Oblast the cellulose content was 33 mass%, hemicellulose – 18 mass %, lignin – 26 mass %, substances soluble in alcohol-benzol blend – 2 mass % and in cold and hot water – 5 and 6.8 mass % respectively. The major part of mineral ingredients (up to 14 mass % by dry matter equivalent) falls at silicon dioxide.

According to the literature the rice hulls, recovery of which is the critical mission for all countries of the planet engaged in growing and processing the rice culture, are generally discussed as the unique raw material for producing high-purity silicon dioxide, silicon carbide and silicon [6-8]. The problems of producing the nanostructure silicon-carbon material from the rice hulls and thorough study of this material for its future application particularly as a filler for elastomers were not reviewed. At the same time the rice hulls, which contain two elements of prime importance C and Si, are the potential source for preparing silicon-carbon nanocomposite that may be successfully used when producing rubber of various purposes instead of traditional technical carbon and white carbon.

Thus, preparing and studying of the silicon-carbon nanomaterial from the rice hulls are of current concern both from scientific and application-oriented points of view.

## 2. EXPERIMENTAL SECTION

To prepare the nano-fillers for elastomers, the rice hulls were subjected to the thermal decomposition in the off-gas atmosphere in the temperature range 500÷1000 °C at the heating rate 15 °C/min within 30 min.

The raster electron microscopy (REM), high-resolution transmission electron microscopy (TEM) and X-ray phase analysis (XRPA) were applied to study the macro- and microstructure of silicon-carbon materials and also their phase composition.

The REM involving the electron probe microanalysis was carried out with the use of the microprobe analyzer *Superprobe 783* (JEOL, Japan). The analyses and photographing in secondary electrons and back-scattered (composition) electrons

were performed assisted by the energy-dispersive spectrometer Inca Energy (Oxford Instruments, England).

The TEM was carried out with the transmission electron microscope Philips EM 301 (The Netherlands) at the accelerating voltage 80 kV within the range of the electron microscopic magnification 13–80 thousand times. For exposure the samples powdered in the agate mortar were laid on the object copper mesh pre-coated with the substrate film of the amorphous carbon and fixed in the microscope object holder [9].

The XRPA was performed on the computerized diffractometer DRON-2 with the updated collimation on the filtered  $\text{CuK}\alpha$ -radiation by the procedure [10].

### 3. RESULTS AND DISCUSSION

The data for yield and composition of the rice hulls pyrolysis products are presented in Table 1, which shows that the silicon-carbon material yield decreases with the temperature rise, therewith the organic carbon content in it remains practically constant (~52.0%) while silicon dioxide content increases from ~31.7 to ~35.7%.

Tab. 1. Yield and composition of the solid product from the rice hulls thermal destruction.

Temperature, °C	Yield, %	Content, %					
		C	SiO <sub>2</sub>	Ash	H	N	O
500	43.0	52.0	32.1	36.6	3.5	1.1	6.8
650	41.9	51.7	31.7	38.5	3.4	0.8	5.6
800	38.8	52.0	35.5	40.0	2.5	0.7	4.8
1000	38.0	51.6	35.7	40.3	2.0	0.9	5.2

As follows from the REM data (Figure 1), the rice hulls maintain their topology even at relatively high temperature action (up to 800 °C) most probably due to high content of silicon, which as seen by cluster of the light points in Figure 2c is mainly found in the outer and inner tissues of epidermis and also localized in definite sites of the inner cells (in the thick walls of sclerenchyma) [6]. However in this case the chemical elements redistribution takes place. As a result of the thermal destruction of the rice hulls organic component, a peculiar carbon skeleton is formed (Figure 3b), which is uniformly filled with the siliceous phase, (Figure 3c) (oxygen distribution (Figure 2d; 3d) replicates the form of the silicon distribution that is indicative of their interrelation).

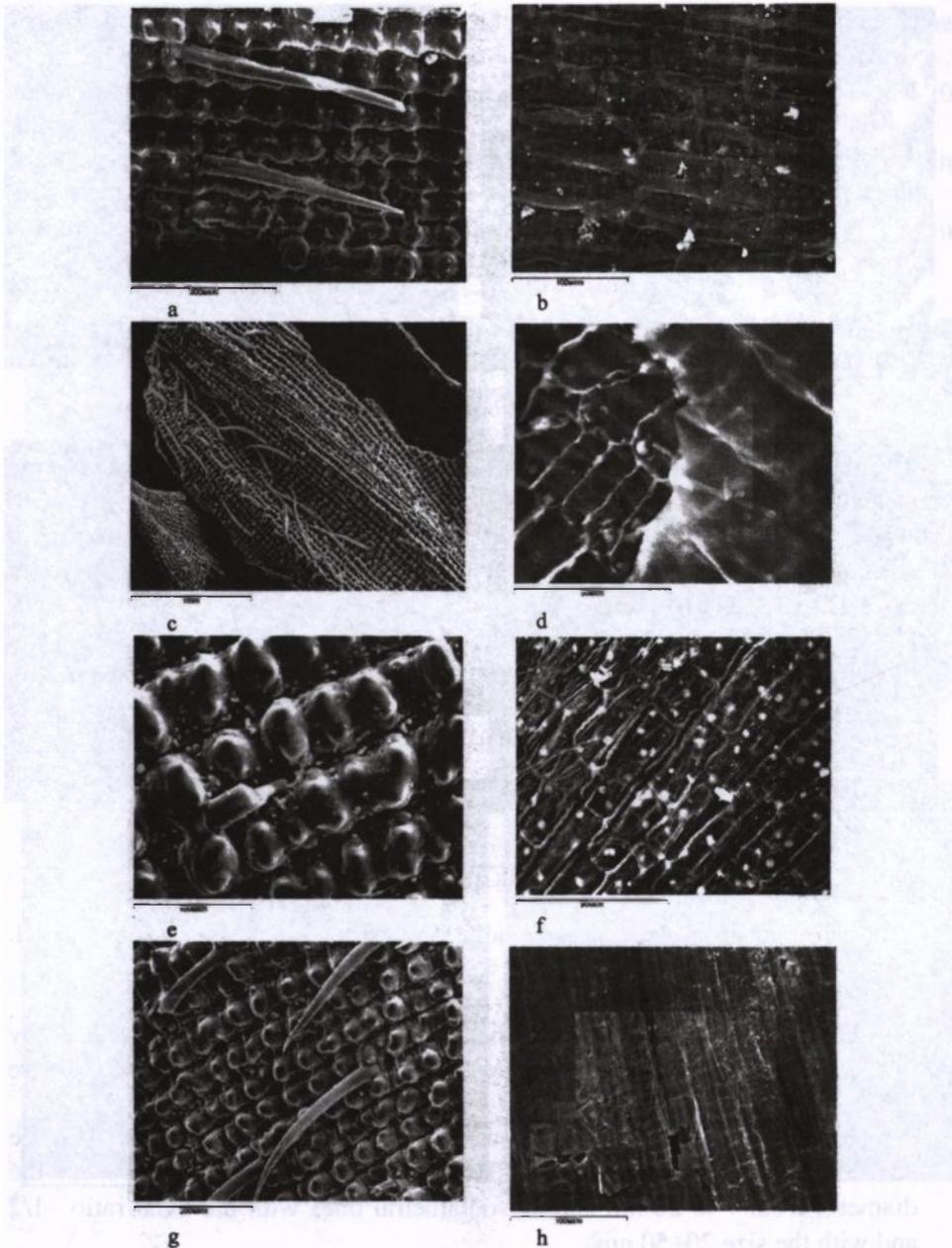


Fig. 1. Screen-type patterns for macrostructure of the initial and carbonized rice hulls: a, c, e, g – the outer side of the initial RH and carbonized RH at 500, 650, 800°C respectively; b, d, f, h – the inner side of the initial RH and carbonized RH at 500, 650, 800 °C respectively.

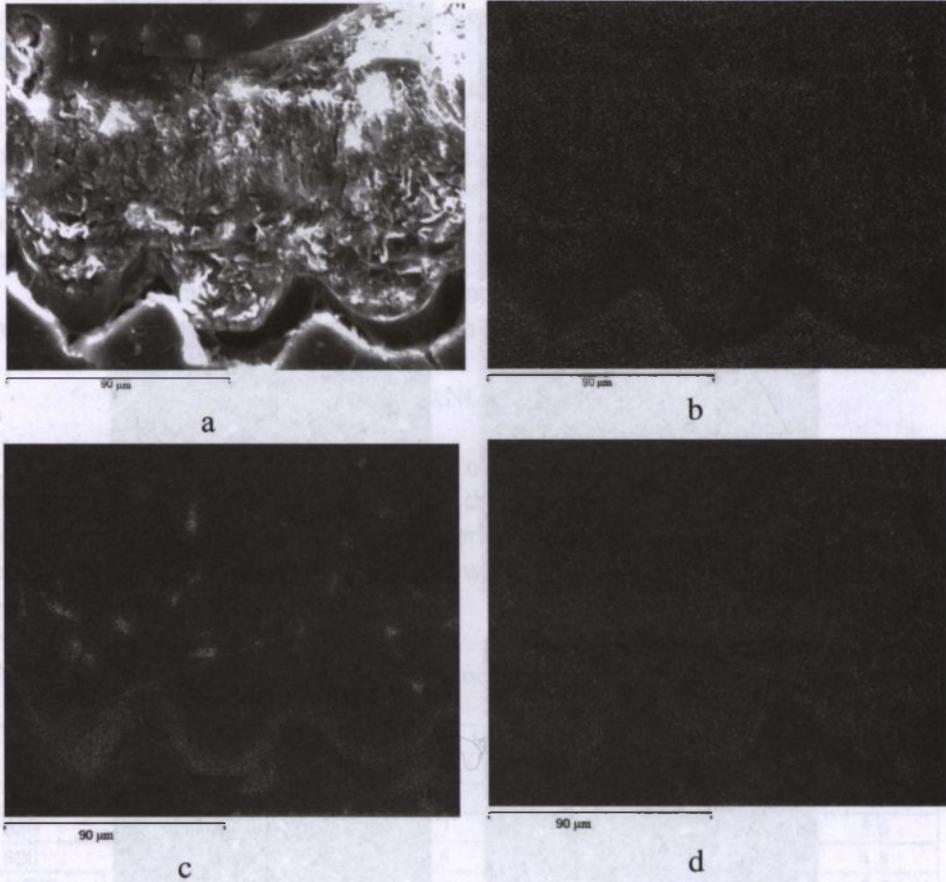


Fig. 2. Microstructure of the initial rice hulls (cross section): a – cross section of RH; b – distribution of C; c – distribution of Si; d – distribution of O.

The TEM results indicated that carbonizates of the rice hulls were formed by particles with different morphostructure among which the following types were defined:

1. Porous formations with well-defined straight lines by fracture edges (Figure 4a). The pores vary in form and size: there are spherical pores with the diameter from 7 to 20 nm and anisodiametral ones with the axial ratio  $\sim 1/2$  and with the size  $20 \div 50$  nm.
2. Compact particles of various forms (Figure 4b).
3. Hybrid structures that represent combination of two phases: laminar formation of the carbon phase penetrated with denser dispersed phase, probably of silicon dioxide (Figure 4c, d). The supposed siliceous phase is presented by

dense particles of the size of  $10\div 20$  nm, which are spread out along the translucent carbon particle.

4. Aggregates formed by translucent particles of round shape of the size  $15\div 20$  nm (Figure 4e).
5. Tubular-form carbon with the diameter from 25 nm to 40 nm (Fragment A in Figure 4f).
6. Prolate crystals of tabular type, translucent and with good faceting, which run in the diameter up to  $25\div 150$  nm (Fragment B in Figure 4f).

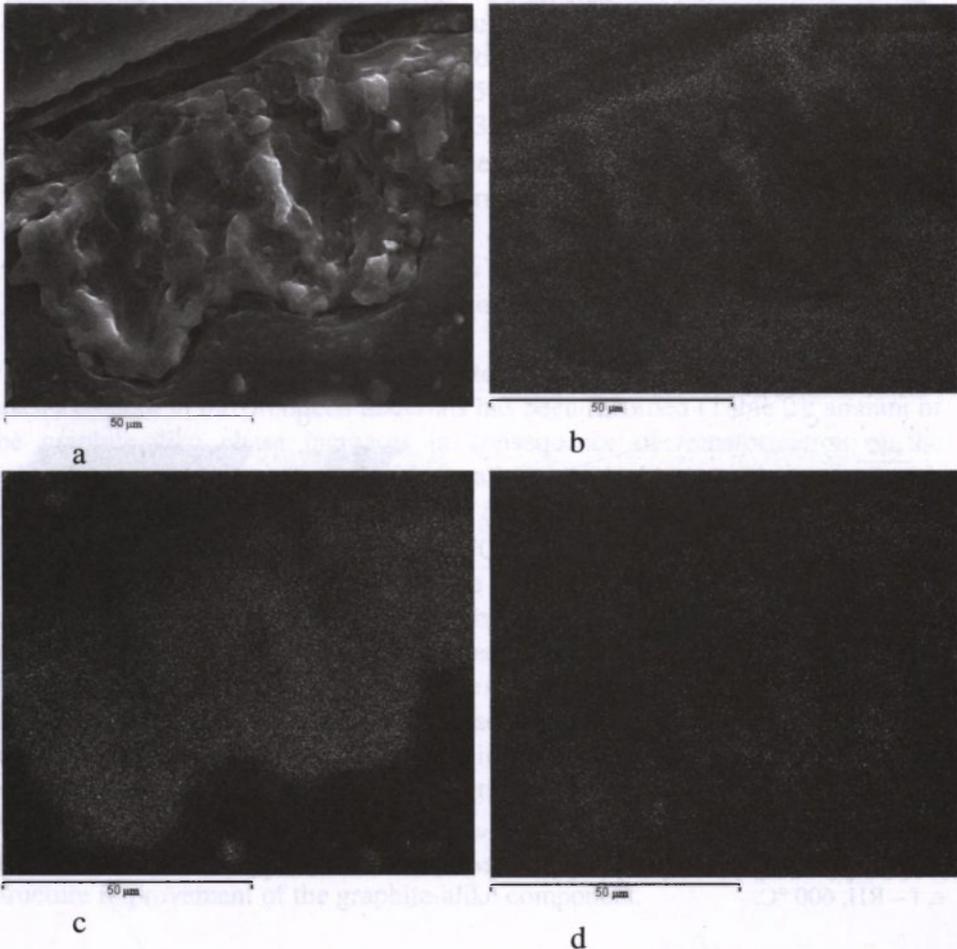


Fig. 3. Microstructure of carbonized rice hulls (cross section): a – cross section of RH, 650 °C; b – distribution of C; c – distribution of Si; d – distribution of O.

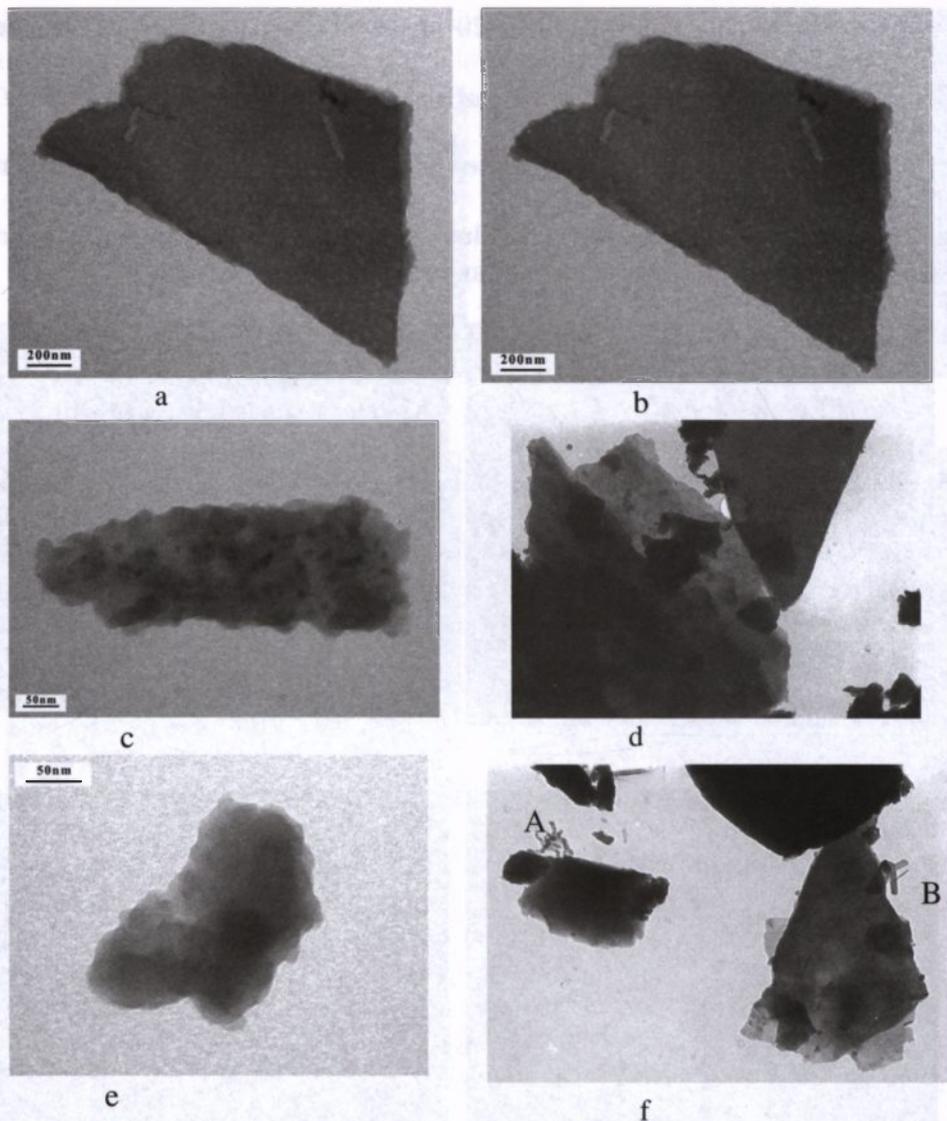


Fig. 4. Micrographics of particles of the rice hulls carbonizates: a, c – RH, 1000 °C; b, d, e, f – RH, 600 °C.

By the X-ray phase analysis method, the presence of the carbon phase (G) and two hydrocarbon phases were detected in the rice hulls carbonizates' composition (Figure 5). One of the revealed hydrocarbon phases is polynaphthenic phase (N) diagnosable by the main reflection with  $d \sim 0.47$  nm

which represents the clathrate structure composed of condensed and separated by methylene bridges naphthene cycles containing alkane chains. The structure of the second hydrocarbon phase (H) with  $d \sim 0.8$  nm was unidentified failing this phase in a pure form [10].

The nanostructure of the carbon phase (G) is presented by crystallites with the cross sectional dimension  $L_{002} \sim 2-3$  nm, which were composed as analogous graphite grids with spacing C–C 0.142 nm [11]. This structure is less perfect as compared with the graphite structure:

- The maximum (002) relating to the interlayer reflection is shifted towards small angles in relation to this maximum of graphite that is due to increase in the mean interlayer distance  $d_{002}$  for polycondensated grids of the RH carbonization products ( $d_{002} = 0.375-0.380$  nm) in comparison with the graphite interlayer distance ( $d_{002} = 0.3354$  nm);
- The line (100) characterizing two-dimensional within-layers reflection begins to appear slightly on the X-ray patterns of the samples heat-treated only at 800°C and higher;
- The diffraction lines with the indices (hkl) that meet the three-dimensional order are absent on the X-ray patterns of the RH thermal destruction products.

With increasing the RH pyrolysis temperature, clear dependence of the phases content in the produced materials has been recorded (Table 2): amount of the graphite-alike phase increases in consequence of transformation of the polynaththenic component, obviously as a less heat-stable one as compared with phase H, the fraction of which holds constant right up to 1000 °C. After treatment of the initial sample at 1000 °C both hydrocarbon phases (N and H) transform into the graphite-alike phase and the amorphous silicon dioxide transfers into cristobalite as evidenced by appearance of the reflections series 0.407; 0.316; 0.249; 0.205 nm in the respective X-ray pattern.

The RH thermolysis conditions also exert influence on X-ray characteristics of G in the produced carbonizates. As seen in Table 3, the temperature rise results in decrease of the parameter  $d_{002}$ , increase in the average thickness of the formation members  $L_{002}$  (it was not practical to calculate the mean diameter of the bedded flat fragments of molecules  $L_a$  for the lack of clear reflections in the area of 21–22 ° $\theta$ ) and graphitization level  $c$ , that is, on the whole it contributes to structure improvement of the graphite-alike component.

Tab. 2. Phase composition of the carbon-containing component in the products from processing the rice hulls.

Pyrolysis temperature, °C	Phase content, %		
	G	N	H
500	45	45	10
650	55	35	10
800	60	30	10
1000	100	N/A	N/A

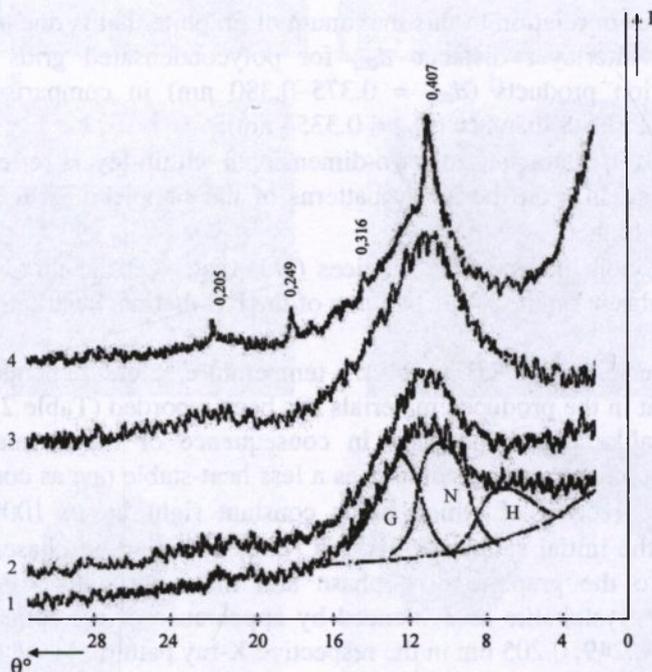


Fig. 5. X-ray photos of the rice hulls carbonizates: 1 – 500 °C; 2 – 650 °C; 3 – 800 °C; 4 – 1000°C.

Pooling the data presented, it is observed that the material produced in the process of the rice hulls carbonization at the temperature up to 800 °C contains amorphous carbon and silicon dioxide; in the product of the RH treatment at 1000 °C silicon dioxide crystallizes in the form of cristobalite. From the position of the structural condition, the silicon-carbon represents a nanocomposite, that is, the object formed by aggregates of various morphologies, which in their turn are formed by close-packed nanoparticles of carbon and silicon dioxide. From the phase composition and parameters of the graphite-alike component, the product under review is similar to industrial carbon black [10], but it is

characterized by a greater content of hydrocarbon phases and less degree of the structure order as indicated by a small size of crystallites and high value  $d_{002}$ .

Tab. 3. The roentgenographic indices of the graphite-like phase of carbonizates from the rice hulls.

Pyrolysis temperature, °C	Parameters for G		
	$d_{002}$ , nm	$L_{002}$ , nm	$c = \frac{L_{002} \times 10^{-2}}{d_{002} - 3.35}$
500	0.380	2.0	0.45
650	0.380	2.3	0.50
800	0.375	2.6	0.65
1000	0.375	3.0	0.75

\*For estimation the degree of graphitization  $c$  in the formula  $L_{002}$ ,  $d_{002}$ , 3.35 are given in Å.

The revealed composition and structural features characterize positively the silicon-carbon material produced by thermal treatment of the rice hulls within temperature interval up to 800°C as a filler for elastomers from the position of technical carbon and white carbon.

It is on record that as of today application of silica fillers in the rubber-processing industry is continuously expanding [12, 13]. It is a current practice with the world manufacturers of tires to apply white carbon in the tire treads with severe requirements as to the tire rolling resistance and road adherence particularly adherence with wet and icy roadway surfaces. But application only of silicic acid as a filler is not so effective in improvement of the tire characteristics for the reason that rubber macromolecule adsorption is obstructed on SiO<sub>2</sub> particles. Generally, to improve technical and especially processing properties of elastomeric composites filled with white carbon, some specially modifying agents are applied, which afford interaction of hydrophobic filler with polymer, that is, so-called agents of white carbon and rubber resin combination [14]. The effect of their action lies in extension of the area of true contact of a filler particle with the polymeric matrix as a result of facilitating the moistening [15].

In view of the fact that the carbon content in the silicon-carbon nanocomposite prevails over silicon dioxide content and there is inevitably strong interparticle interaction between their nanoparticles concealing properties of the isolated particles, it is possible to anticipate that when mixing a silicon-carbon filler with the nonpolar rubber resin in the rubber manufacturing process there will be achieved identically high level "elastomer-filler" interaction both with a carbon component and silica component of the composite under review.

And finally, this will facilitate improvement of physical-mechanical properties of the produced vulcanizates without applying special promoters of white carbon and rubber resin interaction.

In [16, 17], the physical-chemical properties were studied of the silicon-carbon filler produced at 650°C – the most optimal temperature technologically. The outcomes are given in Table 4 [18, 19] in comparison with characteristics of various grades of carbon black. The presented data allow to draw a conclusion that silicon-carbon by the iodine number (56.5 g/kg), which comes out as the specific surface area index, corresponds with technical carbon with the medium degree of dispersion, for example, medium-active technical carbon P 514 (43±4 g/kg). As follows from dibutyl phthalate absorption value (104.00 cm<sup>3</sup>/100) that characterizes structural properties of technical carbon, the material under review outperforms carbon black with this index average value such as P 324 (100±5 cm<sup>3</sup>/100 g), P 514 (101±4 cm<sup>3</sup>/100 g) but similar to P 705 (110±5 cm<sup>3</sup>/100 g) meets the requirements of the grades with high level of structural properties. The pH value indicates that silicon-carbon alike most types of furnace carbon black (P 324 – 7–9, P 514 – 6–8, P 705 – 7.5–9.5) is of weakly alkaline character, and when producing rubber goods it may be used with rubber accelerators of alkaline type.

The silicon-carbon filler of two types: the filler that contained the form of the original rice hulls with the grain size up to 5 mm (granular product) and the filler crushed in the rotary jet-type mill up to 90% size grade -15 mcm (powdered product) – was tested as an alternative filler for various grades of technical carbon and white carbon when producing general rubber goods and tire rubber [20].

In the course of the rubber compounds batching by the mainstream technology, the silicon-carbon, according to expectations, was easily dispersed in the natural rubber matrix. The silicon-carbon application when producing general rubber goods in the case of partial and complete substitution of technical carbon and white carbon and also in combination with them (Tables 5, 7), as seen in Tables 6 and 8, conduces to improvement of technological properties of the rubber mixes and finished vulcanizates: increase in elasticity, improvement of the adhesive behavior and enhancement of the strength properties. Most probably, it is due not only to the common presence but also to the uniform distribution of the nano-size particles of carbon and silicon dioxide present in the active amorphous form. In addition, as the studies demonstrated, the rubber mixes, which are filled with silicon-carbon nanocomposites apparently owing to, as it was stated above, high content in them (against industrial technical carbon) of hydrocarbon phases, were well plasticized that

makes provision for decrease in the plasticizing agent consumption or its complete elimination (Table 5).

Tab. 4. Physical/chemical characteristics of the silicon-carbon filler of rubbers from the rice hulls.

Characteristics	Silicon-carbon	White carbon GOST 18307	Technical carbon GOST 7885			
		BS-100	P 324	P 514	P 705	P 803
Iodine value, g/kg	56.5	-	84±6	43±4	-	-
Absorption of dibutyl phthalate, cm <sup>3</sup> /100	104.00	-	100±5	101±4	110±5	83±7
pH of the aqueous slurry	9.2	7.0–8.5	7–9	6–8	7.5–9.5	7–9
Mass fraction of losses at 105 °C, %	3.4	< 6.5	< 0.9	< 0.9	< 0.5	< 0.5
Ash content, % (Mass fraction of silicon dioxide, %)	45.0 -	- (> 86)	< 0.45	< 0.45	< 0.3 -	< 0.45 -
Bulk density of the granular carbon, kg/m <sup>3</sup>	418	For granulated 220–320	> 340	> 340	-	-
Light transmittance of toluene extract, %	99.00	-	> 85	> 85	-	-

Tab. 5. Mix formulation of the rubber compound for producing the general rubber goods.

Ingredients	Mix ingredients weight, kg				
	Standard 1	Sample 1	Sample 2	Sample 3	Sample 4
Natural rubber SKS-30 ARKM-15	0.500	0.500	0.500	0.500	0.500
Zinc white	0.040	0.040	0.040	0.040	0.040
Technical stearine	0.095	0.0095	0.095	0.095	0.095
Captax	0.010	0.010	0.010	0.010	0.010
White kaolin	0.330	0.330	0.330	0.330	0.330
Carbon black BS-100	0.160	-	-	0.160	0.160
Technical carbon P 803	0.395	0.200	0.200	0.075	0.075
SC granular	-	0.355	-	0.320	-
SC powdered	-	-	0.355	-	0.320
Plasticine	0.031	0.031	0.031	0.031	0.031
Rubrax	0.050	-	-	-	-
Sulfur	0.016	0.016	0.016	0.016	0.016
Thiuram	0.008	0.008	0.008	0.008	0.008
Total	1.635	1.585	1.585	1.585	1.585

Tab. 6. Physical/mechanical properties of general rubber goods.

Properties	Vulcanized sample				
	Standard 1	Sample 1	Sample 2	Sample 3	Sample 4
Rupture strength, kgf/cm <sup>2</sup>	10	12	12.1	28	35.5
Ultimate elongation at rupture, %	120	78	35	100	82
Relative residual elongation after rupture, %	-	5	4	6	4
Hardness	67-80	77	76	68	77

Tab. 7. Compositions of the rubber mixes on the mass parts basis.

Name of ingredients	Coating for the steel-cored breaker: the interplay rubber for truck tires		Insulation of the auto tire bead	
	Standard 3	Sample 3	Standard 4	Sample 4
SKI-3	100	100	100	100
Polymer sulfur	4.2	4.2	-	-
Commercial sulfur	-	-	4.0	4.0
SAC	-	-	0.7	0.7
Santogard RU-1	0.2	0.2	0.3	0.3
Benzoic acid	-	-	0.3	0.3
PND 20908-040	2.0	2.0	3.0	3.0
Stearic acid	1.0	1.0	2.0	2.0
Resinous hydrocarbon	-	-	3.0	3.0
Octophor M, MA	-	-	2.0	2.0
PN-6Sh	3.0	3.0	2.3	2.3
Oil bitumen "G" ASMG	-	-	6.6	6.6
3V-1	-	-	2.0	2.0
Diafen FP	1.0	1.0	0.5	0.5
Common resin EM-3	2.0	2.0	-	-
Acetonanyl	0.5	0.5	-	-

Modifying agent RU	2.0	2.0	–	–
SAM	0.7	0.7	–	–
Cobalt naphthenate	1.0	1.0	–	–
Technical carbon P 234	50	50	–	–
BS-120	10	–	–	–
Technical carbon P-514	–	–	50	25
SC	–	10	–	25

Tab. 8. Physical/mechanical properties of the rubbers.

Rubber mix	Ductility property, Nominal unit	Conventional modulus at 300%, MPa	Nominal tensile strength, MPa	Specific elongation at rupture, %	Tear resistance, kNm
Standard 2 (GOST requirements)	0.39 (0.30±0.05)	13.0 (12.7±1.96)	23.2 (22.6±2.45)	560 (400±70)	84 (83±1.96)
Sample 5	0.40	13.7	23.3	563	90
Standard 3 (GOST requirements)	0.32 (0.28±0.05)	4.9 (8.6±2.0)	16.5 (17±2)	500 (460±60)	–
Sample 6	0.36	9.1	19.8	505	–

#### 4. CONCLUSIONS

The experimental material presented in this paper on studying the products of the rice hulls carbonization applying the standard test methods used for investigating macro- and microstructures enables to consider them as nanocomposites, that is, as macroscopic objects formed by close-packed nanoparticles of carbon and silicon dioxide, the strong interparticle interaction between which conceals properties of the isolated particles.

The temperature of the initial raw material carbonization entails no changes in the silicon-carbon nanocomposite macrostructure but it exerts a significant influence on the phase composition and microstructure of the nanocomposite components. With the temperature rise, improvement in the carbon particles structure is observed at the level of nanocrystallites and amorphous silicon dioxide crystallizes into cristobalite.

Owing to its structural features, silicon-carbon nanocomposite as a filler for elastomers is easily dispersed in the rubber matrix, improves technological properties of the rubber mixes and strength properties of vulcanizates, as well as conduces to enhancement of adhesive properties of the rubber mixes and improvement of the rubber-to-metal case bond strength.

Deployment of the new nanostructural filler in combination with technical carbon and white carbon will afford opportunity to produce rubber technical articles with the specified properties that can find practical implementation when producing rubber for various purposes. In this case even partial substitution of the standard reinforcing fillers will ensure lowering the production costs of the rubber goods industry due to relatively low cost of silicon-carbon produced from cheap raw material.

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