

Some Advanced Materials from Rice Processing Wastes and their Application in Various Branches of Industry and Agriculture

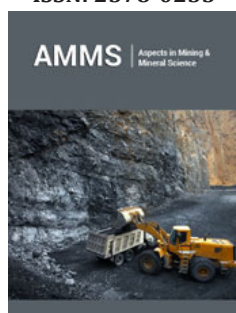
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Abstract

Rice Hulls (RH) are the annually renewable wastes from processing of rice hulls. They are often dumped, and toxic gaseous substances are emitted while the rice hulls decompose. That creates considerable environmental problems. Over 50 thousand tons of rice hulls are annually produced in Kazakhstan. The world volume of the rice hulls generation amounts to 150-180 million tons per year. To address this environmental challenge, the following novel intellectual property methodologies have been proposed for processing waste rice hulls thereby producing new materials such as the solid product silica-carbon. It is a nanocomposite produced by nanoparticles of carbon and silicon dioxide, which were present in the amorphous form and has proven to be advanced filler, sorbent, and fodder supplement for the farm poultry. In addition, this product can be used for producing crystal whiskers B-SiC and polycrystalline silicon. The organic product (aqueous solution of carboxylic acids, phenols, ketones, cyclic aliphatic hydrocarbons, and spirits) is a high-selective collector, plant growth stimulant and an antiseptic agent. During development, it was found that in the temperature range of 300-500 °C three hydrocarbon phases were present in silica-carbon: the stable phase, unstable polynaphthenic phase and resinous phase. At temperatures of 600-700 °C the polynaphthenic phase partially transformed into the carbon phase and resinous hydrocarbons volatilized. Hence, technology was developed to produce silica-carbon with the stable-composition hydrocarbons that met the requirements as to the fillers for hydrocarbon structural materials and elastomers as well as the charge material for production of high-purity silicon.

In summary, silica-carbon is a polyfunctional material and can be used as a filler of elastomers and carbon structural materials, and as a louder additive in poultry farming. Considering the great demand for Kazakhstan and the world in for this material, its production is timely.

Keywords: Rice hulls; Filler for elastomers; Fodder supplement; Sorbent; Stimulant; Antiseptic

Introduction

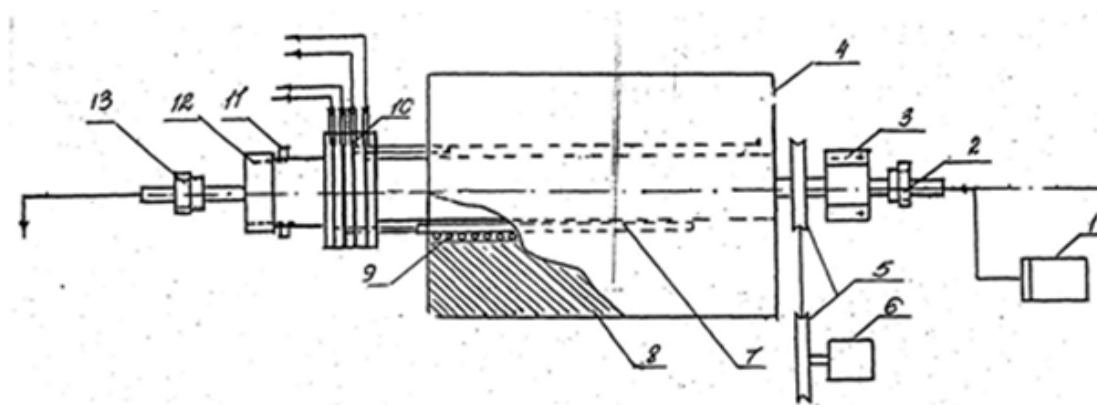
Rice Hulls (RH) are the annually renewable wastes from rice hulls processing. These are dumped where toxic gaseous substances are emitted while the rice hulls decompose in the open air. That creates considerable environmental problems. Over 50 thousand tons of the rice hulls are annually produced in Kazakhstan. The world volume of the rice hulls generation amounts to 150-180 million tons per year. Similar problems are available in every rice growing country (e.g., USA, Russia, China, India, Re-public of Korea, and some others) but there are no efficient technologies for the RH processing implemented on the industrial scale. The recovery of the rice hulls is a topical and complex problem, which the scientists of many countries of the world have been working at over the period of many years. The list of the proven trends in the RH application is so great that this problem should not exist in principle. But none of the proposed technologies have yet had commercial implementation. Consequently, the method of thermal processing is the most prevalent method [1-3].

Experiments and Results

Studies of the RH pyrolysis process were conducted at laboratory plants with automatic temperature maintenance and control. The shaft kiln and tube-type rotary kiln (which best of all simulates the pilot plant operation mode) were used in the temperature range of 500-1000 °C under inert atmosphere and in the reaction products atmosphere within 10-40 minutes. The physicochemical, physical, and functional properties of the silicacarbon (SC) were studied using chemical and phase analyses. The organic chemical composition was determined by applying the gas chromatograph with the mass-spectrometer detector GC/MC Agilent 6890 N/5973N. The separation was performed on the column DB-XLB of 30m in length with the inner diameter 0.25mm and with the film thickness 0.5µm.

According to theoretical assumptions, the RH pyrolysis experiments were performed in the temperature range 500-1000 °C in the reactor without access to air. To create better heat

conductivity conditions, the process was conducted in the stirred layer of the material. The laboratory plant testing was comprised of the rotary tube-reactor with a diameter of 53mm, total length of 450mm and the working zone length of 250mm where the predetermined temperature (500-800 °C) is maintained as seen in Figure 1. Pyrolysis of the material resulted in formation of a solid residue and pyrolysis gases, which represented a mixture of condensable vapor and non-condensable gases (Table 1). The solid product was continuously discharged into the receiving bin. The formed vapor-gas mixture was captured in the condensation system. The condensable substances were accumulated in the collector [4]. To specify the chemical composition, the condensable products were separated into ether- and spirit-soluble-, phenol-, acid- and neutral fractions. It was determined that depending on the pyrolysis temperature the condensate yield and mixture ratio changed within wide range that could be seen from the results given in Table 2.



1. Compressor, 2. Coupling with the bearing part, 3. Support bearing, 4. Metal casing, 5. Pulleys 6. Motor, 7. Thermocouple, 8. Insulation, 9. Heating element, 10. Contacts, 11. Support bearing, 12. Reactor (kiln) 13. Coupling with the bearing part

Figure 1: Rotary tube-type kiln.

Table 1: Composition and yield of the products produced during the rice hulls pyrolysis.

##	Name	Composition and Content, %	Yield at Pyrolysis, %
1	Rice hulls	C-43-45	100
		O ₂ -35-37	
		H ₂ -5-6	
		SiO ₂ -15-18	
2	Silicacarbon	C-52-55	38-39
		SiO ₂ -32-35	
		Hydrocarbon compounds-8-9	
		Inorganic elements impurities-<1	
3	Sublimates, Including the condensate (the organic product)	Phenols-13-15	61-63
		Acids-21-23	
		Ketones-11-13	38-39
		Hydrocarbons-22-24	
		Spirits-4-5	
		H ₂ O-the rest	

Table 2: Yield and composition of the products depending on temperature of pyrolysis.

Pyrolysis Temperature, °C	Condensate Yield, in % of the Rice Hulls	Yield of Fraction, in % of Condensate		Yield of Fraction, in % of Sulfur-Ether Fraction		
		Sulfur-Ether Fraction	Spirit Fraction	Phenol Fraction	Acid Fraction	Neutral Substances
300	12	4.0	0.1	5.0	49	20
350	20	10.2	0.3	10.0	48	22
400	22	39.1	0.4	15.0	47	25
500	35	41.0	0.5	19.0	43	38
800	30	24.5	11.0	15.0	46	29

The data from (Table 2) indicate that with a rise in the pyrolysis temperature up to 500 °C, the yield of condensate and content of phenols and neutral substances increase while the yield of the acid fraction holds constant. When raising the pyrolysis temperature up to 800 °C, a decrease of the total yield of condensate and content of phenols and neutral substances in it is observed. By virtue of the fact that the SiO₂ contained in the rice hulls is in close connection with the organic components, because of the plant tissue thermal destruction, it forms a non-separate structure, and on account of high interparticle interaction with carbon it forms a nanocomposite assembly. Therefore, one may draw the conclusion that the solid product from the thermal processing of the rice hulls represents nothing other than a nanocomposite, i.e., a macroscopic object formed by densely packed nanoparticles of carbon and silicon dioxide, which are present in equal quantities (50-55 and 40-

45% by mass respectively) and uniformly distributed between them [5]. Comparison of the silicacarbon physical and chemical characteristics with the data for various carbon black grades renders, as shown in Table 3, makes it possible to conclude that silica carbon by its iodine adsorption number, (which is the factor of the specific surface area), complies with the carbon black that has the average degree of dispersion of the activated technical carbon P324. The specific surface area of the technical carbon determines the rubber-carbon white interface and consequently, the rubber mixture properties and characteristics of finished rubbers. The most critical characteristic is the impact of dispersion of the activated carbon filler on the strength properties of the vulcanizates. The more dispersiveness of the silica white, the higher is the ultimate tensile strength, tearing resistance and wear-resisting property, and hardness and rigidity of the rubbers.

Table 3: Physical and chemical characteristics of rubber fillers.

Name of Characteristic	Silica-Carbon	Silica White GOST 18307	Technical Carbon GOST 7885		
		BS-120	P 324	P 514	P 705
Iodine adsorption number, g/kg	81.20	-	84±6	43±4	-
Dibutyl phthalate adsorption, cm ³ /100	109.00	143-275*	100±5	101±4	110±5
Before compaction After compaction		80-93*			
pH of the water slurry	8.00	7.0-8.5	7-9	6-8	7.5-9.5

Based on analysis of the physical and chemical properties of the silicacarbon produced from the rice hulls, it can be expected that as a rubber filler with the average factor of dispersion and high structural factor the silicacarbon will be easily dispersed in the rubber matrix facilitating the mixing and improving the technological properties of the rubber. Deployment of the silicacarbon with the optimal selection of ingredients will enable to increase the strength characteristics and enhance the plasto elastic properties of vulcanizates. The amorphous silicon dioxide

occurrence in the silicacarbon can improve the adhesive properties of the rubber mixes and increase the bonding strength with the metal housing. The tests shown in Table 4 have demonstrated that in technical rubbers (e.g. hoses, agricultural plates, etc.) and tire rubbers, the application of the silica carbon instead of the technical carbon (TU P-514) and silica white (BS-120) improves the physical and mechanical characteristics (i.e. dynamic fatigue strength, nominal strength, tear resistance, engineering stress and rubber tack (tread rubber, valve-side rubber, breaker rubber, etc.).

Table 4: Physical/mechanical properties of general rubber goods.

Properties	Vulcanizate Sample				
	Standard 1	Sample 1	Sample 2	Sample 3	Sample 4
Rupture strength, MPa	1.0	1.2	1.2	2.8	3.5
Ultimate elongation at rupture, %	120	78	85	100	110
Relative residual elongation after rupture, %	-	5	4	6	4
Hardness	67-80	77	76	68	77

Based on the investigations conducted and results obtained, the following may be concluded:

a. The granulated silicacarbon material and powdered silicacarbon material produced from the rice hulls can be used as filler for rubber mixtures instead of the use of technical carbon and silica white subject to the requirements specified for physical and mechanical characteristics of the produced rubber articles [6].

b. The application of silicacarbon fillers from the rice hulls renders it possible to eliminate the use of plasticizers in producing the rubber mixtures that can reduce the product cost.

The silicacarbon from the rice hulls was further evaluated to use it as sorbent in various engineering processes. It has been shown that the macroporous SC from the rice hulls as a sorbent is highly active in waste-water treatment from petroleum- and oil contamination. The static volume capacity (SVC)= 50mg/g when recovering 98% from solution that contains petroleum products and oils in the quantity of 200 mg/dm³. The dynamic volume capacity (DVC)=80mg/g when recovering 99.5%. In the process of the precious metal's recovery from the production solutions, by its absorption characteristics on gold the SVC=2.4mg/g when recovering 91.4% from the solution that contained 8.1mg/dm³ of Au⁺ ions. Hence, the activated silicacarbon is equal to the available industrial sorbents: resin AM-2B and coking coals. When recovering rhenium from production sulfurous solutions (pH=2), in one sorption-desorption cycle the SC provides an increase in concentration of rhenium by 2.4 times thereby opening prospects for its application in the sorption technology for rhenium recovery [7]. The use of silicacarbon as the initial raw material for producing silicon carbide composites offers to be the most promising. The materials testing of these shows that their basic functional properties comply with the requirements required of them. Composite materials reinforced with silicacarbon, filamentary crystals and dispersed powder of silicon carbide can be applied for manufacturing critical parts of airplanes, motorcars and high efficiency cutting tools for metalworking. The specific strength of the metal composite materials is 50-100 times as high as steel and special alloys, and they can be used reserving their properties at the temperature up to 1,700 °C. The process has been studied for producing pure silicon from the silicacarbon-based burden materials. The principal impurities content (Fe, Al, K, V, Mn) in the produced silicon (except for Ca) is near to the standard polycrystalline silicon of the KBD-1 Grade while boron and carbon contents are even just below [8].

Further studies [9] have demonstrated the efficiency in application of the silicacarbon as filler in the carbon antifriction products. During the experimentation it was specified that feeding hens with mixed fodder with 3%-content of "Risostim" increased by 8.6% the egg-laying capacity of the laying poultry and decreased the egg-breakage by 31%. The hen's mortality was decreased by 28%. The body weight increased by 25-38%. The fodder consumption per unit of gain decreased by 25-29%. This effect was because of the activity of the sorbing properties of

silicacarbon by removal of toxins from the organism. It was also because of the positive influence of silicon compounds present in this material in easily digestible amorphous form on formation and adequate performance of epithelial- and connective tissues, which are given strength, elasticity, and impermeability. The Organic Product (OP) has proven to be a highly selective collector for lead minerals in benefiting complex lead-zinc ores [10]. The OP is active as a high-efficiency plant growth stimulant [11]. The presowing treatment of the seeds with an 0.5%-solution of OP secures increase in productivity of the Lucerne green material by 22.4% and barley corn by 26.3% when growing them on the meadow brown-, alkali-saline-, strongly saline-, medium loamy soils against the background of the zonal agrotechnology. It is found to increase Indian corn productivity by 28.6% when growing it on the light-chestnut-, irrigated-, weakly eroded-, medium loamy soils against the background of mineral fertilizers (N₉₂P₉₂). The organic product has a wide spectrum of antimicrobial properties, and it can be used as antiseptic [12]. 50%-solution of it exerts the more intense inhibitory action on the tuberculosis cell culture growth *Mycobacterium bovis* St. 8 as compared with the industrial preparation "Glutar", and 0.8%-solution has an active effect on colibacilli, salmonella and fungi. For thorough elaboration of the SC and OP technology, the pilot production facility was built to process the RH with the production capacity 4.5 kg of SC and 4.5kg of OP per hour as seen in Figure 2. One of the major advantages in technology is the comprehensive non-waste recovery of all useful components from the rice hulls without the organic compounds' incineration and emissions into the environment.



Figure 2: The pilot plant for the thermal treatment of RH.

The results of the chemical analysis of silica-carbon obtained on the pilot plant, labeled as SC-1, indicated that its content is different from the silica-carbon content obtained under laboratory conditions. The content of volatile hydrocarbons increased almost twofold from 8-12% to 22-26%. There were some noted changes in the X-ray phase composition of the carbonaceous component of silicacarbon. The silicacarbon diffractogram shown in Figure 3

reflects the $d=4.0\text{\AA}$ and $d=8.6\text{\AA}$, close to the previously identified silica-carbon, which were obtained in the stationary mode and could indicate the presence of such phases therein. The occurrence of three hydrocarbon phases in SC-1 was found: polynaphthenic phase (Np) with $d=4.0\text{\AA}$, stable hydrocarbon phase (Hp) with $d=8.6\text{\AA}$ and hydrocarbon phase in the form of resinous components with $d=8.6\text{\AA}$. It is important to note that a substantial number of resinous substances is unacceptable in silica-carbon which is a polyfunctional product. The presence of resinous substances influences the properties of elastomers and the carbon composite materials produced from silica-carbon. Conversely, with smelting of ferrosilicon and silica, the heating of the granules and briquettes prepared from silica-carbon will result in their destruction. To optimize the composition of silica-carbon SC-1, its reheat treatment was conducted under stationary conditions. The effect of heat treatment temperature on the mass loss and residual of hydrocarbon compounds in the final product was studied.

compounds which appear on the diffractogram shown in Figure 3. It should be noted that in the steady state at $400\text{ }^\circ\text{C}$ the rice hull pyrolysis reaches only the formation of hydrocarbon phases - polynaphthenic (Np) and hydrocarbon (Hp) in the ratio 76:24 percent respectively, with the absence of a graphite-like phase. But the presence of a carbonaceous phase in SC-1 is indicated by chemical analysis as well as on the diffractogram by weak reflex with $d=3.47\text{\AA}$ and angle $27.8^\circ=2\theta$. Taking into consideration the results, the rice hulls heat treatment technology has been subsequently adjusted. The pyrolysis silica-carbon SC-1 produced from the pilot plant was subjected to the secondary heat treatment on the pilot plant at a temperature of $500\text{-}550\text{ }^\circ\text{C}$ for 30 minutes. The silica-carbon labeled SC-2 has the stable chemical composition, mass %: $\text{SiO}_2\sim 39\text{-}41$, $\text{C}\sim 43\text{-}45$, hydrocarbons $\sim 10\text{-}12$, inorganic impurities 2%, and moisture 3-5%. By using electron microscopy of SC-1, which contained about 24% of hydrocarbons, it was demonstrated that it was formed by the packed nanoparticles of amorphous carbon and silicon dioxide with a size 10-100nm equally distributed between each other. Because only the hydrocarbons content in SC-2 was changed (10-12%), the particle sizes will remain the same. Decrease in the volatile hydrocarbons content from 24% to 10-12% in the silica-carbon as the result of the secondary pyrolysis is reflected on the diffractogram of this sample by reducing almost twice the intensity of the reflection with $d=8.6\text{\AA}$ as seen in Figure 4, that confirms the earlier conclusion concerning the presence of three hydrocarbon phases in SC-1 in comparison with SC obtained under the stationary conditions at the temperature of $500, 600\text{ }^\circ\text{C}$.

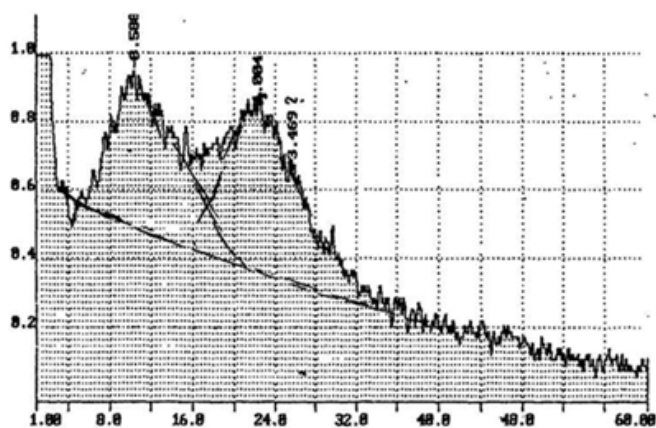


Figure 3: Diffractogram of silica-carbon obtained on the pilot plant.

For this purpose, the laboratory reactor was filled with silica carbon obtained at the pilot plant and was subjected to heat treatment at the three differing temperatures of $400, 500, 600\text{ }^\circ\text{C}$ and cooled to the for 30 and 60 minutes. After this process the residue was weighed, and the mass loss and the residual hydrocarbon content were determined. In Table 5 the summary data are given on the results of the reheat treatment of SC-1 content, mass %: $\text{SiO}_2\sim 29.5$; $\text{C}\sim 40.2$; hydrocarbons ~ 24 , moisture ~ 3 . The data in Table 5 demonstrate that the temperature in the reactor of the pilot plant was known lower than $400\text{ }^\circ\text{C}$, that is why the pyrolysis of the rice hulls reached only the formation of various hydrocarbon

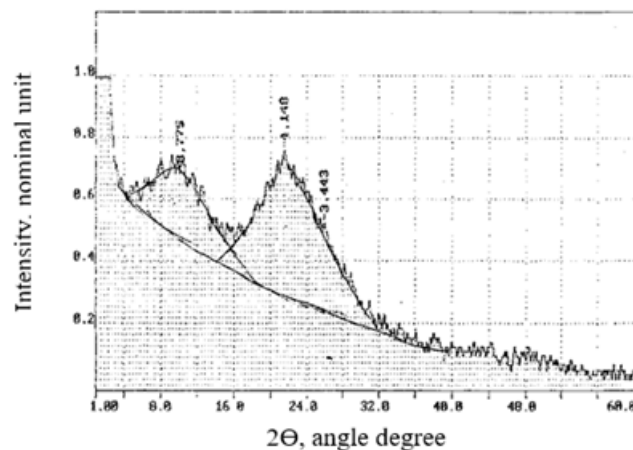


Figure 4: Diffractogram of the silica-carbon (SC-2) obtained on the pilot plant after the secondary treatment of the pyrolysis silica-carbon SC-1.

Table 5: Summary data on reheat treatment of the pyrolysis silica-carbon on the laboratory-scale plant.

Temperature, $^\circ\text{C}$	Duration, min.	Load, g	Mass loss, %	Residual Hydrocarbon Content by Analysis, %	X-Ray Phase Composition of Carbonaceous Components in SC, XRF Analysis, %			
					Np	Hp	Resins	Gp
300	60	250	-	24.0	76	10	14	-
400	30	250	8.0	16.0	78	14	8	-
400	60	250	9.0	16.0	78	14	8	-
500	30	250	12.0	13.5	45	10	2	43

500	60	250	12.0	13.5	45	10	2	43
600	30	250	13.6	12.0	35	10	0	55
600	60	250	13.6	10.0	35	10	0	55

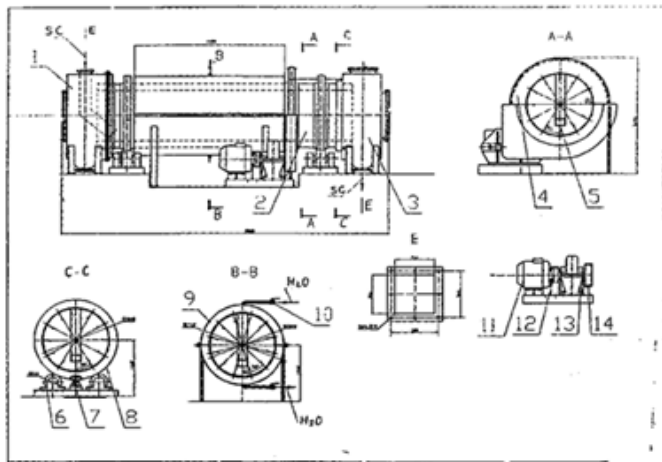


Figure 5: Silica-carbon cooling device. 1. The loading chamber, 2. Drum, 3. Unloading chamber, 4. Crowned crochet, 5. A nozzle sector, 6. Support Roller, 7. Thrust roller, 8. Bandage, 9. Bathtub, 10. Two-way ball valve, 11. Electric motor N=3kW, 12. Elastic sleeve-to-fingercoupling, 13. Reducer, 14. Gear.

Thus, during the research of the rice hull pyrolysis during the pilot plant operating in the continuous mode, the physical and chemical principles of the changes in composition and structure of the obtained silica-carbon products have been defined. And such principles were considered in development of the resultant innovative technology which enables the production of silica-carbon with the required stable composition in a single technological cycle meeting all the requirements specified material for the polyfunctional. Further work has been conducted to justify the technological and instrumental solution for creating a plant for processing of Rice Husk (RH) with the production Silica-Carbon (SC). The main elements of the thermal processing (pyrolysis) unit of the rice husk has been selected. This consists of a rotating pyrolysis rear tor and a silica-carbon cooling apparatus. The required time for the process of thermal destruction of the components of rice husks is 2.8 minutes according to the calculations, the process will be completed by obtaining silica-carbon of the required composition and quality. This is shown in Figure 5. The design of the SC cooling apparatus (drum) at its productivity (P) 250kg/h has been conducted. The designed length (L) is 4m, the inside diameter (d) is 2m, the angle of inclination (α) is 1°, the number of revolutions (n)-1r/m. The designed progressive speed of the SC in the drum (ω):

$$\omega = 5,78 \times d \times \alpha \times n = 5,78 \times 2 \times 1 \times 1 = 11,56 \text{ m/h} \quad (1)$$

The deigned dwelling time of SC in the cooling drum (τ):

$$\tau = \frac{L}{\omega} = \frac{4}{11,56} = 20,76 \text{ min} \quad (2)$$

The designed coefficient of filling the drum with the material (ψ):

$$\psi = \frac{P}{\omega \cdot F \cdot \delta} = \frac{250}{11,56 \cdot 3,14 \cdot 100} = 0,069 \quad (3)$$

where P consumption RH, kg/hour.

F - the area of the internal section of the drum (m²), δ -bulk density RH, kg/m³. From the formula $\psi = (\varphi/360 - (\sin \varphi)/2\pi)$ is found the central angle (φ) of the material lay-er in the drum, $\varphi = 77^\circ$. The height of the SC layer in the drum (hsc) is found from the formula:

$$hsc = d/2 (1 - \cos \varphi/2) = 1 \times (1 - 0,782) = 0,218 \text{ m} \quad (4)$$

At the cooling 250kg/h of SC from 600 °C (t) to 60 °C (t₁), the heat (Q) must be extracted in the quantity:

$$Q = (P \times (t - t_1) \times C_{sc}) / \eta \quad (5)$$

So, $Q = (250 \times (600 - 60) \times 0,816) / 0,6 = 183600 \text{ kJ/hour}$, where $C_{sc} = 0,816$ is the specific heat of SC, kJ/kg · °C, $\eta = 0,6$ - is the cooling efficiency of the drum. For cooling of 250kg/h SC from 600 °C (t) to 60 °C (t₁), the water (P₁kg/h) is required at its initial temperature of 20 °C (t₀) and the final temperature of 60 °C (t₁):

$$P_1 = Q / ((t - t_1) \times C_{water}) = 183600 / ((60 - 20) \times 4,18) = 1098 \text{ kg/h} \quad (6)$$

where $C_{water} = 4,18 \text{ kJ/kg} \cdot ^\circ\text{C}$ - specific heat of water. To cool this volume of water from 60 °C to 20 °C, the cooling tower design should provide a water flow of ~1.17m³/h.

Conclusion

The processing of rice hull pyrolysis was studied in a pilot plant operating in a continuous mode. It was found that the content and structure of the silica-carbon products obtained on the pilot plant were different from the content and structure of the silica-carbon produced in the stationary mode. Stabilization of the composition and the structure of the pyrolysis silica-carbon produced is controlled by its reheat treatment at the temperature of 550-600 °C and holding it for 30 minutes on the pilot plant. The main elements of the thermal processing (pyrolysis) unit of the rice husk were finalized. These were formalized by using a rotating pyrolysis reactor and a silica-carbon cooling apparatus. In summary, silica-carbon is a polyfunctional material and can be used as a filler of elastomers and carbon structural materials, and as an additive in poultry farming.

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