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SILICON-CARBON TREATMENT OUT OF INORGANIC IMPURITIES PRODUCED FROM THE RICE HUSK

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Abstract: The process of silicon-carbon produced out of the rice husk treatment with HCl solution has been investigated and elaborated to purify from aluminum and phosphorous to their contents were less than 0.1 % each. Silicon-carbon with such contents of these impurities meets the requirements to as the multifunctional material for its application in various branches of industry and agriculture. It was demonstrated that the required content of aluminum and phosphorus in silicon-carbon can be achieved by two ways. Namely, by silicon-carbon production out of the pre-treated rice husk or by purification of silica-carbon obtained out of untreated rice husk. Considering the output of silicon-carbon out of rice husk equals to 33 %, it is more reasonable to submit the minor material mass to the acid treatment that is the silicon-carbon. The silicon-carbon treatment produced out of untreated rice husk (52.3 % C, 35.9 % SiO_2, 0.2 % Al and 0.12 % P) was carried out with 0.5 – 3 % hydrochloric acid solutions at the temperature of 70-80 °C. As a result of laboratory studies and pilot testing, the processing method hydrochemical treatment of silicon-carbon was elaborated. The following optimal processing parameters were recommended: hydrochloric acid concentration in aqueous solution – 1 %, 75-80 °C, and treatment time is 60 minutes. The aluminum content was at the level of 0.085 %, and phosphorus was 0.04 % in the processed silicon-carbon. The obtained silicon-carbon with low level of inorganic elements can be applied as the charging material for smelting low-aluminum ferrosilicon and pure silicon, as the filler of carbon construction materials and elastomers.

Key words: rice husk, silicon-carbon, treatment from inorganic impurities.

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ОЧИСТКА КРЕМНЕУГЛЕРОДА, ПОЛУЧЕННОГО ИЗ РИСОВОЙ ШЕЛУХИ, ОТ НЕОРГАНИЧЕСКИХ ПРИМЕСЕЙ

Резюме: С целью очистки кремнеуглерода, полученного из рисовой шелухи, от алюминия и фосфора до их содержания менее 0,1 % исследован и отработан процесс его обработки раствором HCl. Кремнеуглерод с таким содержанием этих примесей отвечает требованиям как к полифункциональному материалу для применения в различных областях техники и сельского хозяйства. Показано, что требуемое содержания алюминия и фосфора в кремнеуглероде может быть достигнуто двумя путями. Путем получения кремнеуглерода из предварительно очищенной рисовой шелухи или путем очистки кремнеуглерода, полученного из неочищенной рисовой шелухи. Учитывая, что выход кремнеуглерода из рисовой шелухи составляет 33 %, более целесообразно подвергать кислотной очистке меньшую массу материала, то есть кремнеуглерод. Очистка кремнеуглерода, полученного из неочищенной рисовой шелухи (52,3 % С, 35,9 % SiO_2, 0,2 % Al, 0,12 % P), проведена 0,5 – 3 %-ными растворами соляной кислоты при температуре 70-80 °С. В результате лабораторных исследований и опытных испытаний отработан технологический режим гидрохимической очистки кремнеуглерода. Рекомендованы оптимальные параметры процесса: концентрация соляной кислоты в водном растворе 1 %, температура 75-80 °С, время обработки 60 мин. Содержание в очищенном кремнеуглероде алюминия было на уровне 0,085 %, а фосфора 0,04 %. Полученный кремнеуглерод с низким уровнем неорганических элементов может быть использован как шихтовый материал для выплавки низкоалюминиевого ферросилиция и чистого кремния, как наполнитель углеродных конструкционных материалов и эластомеров.

Ключевые слова: рисовая шелуха, кремнеуглерод, очистка от неорганических примесей.

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КҮРІШ ҚЫПЫҒЫНАН АЛЫНҒАН КРЕМНИЙКӨМІРТЕГІН БЕЙОРГАНИКАЛЫҚ ҚОСПАЛАРДАН ТАЗАРТУ

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Түйіндеме: Күріш қыпығынан алынған кремнийкөміртегін алюминий мен фосфордан, олардың құрамын 0,1 % - дейін азайта отырып тазарту мақсатында, оны HCl ерітіндісімен өңдеу процесі зерттелді және пысықталды. Осындай қоспалардан тұратын кремнийкөміртегі техника мен ауыл шаруашылығының әртүрлі салаларында қолдануға арналған полифункционалды материал сияқты талаптарға жауап бере алады. Кремнийкөміртегіндегі алюминий мен фосфордың қажетті мөлшеріне жету үшін екі жол бар екені көрсетілген. Алдын ала тазартылған күріш қыпығынан кремнийкөміртегін алу арқылы, және тазартылмаған күріш қыпығынан алынған кремнийкөміртегін тазарту жолымен. Күріш қыпығынан кремнийкөміртегінің шығуы 33 %-ды құрайтынын ескере отырып, материалдың аз массасын, яғни кремнийкөміртегін қышқылмен тазалаған жөн. Тазартылмаған күріш қыпығынан алынған (52,3 % C, 35,9 % SiO_2, 0,2 % Al, 0,12 % P) кремнийкөміртегін тазарту 70-80 °C температурада тұз қышқылының 0,5 - 3 % ерітіндісімен жүргізілді. Зертханалық зерттеулер мен тәжірибелік сынақтар нәтижесінде кремнийкөміртегін гидрохимиялық тазартудың технологиялық режимі пысықталды. Процестің оңтайлы параметрлері ұсынылады: сулы ерітіндідегі тұз қышқылының концентрациясы 1 %, температура 75-80 °C, өңдеу уақыты 60 минут. Тазартылған кремнийкөміртегінде алюминий мөлшері 0,085 %, ал фосфор 0,04 % деңгейінде болды. Бейорганикалық элементтері аз денгейде болатын осындай кремнийкөміртегі - төменалюминилі ферросилицийді және таза кремний балқыту үшін шихта материал ретінде, сонымен қатар конструкциялы материалдар мен эластомерлердің толтырғышы ретінде қолдануы мүмкін.

Түйін сөздер: күріш қыпығы, кремнийкөміртегі, бейорганикалық қоспалардан тазарту.

Introduction. The direct reduction technology of high-quality silica-containing raw materials with a carbonaceous reducing agent in the ore-thermal furnaces to produce ferrosilicon of a certain degree of purity, after refining of the latter is possible to obtain ferrosilicon with an aluminum and phosphorus content less than 0.1% is one of the accepted technologies for producing low-aluminum ferrosilicon.

That is why a great attention should be paid to improve the quality of original raw materials when reviewing the alternative technologies for producing low-aluminum ferrosilicon.

The quartzites deposits are in Kazakhstan of a low content of inorganic elements impurities suitable for the production of silicon alloys of just enough high quality.

However, the lack of low-price reducing agents with low levels of inorganic elements is a difficulty. Currently used charcoal and other carbon-containing materials are quite expensive, so their use leads to a significant increase in the cost of the resulting silicon alloys.

Resuming the above, a need to make researches of unconventional sources of carbon and silicon raw materials to be applied is explained. Rice husk (RH) becomes one of these sources containing up to 15% SiO_2 and 40% of carbon in the polymeric organic compounds [1-3]. Whereby, the content of impurities in it is significantly lower than in natural quartzites and coals [4-7].

A wide range of engineering and agriculture branches make use of the rice husk [8-10] and its thermal processed products [11-14]. However, to smelt pure silicon and low-aluminum ferrosilicon, the charge materials with a lower level of aluminum are required [15, 16].

In this regard, this study **is aimed to** research silica carbon treatment ou of inorganic elements produced from the rice husks [17].

Testing and the results. Treating the rice husk by washing it with the tap water does not provide the required content of impurities in it (Table 1). The required aluminum and phosphorus content in silica carbon (<0.1%) can be achieved in two ways.

Table 1 - Components are in the rice husk

Materials	Chemical compound,% mass *						
	SiO_2	Al_2O_3	CaO	FeO	MgO	Na_2O	K_2O
Original rice husk	15,60	0,18	0,28	0,12	0,08	0,52	0,80
Washed out RH	15,9	0,12	0,22	0,09	0,044	0,15	0,16
Hydrochloric acid solution to purify RH							
0,5 %	22,26	0,08	0,005	0,016	0,010	0,02	0,012
1,0 %	22,25	0,06	0,003	0,013	0,009	0,010	0,010
1,5 %	22,29	0,06	0,003	0,009	0,009	0,010	0,010
2,0 %	22,30	0,06	0,003	0,009	0,009	0,010	0,010
*Cr, Ni – <0,0001; Ti – 0,001; Mn – 0,002-0,003; Cu – 0,0003-0,0004; Zn – 0,002; B – N/A							

Silica carbon produced out of the rice husk that is previously treated by HCl solution, or by an HCl solution of silica carbon produced from untreated rice husk. Each of these methods has its benefits and drawbacks.

The rice husk (RH) is profoundly purified out of impurities by its being treated with HCl solutions. RH treatment with hydrochloric acid solutions with a 0.5% to 2% concentration was carried out for 60 minutes at 70-80 °C.

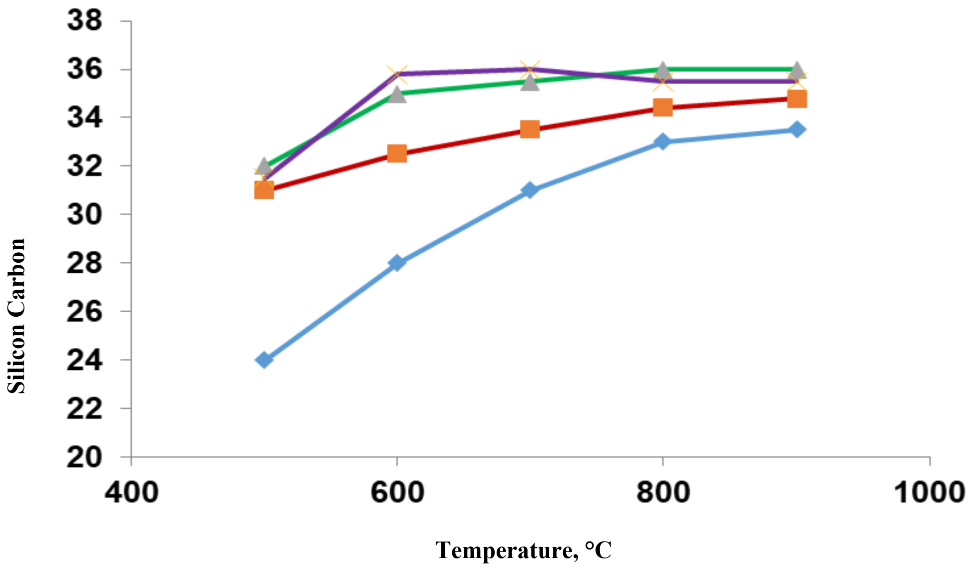
Washed out treated RH until neutral reaction of the rinse water was squeezed, dried and analyzed to have inorganic impurities content. An increase in the acid concentration of more than 1% (Table 1) has been established not actually to improve the treatment rates, therefore, an HCl concentration of 1% is assumed.

When the rice husk acid treated by silica its organic component undergo structural changes. By the infrared spectroscopy method [18], amorphous silica, which is present in the rice husk in an opal modification, was established to transform into a modification of silicic acid.

An experimental batch of purified rice husk has been accumulated and its further thermal treatment with the production of silica carbon is provided.

Figure 1 provides the silica carbon output during the thermal processing of the rice husk at various temperatures.

Based on the results analysis, the maximum silica carbon output from the rice husk can be demonstrated to be provided by its treatment at 600-650 °C for 30 minutes.



1 – 10 min; 2 – 20 min; 3 – 30 min; 4 – 60 min

Figure 1 – Impact of treatment temperature of the rice husk at the silica carbon terminal stage

Table 2 - The silica carbon chemical compound made of refined rice husk

Temperature °C	Content, % mass.					
	C	SiO_2	CaO	Fe_2O_3	P_2O_5	Al_2O_3
30 min						
500	52,0	31,74	0,22	0,39	<0,05	<0,1
600	51,7	32,06	0,14	0,40	<0,05	<0,1
700	51,8	35,02	0,27	0,42	<0,05	<0,1
800	52,0	35,66	0,25	0,43	<0,05	<0,1
60 min						
500	50,9	33,86	0,22	0,42	<0,05	<0,1
600	51,4	34,54	0,16	0,35	<0,05	<0,1
700	52,6	35,96	0,32	0,44	<0,05	<0,1
800	51,9	35,76	0,20	0,40	<0,05	<0,1

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Table 3 - The chemical compound of the rice husk (RH) and silicon-carbon before (original SC) and after (treated SC) acid treatment

Sample	Content, % mass				
	C	SiO_2	CaO	Al_2O_3	FeO
Original RH		15,60	0,28	0,18	0,23
Original SC	52,30	35,90	0,60	0,32	0,70
Treated SC by HCl (0,5%)	51,88	35,40	0,20	0,19	0,26
Treated SC by HCl (1%)	52,10	35,38	0,18	0,15	0,25
Treated SC by HCl (1,5 %)	52,34	35,35	0,16	0,15	0,23
Treated SC by HCl (2%)	52,38	35,35	0,16	0,15	0,22
Treated SC by HCl (3%)	52,42	35,34	0,16	0,15	0,22

The silica carbon produced from non-demineralized (not treated) rice husk contained 52.3% C; 35.9% SiO_2, 0.2% of aluminum and 0.12% of phosphorus. The direct use of such silica carbon for smelting low-aluminum ferrosilicon will not provide the required aluminum content. Therefore, the studies to purify this silica carbon have been conducted by treating it with hydrochloric acid solutions. The results are provided in Table 3.

A sample weight of silicon carbon was treated for 60 min by hydrochloric acid solutions at 80 °C. The acid concentration was varied within 0.5–3%. As a result, an acid treatment of silica carbon reduces the content of calcium and iron by 3-4, and aluminum by 2-2.5 times. An increase in the hydrochloric acid concentration in the studied limits has actually no effect on the residual content of impurities; therefore, the concentration of HCl - 1% is recommended for treating silica carbon.

The benefit of acid treatment of the rice husks is that the rice husks are strong enough and have dimensions (6×3×0,2) mm. This makes it more convenient for hydrotreatment, filtration and drying.

The silicon carbon has a smaller fractional composition (3×1,5×0,15), contains up to 10% of the fraction 0.1 mm less and is weaker. This complicates its filtration and drying. However, the mass of silica carbon is three times less than the rice husk (SC output from RH is 33%). Treatment of the silicon carbon reduce labor costs and consumption of water and hydrochloric acid. Therefore, the silica carbon treatment is recommended rather than the rice husk.

A pilot batch at the pilot plant, with a capacity of 100kg per day, of silicon carbon was produced for its subsequent hydrochemical treatment. The silica carbon obtained in the amount of 220kg had an average composition, wt%: carbon - 51.7; silicon dioxide - 34.9; hydrocarbons - 10.4; aluminum — 0.17; phosphorus - 0.11.

Silica carbon (SC) treatment from impurities was performed with a 1% HCl solution for 60 minutes at 70-80 °C in the hydrometallurgical section of the testing block of RSE "NC CPMC RK" in the following sequence of operations: SC acid treatment → SC squeezing → SC washing → SC squeezing → SC drying.

The silicon carbon in batches of 3.5 kg was loaded into plastic reactors with a volume of 60 liters, and 40 liters of hot water and 1 liter of concentrated hydrochloric acid were added in here. The contents were mixed for 30-40 minutes.

At the end of the leaching, the pulp was settled, and the upper clarified layer was passed through the porcelain nutsche filters. Filtration was carried out through the flax linen. The clean filtered solution was drained.

After the upper clarified acidic solution was separated, a hot water was poured into the reactor to wash the precipitate from the residual acid. Stirring was carried out for 10-15 minutes. After settling, the clarified solution was filtered through the nutsche filters and poured into the sewage system, and the sedimented precipitate was again poured with hot water, and this operation was repeated many times until neutral rinse water (pH = 5-6). The precipitate washed from the acid was squeezed using nutsche filter and discharged into a plastic container.

Drying the precipitate was carried out in a drying chamber at 105-110 °C. After drying, the purified silica carbon was poured into plastic bags. In total, 150kg of silica carbon were subjected to acid treatment.

Table 4 - Treated silica carbon

Test portion No.	Content, %						
	C	SiO_2	Al	P	CaO	FeO	H_2O
1	51,6	34,7	0,083	0,04	0,19	0,26	2,7
2	51,7	34,8	0,087	0,04	0,23	0,27	2.7
3	51,6	35,1	0,085	0,04	0,22	0,28	2,7
4	51,8	34,9	0,090	0,03	0,25	0,26	2.8
5	51,6	35,5	0,086	0,04	0,20	0,26	2,7
6	51,8	35,6	0,084	0,04	0,22	0,28	2,9
7	51,6	34,1	0,087	0,04	0,23	0,29	2,9
8	51,9	34,5	0,086	0,05	0,20	0,29	2,9
9	51,7	34,9	0,085	0,04	0,22	0,28	2,8

Analysis of individual batches of treated silica carbon is provided in Table 4.

Thus, the technological mode of the process of hydrochemical treatment of silica carbon from impurities was developed and a pilot batch of it was prepared for further testing.

The produced silicon carbon is a polyfunctional material that can be used as a charge material for smelting low-aluminum ferrosilicon [19], as well as a filler for elastomers and carbon construction materials [20, 21].

Findings. The silica carbon was set to be treated from aluminum and phosphorus to the required level (less than 0.1%) by HCl solution. The appropriate treatment conditions were determined: 1% of HCl concentration, temperature is 75-80 °C, 60 min time.

At the pilot plant, with a capacity of 100kg of silica carbon per day, a batch of 220kg of silica carbon containing 51,7 % C, 34,9 % SiO_2, 0,17 % Al and 0,11 % P was accumulated.

At the hydrometallurgical site of the testing block of RSE "NC CPMC RK" the acid treatment of silica carbon was carried out in the best performance mode.

The produced silica carbon containing 0.085% of Al and 0.04% of P meets the requirements as a polyfunctional material used in various fields of technology.

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