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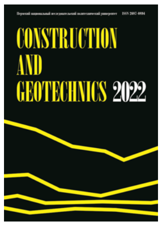


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# PROCESSING OF RICE HUSK ASH WITH PRODUCTION OF POPULAR SILICATE PRODUCTS

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ABSTRACT

Rice husk is a multi-tonnage waste of all countries producing rice, and its disposal is still an unsolved problem. When burning rice husks, energy can be obtained, but a significant amount of ash is formed as a waste, which is an amorphous silicon oxide with various impurities. It is the presence of impurities of residual carbon and calcium and magnesium oxides that do not allow processing rice husk ash as pure silicon oxide to obtain the demanded silicate products. The article proposes and experimentally confirms the possibility of oxidation of residual carbon by nitrates with further dissolution of the resulting product with a sodium alkali at atmospheric pressure. The separate processing of the obtained solution and sediment with the production of marketable products – a solution of liquid glass and granular foam glass is justified. A waste-free complex technology for rice husk ash utilization is proposed in order to obtain valuable market products.

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# УТИЛИЗАЦИЯ ЗОЛЫ РИСОВОЙ ШЕЛУХИ С ПОЛУЧЕНИЕМ ВОСТРЕБОВАННЫХ СИЛИКАТНЫХ МАТЕРИАЛОВ

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*Ключевые слова:*
зола рисовой шелухи, аморфный оксид кремния, жидкое стекло, гранулированное пеностекло.

АННОТАЦИЯ

Рисовая шелуха является многотоннажным отходом во всех странах, производящих рис, и ее утилизация до сих пор остается нерешенной проблемой. При сжигании рисовой шелухи можно получить энергию, но в качестве отхода образуется значительное количество золы, которая представляет собой аморфный оксид кремния с различными примесями. Именно наличие примесей остаточного углерода и оксидов кальция и магния не позволяет перерабатывать золу рисовой шелухи в чистый оксид кремния для получения востребованных силикатных продуктов. В статье предложена и экспериментально подтверждена возможность окисления остаточного углерода нитратами с последующим растворением полученного продукта натриевой щелочью при атмосферном давлении. Обоснована раздельная переработка полученного раствора и осадка с получением товарной продукции – раствора жидкого стекла и гранулированного пеностекла. Предложена безотходная комплексная технология утилизации золы рисовой шелухи с получением ценных товарных продуктов.

## Introduction

Rice husk is a large-tonnage waste left from rice production. It is not subject to humification and therefore becomes a serious environmental pollutant. A distinctive feature of rice husk is its high ash content due to the presence of silicon dioxide [1]. Burning of rice husk is the most perspective method of its utilization because it produces energy and ash which is possible to use for goods manufacture. High quota of silica in ash allows to generate ahigher profit margin, value-added product such as silica gel creating a profitable new industry [2]. The utilization of rice husks is especially relevant for China, where of it are produced up to $4 \cdot 10^7$ tons annually [3]. During the processing of this rice, about 37.8 million tons of rice husks are produced annually in China [4].

Rice husks are characterized by an abnormally high for plant waste ash content. Thus, according to the authors [5], the ash content in various types is varied from 18.3 mass. % up to 28.6 mass. %. The main component of this ash is silica, the content of which reaches 89–96 mass. %. The remaining proportion in the ash is made up of oxides of alkaline, alkaline earth metals, iron and phosphorus. For example, according authors [6] in Nigeria the ash consists for 79–87 % wt. of $SiO_2$ and such admixtures as 0.6–1.8 $Al_2O_3$; 0.6–2.2 $Fe_2O_3$; 0.7–1.6 CaO; 1.5–2.3 MgO; 1.9–2.4 $K_2O$; 0.1–0.2 $Na_2O$ and 4.2–9.9 $P_2O_5$. In the ash, some differences in composition can be found depending on the place where rice grows. Thus, based on the above data, it can be concluded that when burning rice husks in China, 6.9–10.8 million tons of rice husk ash can be obtained annually, which mainly consists of amorphous silicon oxide.

It is very significant to compare the indicator of annual ash production with the extraction of a mineral analogue, also predominantly consisting of amorphous silicon oxide – with diatomite.

Thus, according to the authors, the annual production of diatomite in China is 0.42 million tons [7]. Indeed, the content of amorphous silica in diatomites of various deposits is 63–90 mass. % [8] and the rest part of the mineral consists of oxides such as $Al_2O_3$, $Fe_2O_3$, $TiO_2$, $Na_2O$, $K_2O$, CaO and MgO.

The diagram (Fig. 1) shows the average value content of amorphous silica and admixtures in rice husk ash and diatomite. Obviously that chemical composition is similar for ash and for diatomite and so both materials are valuable as a source of amorphous silica. However, diatomite is considered a valuable fossil raw material and mining companies incur high costs for its extraction, while rice husk ash is considered a useless waste. We propose to consider rice husk ash as a valuable resource of contaminated amorphous silicon oxide, similar to diatomite, but rice husk ash, in contrast with diatomite, is a renewable resource. Therefore, for developing of rice husk ash processing it is necessary to focus not on destroying of the material, but on full using of all its components and obtaining of demanded products.

Based on the chemical structure of both diatomite and rice husk ash, two ways of processing of these feedstock materials can be proposed. The first case is to produce certain products from the entire material without separating silica from impurities. So concrete manufacture is the most typical solution for rice husk ash utilization [9, 10]. Sustainable geopolymer [11] and colored glasses [12] as more expensive materials are also can be manufactured from rice husk ash. The oxide impurities in the feedstock do not significantly affect the quality of the obtained products in these examples.

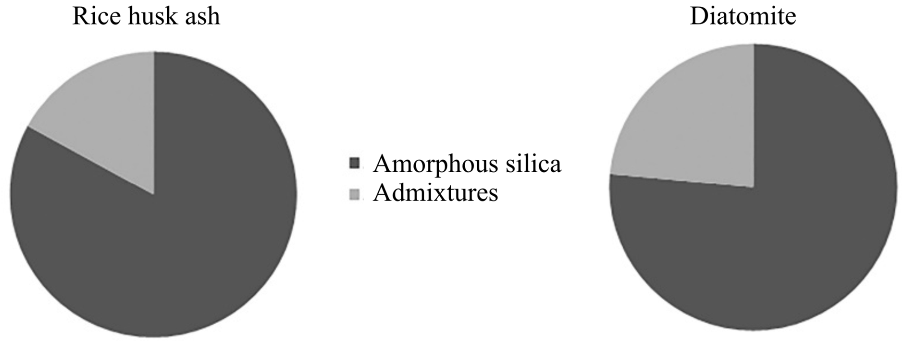



Fig. 1. Average value content of amorphous silica and admixtures in rice husk ash and diatomite
Рис. 1. Среднее значение содержания аморфного кремнезема и примесей в золе рисовой шелухи и диатомите

In the second case, the main component must be cleaned from the impurities for further processing and obtaining products with high added value. The main component of rice husk ash is amorphous silica and therefore the most obvious solution for rice husk ash processing can be offered the production of liquid glass by dissolution in alkali. The dissolution of pure amorphous silica in an alkali solution for obtaining liquid glass at 100 °C and atmospheric pressure is known [13]. However, the presence of a significant amount of impurities in rice husks ash makes impossible the direct synthesis of liquid glass. The presence of such admixtures as carbon and inorganic compounds makes it difficult to produce pure products with high surplus value from rice husk ash.

From various silicate materials, liquid sodium glass is one of the most popular products and it has a high added. Despite the high content of silicon oxide in ash it is rarely used to produce liquid glass. Rice husk ash is possible to use for liquid glass production by interaction with sodium hydroxide solution but such product polluted with carbon and inorganic ions. So, the consumption of such liquid glass is limited with constructions needs namely for geopolymer manufacture [14, 15]. It is possible to remove iron ions impurities by preliminary treating ash with acids [16], but this method is very expensive. Nevertheless, liquid glass is one of the most important products of the silicate industry and is widely used for the manufacture of heat-resistant and acid-resistant materials, paint coatings, as protective and decorative coatings, for corrosion protection and many other fields of industry [17]. Therefore, in order to obtain pure liquid glass, it is necessary to clean the rice husk ash from carbon and oxides, primarily from $Al_2O_3$, $Fe_2O_3$, CaO, MgO and $P_2O_5$. So, the aim of this article was not only to receive pure liquid glass from rice husk ash but also to concentrate these admixtures in commercial product in order to propose the technologically effective solution of wasteless utilization of rice husk ash.

## Materials and Methods

Rice husk produced in the Krasnodar region of Russia were used. Pyrolysis of the rice husk was performedwith a STA 449 F1 device for the synchronous thermal analysis (Netzsch, Selb, Germany), allowing thethermal analysis of a sample to be performed with simultaneous recording of its thermal gravimetricand calorimetric characteristics. The gaseous products were analyzed with a QMS 303 CF Aeolos massspectrometer (Netzsch, Selb, Germany). The results were processed using the appropriate software.

A scanning electron microscope Hitachi S 3400N (Japan) was used.

For the synthesis of liquid glass with silicate module 3.0, rice husk ash was poured with an estimated amount of sodium alkali and placed in a thermostat at 90°C for a day with stirring. The amount of sodium contained in the nitrate was additionally considered in the case of carbon oxidation of ash with sodium nitrate. The product obtained as a result of ash oxidation after dissolution in alkali was defended for a day at 90°C and the precipitate with the lower half of the solution was separated from the upper transparent solution. Both parts of the resulting solution were dried separately at 90°C until a solid vitreous materials were formed for further analysis.

## Results and discussions

### *Initial rice husk ash*

The initial rice husk was used in energy generator and the resulted ash is shown at the Fig. 2, as an optical photo and at the SEM photo at Fig. 3.

Obviously that carbon presents as in particles of ash as in the free state. Thermogravimetric analysis in air of this sample gives the curves of mass and ion current $m/z = 44$ (carbon dioxide) shown at Fig. 4.

Up to a temperature of 360 °C, a loss of 1.42 mass. % occurs, that may be due to the desorption of water and adsorbed volatile organic compounds. The intensive oxidation of carbon starts at 360°C, speed of oxidation is the highest at 533 °C and it finished approximately at 650°C. The sample contains about 4.01 mass. % of carbon. Therefore, the first task on the way to obtaining pure liquid glass is the task of removing carbon. The most obvious way for carbon removing is its oxidation at heat treatment in the presence of oxidants.

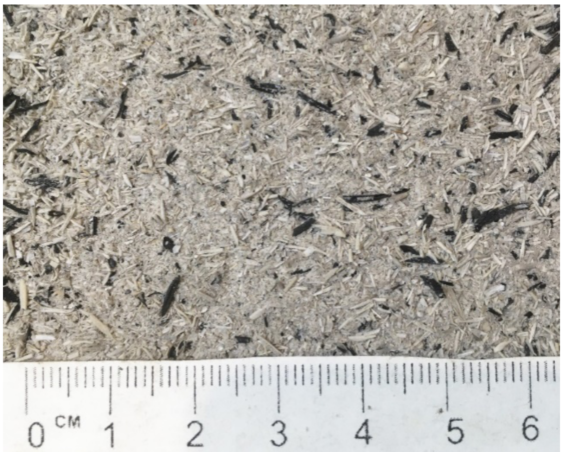



Fig. 2. Optical photo of initial rice husk ash
Рис. 2. Оптическая фотография исходной золы рисовой шелухи

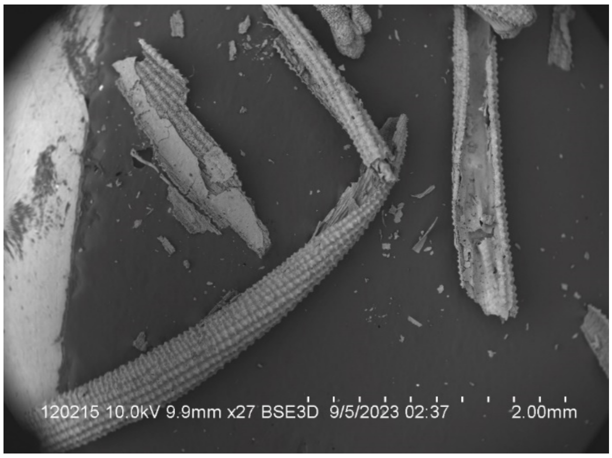



Fig. 3. SEM photo of initial rice husk ash
Рис. 3. СЭМ фотография исходной золы рисовой шелухи

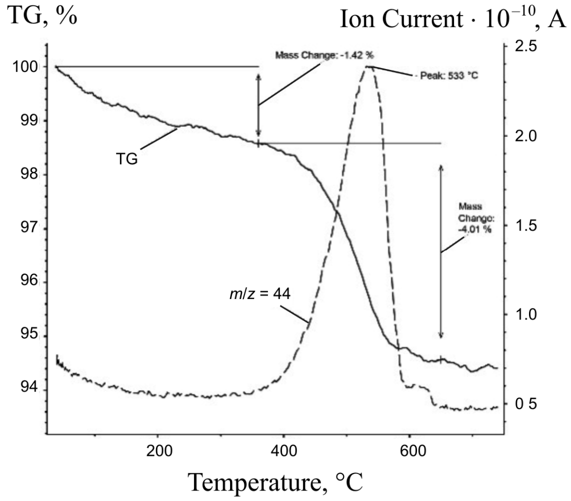



Fig. 4. Thermogravimetric analysis of rice husk ash synchronous with mass spectrum analysis *m*/*z* = 44 (carbon dioxide)

Рис. 4. Термогравиметрический анализ золы рисовой шелухи, синхронизированный с масс-спектральным анализом *m*/*z* = 44 (диоксид углерода)

There are no clear peaks in the diffractogram of the rice husk ash. The perturbation identified in the diffractogram of the sample is characteristic for X-ray amorphous silica.

### *Oxidation of carbon in rice husk ash*

Oxidation of residual carbon in the rice husk ash is difficult due to the need for high temperatures and the difficulties of mass transfer of the air stream inside the ash layer. Therefore, it has been proposed to oxidize carbon of ash with salts what is known for wastes oxidation [18]. Sodium salts with oxidizing potential can additionally form sodium silicate after thermal treatment, which is a valuable product and the basis for the further production of liquid glass. For example, sodium silicate is known to be produced by the interaction of sodium carbonate or sodium hydroxide with natural crystalline sand [19].

The sodium hydroxide, carbonate and nitrate were used for oxidation of carbon in rice husk ash and the corresponding curves of obtained ion currents of carbon dioxide are shown at Fig. 5.

The temperature of the maximum rate of carbon oxidation appeared 458, 453 and 483 °C correspondently for sodium hydroxide, sodium carbonate and sodium nitrate. These temperatures are about

80 degrees lower than the temperatures of carbon oxidation by oxygen in the air. An additional argument in favor of using nitrates to remove carbon from the ash is the absence of the need for external diffusion of oxygen in the air and the associated possibility of entrainment of small ash particles by the exhaust gas. Moreover, these oxidants also form sodium silicates during heat treatment.

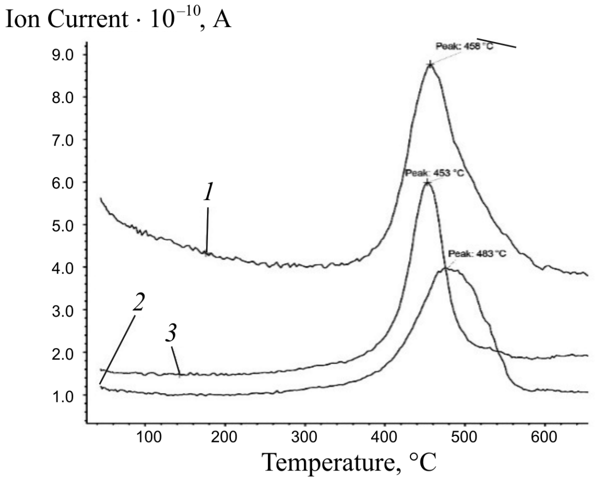



Fig. 5. Mass spectrum curves of carbon dioxide $m/z$ = 44 for 10 mass. % of sodium hydroxide (1), sodium carbonate (2) and sodium nitrate (3) with rice husk ash

Рис. 5. Кривые масс-спектра диоксида углерода $m/z$ = 44 для 10 масс. % гидроксида натрия (1), карбоната натрия (2) и нитрата натрия (3) с золой рисовой шелухи

For example, the diffractogram of the product of the interaction of rice husk ash with sodium nitrate is shown in the Figure 6. The peaks corresponding to cristobalite, tridymite and sodium silicate $Na_2Si_2O_5$ are marked.

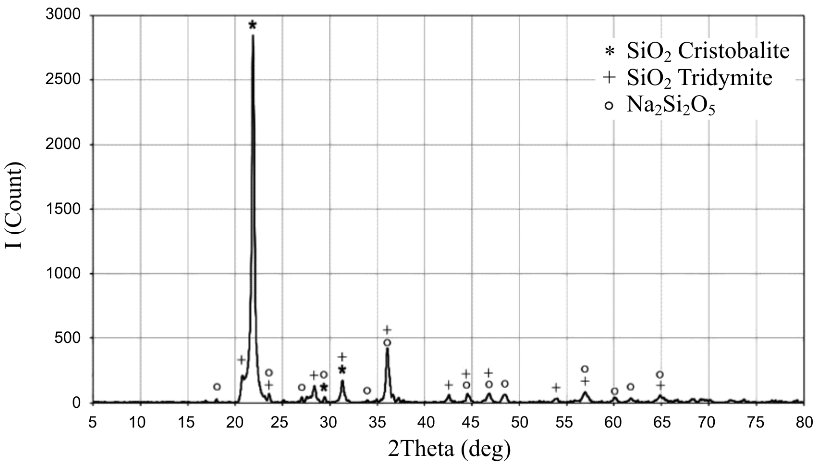



Fig. 6. X-ray diffractogram of the product of the interaction of rice husk ash with sodium nitrate
Рис. 6. Рентгеновская дифрактограмма продукта взаимодействия золы рисовой шелухи с нитратом натрия

The products received from rice husk ash and other two oxidants look similar and have the same crystal phases. The resulted products have the light color and are soluble in hot slight alkali solution at heat treatment at 90 °C.

### *Precipitation of admixtures*

As a result of the dissolution of the product after the oxidation of ash in sodium alkali under mixing at 90 °C during four hours, a solution of liquid glass with a silicate module 3.0 was obtained. A loose sediment falls to the bottom during settling. It can be assumed that the precipitate consists of insoluble magnesium and calcium silicates and partially of crystalline forms of silicon oxide.

To confirm this assumption the solution of liquid glass and sediment formed at the bottom of the liquid glass solution were separated and dried. The results of X-ray fluorescence analysis of the initial ash after oxidation of carbon by ammonia nitrate, as well as the dried solution and sediment are shown in Table.

Atomic concentration of elements in initial ash and in resulted products after interaction with sodium alkali

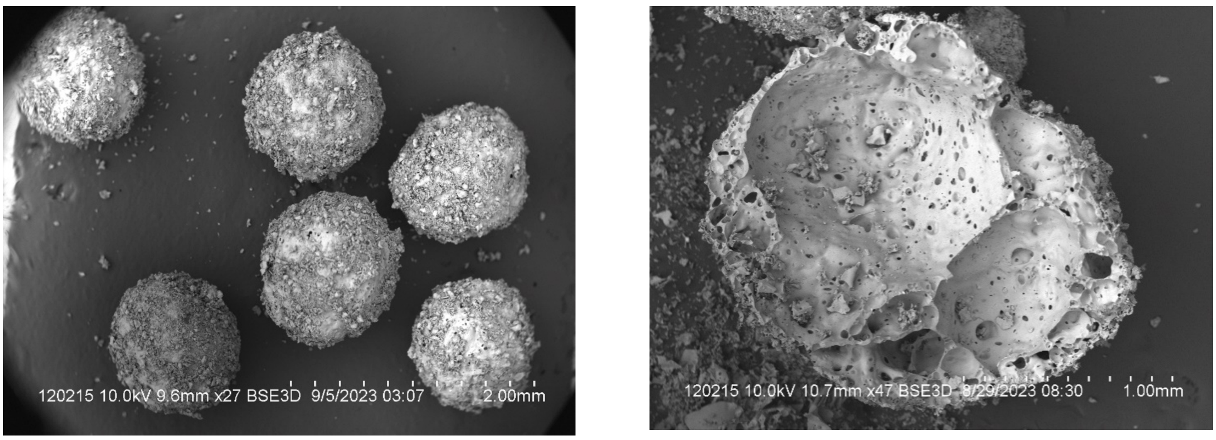
Атомная концентрация элементов в исходной золе и в полученных продуктах после взаимодействия с натриевой щелочью

| Element | Concentration, at. % | | |
|---|---|---|---|
| | initial rice husk ash | solution | sediment |
| Silicon | 28.57 | 22.83 | 18.75 |
| Potassium | 0.57 | 0.82 | 0.36 |
| Magnesium | 0.81 | 0.00 | 1.12 |
| Sodium | 1.00 | 9.43 | 10.08 |
| Calcium | 0.37 | 0.00 | 1.72 |
| Oxygen | 68.68 | 66.92 | 67.97 |
| Total | 100 | 100 | 100 |

It is obvious that the alkali treatment makes it possible to obtain a solution of sodium silicate in which calcium and magnesium silicates are precipitated. Magnesium and calcium silicates have variable compositions and do not crystallize well, but are nevertheless practically insoluble and form precipitates [20]. Cristobalite and tridymite founded in the silicate after removal of carbon by oxidation are also weakly soluble in alkaline solution in contrast to amorphous silica [21]. Therefore, alkaline treatment can separate amorphous silica in the form of soluble glass from insoluble impurities such as magnesium and calcium silicates and crystalline forms of silica.

Sodium silicate solution or liquid glass is a marketable product but the precipitate with a part of solution is also possible to transform in such useful material as granulated foamed glass [22].

Heat treatment of a dried solution with a precipitate at 750°C leads to the formation of a granular cellular material, the photo of which is shown in the Fig. 7.




*a* *b*

Fig. 7. Granules of foamed glass obtained from precipitate and liquid glass (*a*); inner structure of foamed granule (*b*)

Рис. 7. Гранулы вспененного стекла, полученные из осадка и жидкого стекла (*a*); внутренняя структура вспененной гранулы (*b*)

Granulated foamed glass is a well-known and popular product at the market of on the construction materials [23–25]. So, the calcium and magnesium oxide admixtures to the rice husk ash may be not the waste but the base for manufacture of such marketable product as granulated foamed glass.

## Conclusions

Utilization of rice husk ash for manufacturing of products with high surplus value is difficult due to the presence of two main types of impurities in the ash – calcium and magnesium oxides and carbon. The separation of these impurities inevitably needs in high operating costs. But another solution is also possible in which carbon is removed by oxidation, and magnesium and calcium oxides are separated not in the waste format, but in the form of a valuable product as granulated foamed glass. A waste-free complex technology for rice husk ash utilization is proposed in order to obtain valuable market products – liquid glass and granulated foamed glass.

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