

# Assessment of synthesis methods of SiO<sub>2</sub> produced from rice husk and its main physicochemical characteristics

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## ABSTRACT

Extraction of silica from rice husk is an emerging trend in the current research field. Large amount of rice husk (RH) are treated as waste and disposed of at the landfill site. These waste materials can also cause fire, which may lead to severe environmental pollutions. The airborne particles produced from dust may induce respiratory disease to human beings. The burning of rice husk results in the formation of rice husk ash (RHA) with major SiO<sub>2</sub> content with 10 to 20% of carbon and organic components depending on the burning conditions, the furnace type, the rice variety, the climate and the geographical area. Moreover, the commonly used silica precursor like tetraethoxysilane is more expensive, and hence rice husk ash (RHA) and other waste sources having silica are used as an alternative. Acid leaching of the rice husk can be carried out to remove soluble elemental impurities and hence it increases the purity of the silica content. The organic compounds in rice husk and other waste materials can be decomposed under burning conditions. In recent years, environmental demand and sustainable development have become increasingly important. It is important to study and utilize RH biowaste, and convert RHs into valued materials. This is the focus of this research. This paper article will be methods of synthesis SiO<sub>2</sub> from rice husk and its physical-chemical characteristics

*Keywords:* rice husk, silica, SiO<sub>2</sub>, rice husk ash, waste

## 1. Introduction

Rice is the second largest produced cereal in the world. According to information from the Food and Agriculture Organization of the United Nations, world rice production in 2016 was 748.0 million tons in the global rice market (Fig. 1) [1]. While in Kazakhstan, according to the information of Ministry of Agriculture of the Republic of Kazakhstan, the main regions in the territory of the republic involved in rice production are: Kyzylorda Region (78,4 thousand ha), Almaty Region (11,1 thousand ha) and Turkestan Region (3 thousand ha) [2-3]. Rice growing in Kazakhstan is developed mainly in the land areas of the lower reaches of the Syrdarya River, in the territory of the Kyzylorda Region.

In most varieties rice is composed by approximately 20% of rice husk, which contains a fibrous materials and silica (from 1 ton of paddy it is possible to produce 220 kg of rice husk).

Major constituents of RH are cellulose, hemicellulose, lignin, and silica, and a typical composition of these constituents is tabulated in Table

1 [4-6]. The inorganic compounds contained within the rice husk reached about 20% of the dry husk with silica occupying 15-17% of the total while cellulose and lignin which are the main organic components take more than 75% of the total dry weight of the husk while the remaining percentage as oils, proteins, etc. The percentage composition varies with soil type, climatic conditions, crop type, plantation year and fertilizers.

## Recycling of Rice Husk

Depending on the combustion process, the RHA can contain silica in an amorphous form. The amorphous form of silica is obtained from the RHA obtained by burning rice husks at controlled temperatures below 700 °C. The transformation of this amorphous state into a crystalline state occurs if the ash is exposed to high temperatures above 850 °C [7]. While crystalline silica is used in ceramics [8] and in the cement industry [9], amorphous silica has even more applications [10]. Therefore, this residue can be considered as a new economically advantageous raw material for the production of silica [11].

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Rice husk ash, obtained by burning rice husks, can contain more than 90% of silica and an amount of metallic impurities. Metallic impurities such as iron (Fe), manganese (Mn), calcium (Ca), sodium (Na), potassium (K) and magnesium (Mg), which affect the purity and color of silica, can be eliminated by pre-treatment with hydrochloric acid, sulfuric acid or nitric acid before burning [12].

India, Pakistan, Bangladesh, Sri Lanka, Australia, Thailand, Indonesia, and USA were pioneers in the recycling of rice husk during 1970–1985, which were supported by government and other organizations. Unique characteristics of rice husk in comparison

with other agricultural residues, such as high silica contents (87-97 wt% SiO<sub>2</sub>), high porosity, lightweight and very high external surface area make it a valuable material for industrial applications.

Rice husk during combustion gives ash, which contains a very high percentage of crystalline silica. Nevertheless, if it is burned under controlled conditions is obtained amorphous silica, which has a high reactivity.

The heat, generated by burning rice husk, eventually converted into mechanical energy for grinding the ash. RHA during mixing with Portland cement produces high-strength concrete. The conducted

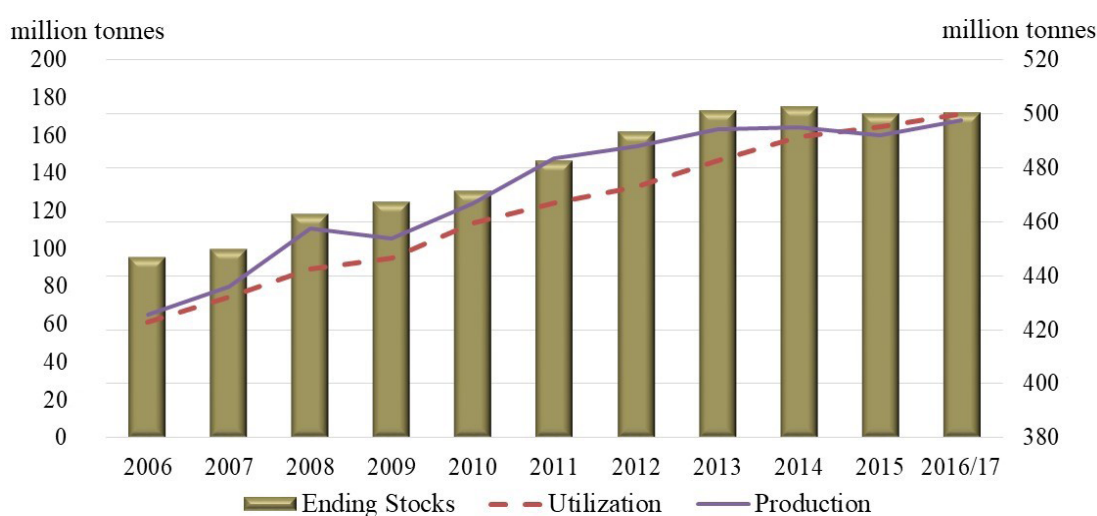


Fig. 1. Rice monitoring of the world market [1].

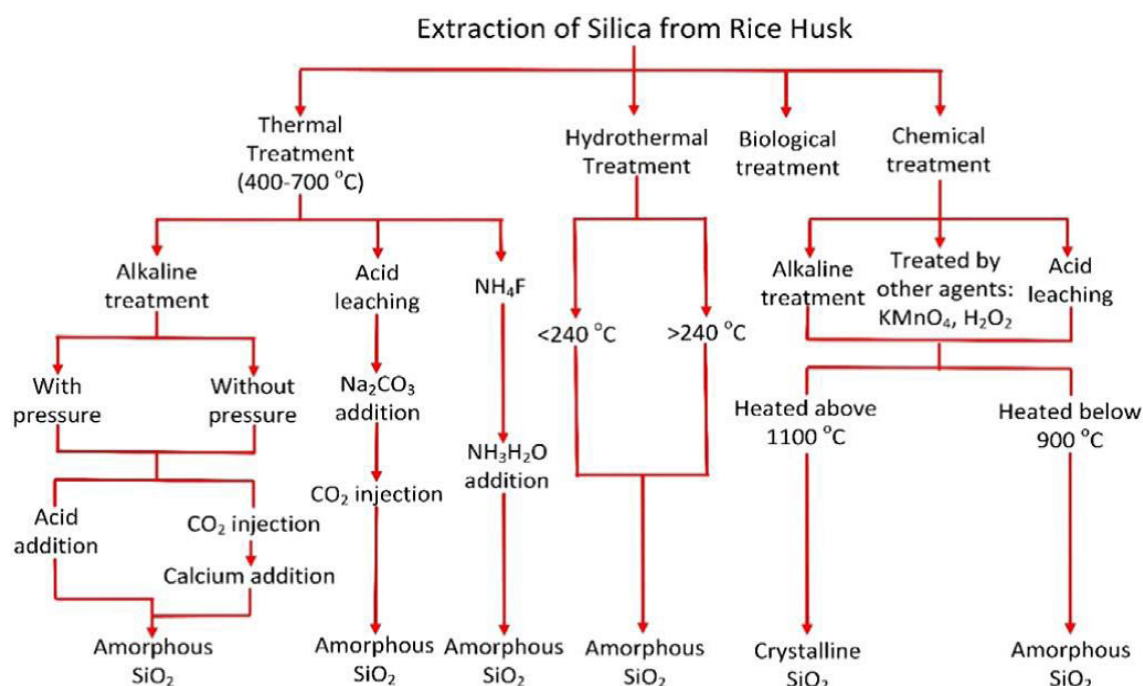


Fig. 2. Different methods for producing different structural silica from RH. Adapted from [13].

**Table 1**

Typical composition of rice husk [4-6]

Composition	Percentage
Cellulose	25-35
Hemicellulose	18-21
Lignin	26-31
Silica	15-17
Soluble	2-5
Moisture content	5-10

studies indicate the possibility of converting RH into amorphous silica with high reactivity, which can be applied in high-strength concrete. Chouhan et al. reported that the temperature of complete combustion of the rice husk affects the compression strength of the lime-RHA solution [14]. In addition, here the main role is played by the reactivity of silica. Thus, the quality of the RHA depends on various factors, such as the temperature of complete combustion, the time, the heating rate, the type of oven/kiln, etc. However, the efficiency of combustion, the optimal use of energy is extremely important for determining the economic benefits of the entire process, which requires careful study.

Chen et al. used fluidized bed combustion (bed material: quartz sand) to produce amorphous silica whose purity level does not exceed 94.8% and, therefore, this method is not suitable for the production of many value-added products [15]. The method of using 1M NaOH was used by Kalapathy et al. to obtain silica from rice husk ash with a purity of more than 97%, in order to create a silicate heat-insulating material [16]. Later, in 2011, Ma et al. was proposed a recyclable technology for the production of silica powder using rice husks and ammonium fluoride. The purity of the final dioxide reached up to 94.6% [17]. Liou et al. obtained amorphous nanostructured silica powders with an average particle size of 60 nm and a high specific surface area by using nonisothermal decomposition of rice husk at temperatures between 27-727 °C by using different burning rates [18]. Yalcin et al. demonstrated that the homogeneous particle size distribution of nano-sized silica can be obtained by burning pre-treated rice husks at 600-800 °C in an atmosphere of pure oxygen [19]. However, by analyzing these methods, it can be concluded that methods that are capable to obtaining high purity nano-silica are chemical based on hazardous and environmentally hazardous products, and can be expensive, requiring additional precautions.

Sodium silicate is used as a source of silicon in the industrial production of silica. However, sodium silicates obtained by melting quartz sand and sodium carbonate at 1300 °C require not only a large amount of energy but also further purification (Affandi et

al., 2009 [20]). In addition, this can be the cause of widespread environmental pollution. Alternative solution of this problem can be the low-temperature extraction of amorphous silica from plant biomass, which makes it possible to produce a high-quality, environmentally friendly and economical product (Liou et al., 2011 [21]).

Ghorbani et al., 2013 showed that 600 °C is the optimum temperature for the production of amorphous silica [22]. Nevertheless, Rozainee et al. found that the RHA remains amorphous at calcination at 700 °C for 6 h [23].

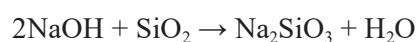
Many researchers used heat treatment of husks and processing with various chemical reagents (HCl, H<sub>2</sub>SO<sub>4</sub>, HNO<sub>3</sub>, NaOH, NH<sub>4</sub>OH, etc.) to produce amorphous high purity silica from rice husks [24-27] before and after burning at temperatures from 773 to 1673 K and at different time intervals. Chemical processing before burning was more beneficial. Whereas the formation of black particles in the composition of silica from the untreated husk was found to be higher than the acid-treated husks. It has been proved that potassium in the husks causes this phenomenon, which is largely removed by acid treatment [25-26].

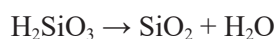
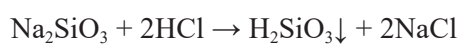
## 2. Experimental Part

### *Extraction of silica nanoparticles*

The versatile material RHA has generated great amount of interest in researchers worldwide. Researchers have worked on various techniques for the synthesis of silica nanoparticles using different templates to control the shape and size of the particles. However in this present study, a simple, template free methods such as pre-acid wash treatment (hydrochloric acid, citric acid) and thermal treatment was used to prepare silica nanoparticles. The aim of acid pre-treatment is to improve the purity of silica product. It proves to be an effective way in substantially removing most of the metallic impurities and producing ash silica completely white in color. The acid treatment also gives a high surface area for the silica when it is precipitated. In addition, the quality of producing nanosilica powder depends on the calcinations or combustions temperature and duration of calcinations or combustions.

WRHA that predominantly contains silica dissolves in sodium hydroxide solution after pre-treatment. Sodium silicate formed is used as precursor for silica synthesis. The principal reaction are described as follows:





Reaction of hydrochloric acid with sodium silicate promotes a silanol ( $\text{R}_3\text{Si-OH}$ ) groups formation and condensation of which leads to the formation of extended three-dimensional Si-O-Si network. The appearance of raw rice husk, white rice husk ash and extracted silica is shown in Fig. 3.

### Methodology of obtaining $\text{SiO}_2$ by processing of rice husks

As research objects served the shells of rice (Rice Husks), selected from different regions of the Republic of Kazakhstan (Fig. 4). Samples of rice husk with a length of about 5-10 mm were pre-washed with water and dried in air.

The samples (Almaty, Kyzylorda and Turkestan

region) of rice husks were previously washed with water for the purification of the composition from foreign substances. Then the initial raw materials were dried in the drying oven at the temperature of 90 °C for 2 h (for complete evaporation of the water in the composition). All prepared samples (50 g) were calcinated at 600 °C for 4 h in a muffle furnace (AAF series, Carbolite (UK)) to produce white rice husk ash (WRHA). Before to produce white rice husk ash we had done pre-treatment process. WRHA was mixed with 100 mL of 2M NaOH at 90 °C under continuous vigorous stirring for 2 h in order to convert the solid silica into water-soluble sodium silicate. The sodium silicate solution was filtered via cellulose membrane filter (Millipore, 0.45  $\mu\text{m}$ ) in order to remove insoluble residues and carbonizates (black ashes). Then sodium silicate was converted into insoluble silicic acid via titration with 2M HCl under continuous stirring for 30 min.



Fig. 3. Appearance of raw rice husk, white rice husk ash and extracted silica from treated rice husk in 2M HCl acid followed by combustion at 600 °C.

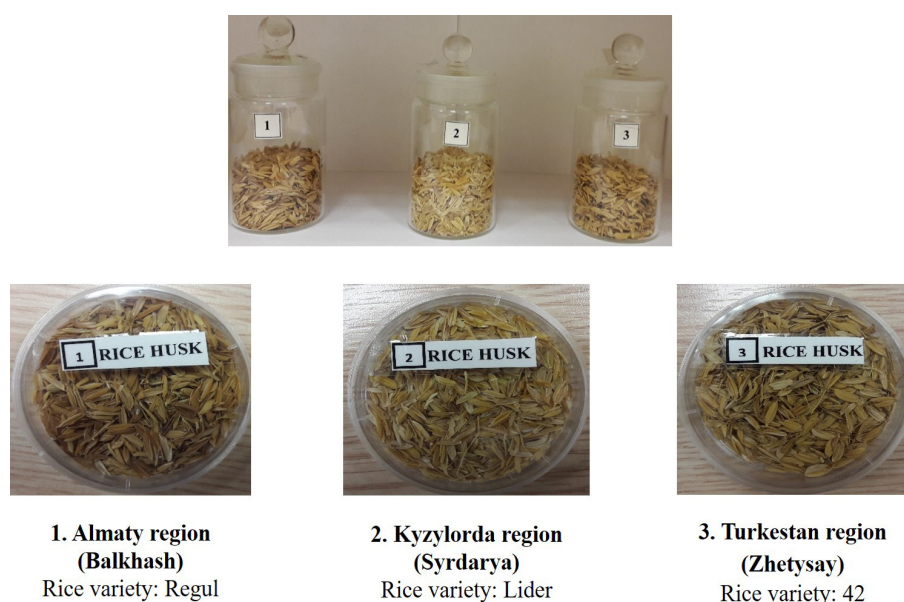


Fig. 4. Rice husks from different regions of Kazakhstan (objects of experiments).

The ash product from combustion water-washed rice husk was completely white. This enabled the distinct determination of the rice husk combustion times, whereby the first formation of completely white ash signified the complete combustion of rice husk.

Scheme of rice husk conversion into the silica by thermal treatment is presented in Fig. 5.

### 3. Results and discussion

It was used two different reagents (hydrochloric acid and citric acid) in the pre-treatment process for washing RH. Silica from pre-treated RH is extracted using 100 mL of 2M NaOH. It was found that the yield of silica is strongly dependent on the type of acid used for washing and concentration of NaOH. The results are shown in the Table 2 and Figs. 6 and 7.

The yield of silica from the rice husks and its purity also depend on the production method. The highest yield of final pure product (17.4%) is formed by preliminary acid treatment of RH from Almaty region. The silica content varies in the range of ~90.1-99.5% depending on the purity of used reagent for washing (acid and its composition).

The dependence of the volume of hydrochloric acid on the pH of solution used for the preliminary washing of RH demonstrates that in the neutral medium (pH = 7) yield of the final product is significantly larger (Fig. 8, Table 3).

As shown by the experiment, from 50 g of rice husks it is possible to obtain 8.71 g of white rice ash, 6 g of pure silicon oxide (purity ~90.1-99.5%), which means that from 1 t of the rice husks it is possible to obtain 120 kg of silicon oxide and from the remains of rice husks in Kazakhstan, approximately in a year 8000 tons of silicon dioxide.

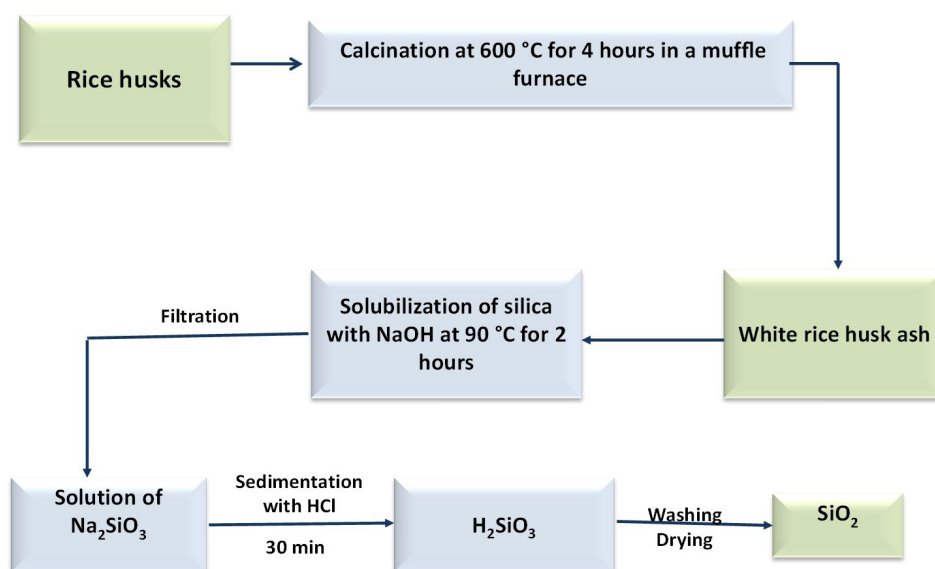


Fig. 5. Scheme of the silicon oxide production from the raw rice husk by thermal treatment.

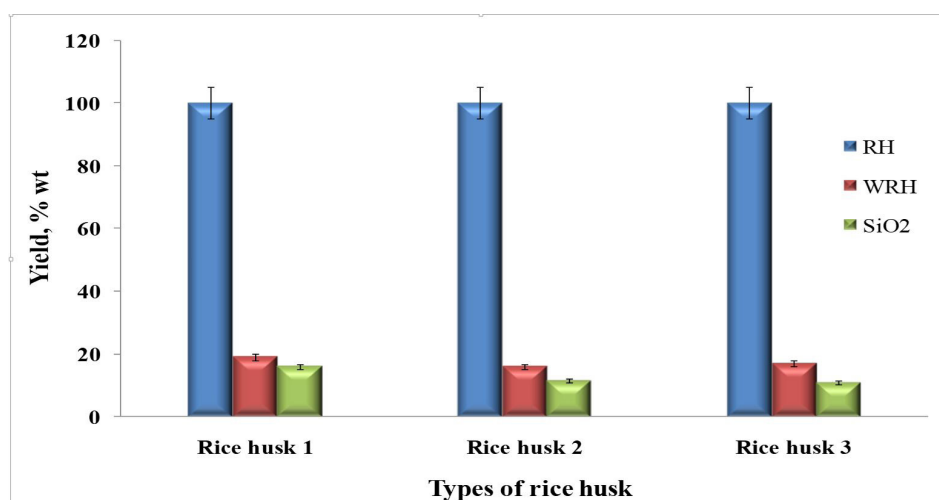


Fig. 6. Percent yield of the white rice husk (WRH) and rice husk silica (RHS) extracted from the rice husk (RH) of different region of Kazakhstan by hydrochloric acid treatment.

**Table 2**

Yield of WRH, Silica from RHs (Almaty region, Kyzylorda region and Turkestan region) during pre-treatment with hydrochloric acid

Sample	Yield, wt%		
	RH 1	RH 2	RH 3
WRH	19	16	17
SiO <sub>2</sub>	17.4	11.5	11
Raw RH	100	100	100

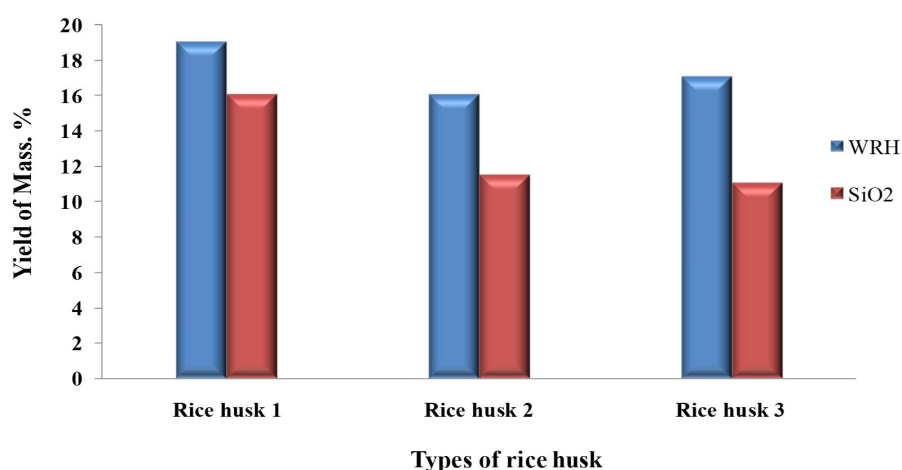


Fig. 7. Percent yield of the white rice husk (WRH) and rice husk silica (RHS) extracted from the rice husk (RH) of different region of Kazakhstan by hydrochloric acid treatment.

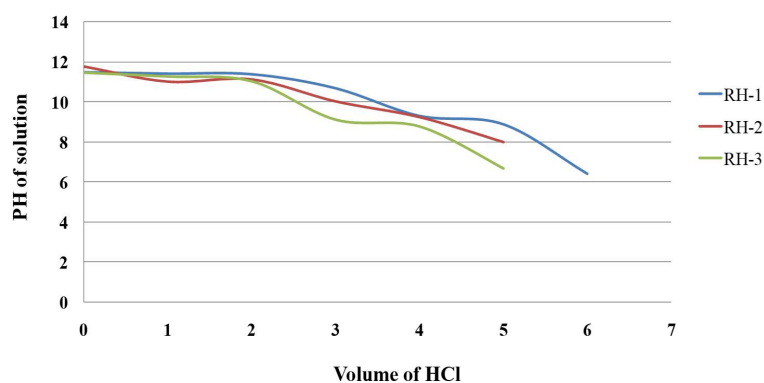


Fig. 8. Influence of volume of HCl on the pH of RH washing.

**Table 3**

Influence of PH & volume of HCl on the yield of obtained final product

PH of solution	RH 1	RH 2	RH 3
1	11.4	11	11.28
2	11.37	11.11	11.04
3	10.67	10.02	9.12
4	9.29	9.24	8.78
5	8.87	8	6.68
6	6.4	—	—
7	11.47	11.75	11.47

## Conclusion

In this paper considers the production of SiO<sub>2</sub> from rice husks, which selected from different regions of the Republic of Kazakhstan.

The experimental data indicate the dependence of the characteristics of the silicon-containing samples extracted from the rice husks on the processing conditions of the raw materials: the size of the formed particles, their shape, percentage of silica in rice husk, influence of volume of HCl on the pH of RH washing. Proposed approach not only assess the feasibility of turning agricultural byproduct in to a valuable resource but also will solve the problem of its disposal in the environment, which otherwise results in water, air and soil pollution.

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### Күріш қауызынан өндірілген SiO<sub>2</sub> синтездеу әдістерін бағалау және оның негізгі физико-химиялық сипаттамалары

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### Андатпа

Кремнеземнің күріш қауызынан өндіру зерттеулердің заманауи саласының жаңа үрдісі болып табылады. Күріш қауызының (КҚ) көп мөлшері қалдық ретінде қарастырылады және полигонда жойылады. Сонымен қатар, бұл қалдықтар қоршаған ортаның ластануына алып келетін өрттің

көзі болуы да мүмкін. Шаңнан пайда болатын ауаның бөлшектері адамның ағзасында тыныс алу органдарының асқынған ауруларын қоздыруы мүмкін. Күріш қауызын өртеу географиялық аймағы, климаты, күріш сұрыпы, пеш түрі мен өртеу шарттарына байланысты 10-нан 20% дейін көміртек пен органикалық компоненттермен SiO<sub>2</sub> көп мөлшерінен тұратын күріш қауызының күлінің пайда болуына әкеледі. Бұдан бөлек, кремний диоксидінің әдетте қолданылатын тетраэтоксисилан секілді негізін құраушы қымбатырақ болып келеді, сондықтан кремний диоксидінен тұратын күріш қауызының күлі (КҚК) және басқа қалдық көздері баламалы таңдау ретінде қолданылады. Күріш қауызын қышқылдық шаймалау еритін элементті қоспалардың жойылуына алып келеді, және сәйкесінше бұл кремнеземнің тазалығын жоғарылатады. Күріш қауызы мен басқа қалдықтардағы органикалық қосылыстар жану жағдайында ыдырауы мүмкін. Соңғы жылдары экологиялық сұраныс пен тұрақты дамудың маңыздылығы артып келеді. КҚ-на қатысты биоқалдықтарды зерттеу мен пайдалану және оларды құнды материалдарға айналдыру маңызды. Бұл зерттеудің негізгі бағыты болып табылады. Келтірілген мақалада КҚ-нан SiO<sub>2</sub> синтездеу әдістері және оның физико-химиялық сипаттамалары қарастырылады.

*Түйінді сөздер:* күріш қауызы, кремнезем, SiO<sub>2</sub>, күріш қауызының күлі, қалдықтар

### Оценка методов синтеза SiO<sub>2</sub>, полученного из рисовой шелухи, и его основные физико-химические характеристики

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### Аннотация

Извлечение кремнезема из рисовой шелухи является новой тенденцией в современной области исследований. Большое количество рисовой шелухи (РШ) рассматривается как отходы и утилизируется на полигоне. Эти отходы также могут стать причиной пожара, который может привести к серьезному загрязнению окружающей среды. Частицы воздуха, образующиеся из пыли, могут



вызывать респираторные заболевания у людей. Сжигание рисовой шелухи приводит к образованию золы рисовой шелухи (ЗРШ) с большим содержанием  $\text{SiO}_2$  с углеродом и органическими компонентами от 10 до 20% в зависимости от условий сжигания, типа печи, сорта риса, климата и географического района. Кроме того, обычно используемый предшественник диоксида кремния, такой как тетраэтоксисилан, является более дорогим, и, следовательно, зола рисовой шелухи (ЗРШ) и другие источники отходов, содержащие диоксид кремния, используются в качестве альтернативы. Кислотное выщелачивание рисовой шелухи может быть проведено для удаления растворимых

элементных примесей, и, следовательно, это повышает чистоту содержания кремнезема. Органические соединения в рисовой шелухе и других отходах могут разлагаться в условиях горения. В последние годы экологический спрос и устойчивое развитие становятся все более важным. Важно изучать и использовать биоотходы в отношении РШ и преобразовывать их в ценные материалы. Это основное направление этого исследования. В данной статье будут рассмотрены методы синтеза  $\text{SiO}_2$  из рисовой шелухи и ее физико-химические характеристики.

*Ключевые слова:* рисовая шелуха, кремнезем,  $\text{SiO}_2$ , зола рисовой шелухи, отходы