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Synthesis of Amorphous and Crystalline Silicon Dioxide Powder Composites from Rice Husk Ash for Thermal Spray Deposit

This paper presents the synthesis of silicon dioxide (SiO₂) from rice husk ash through chemical treatment followed by heat treatment at 800 °C for 3 h. The structural and physicochemical properties of the synthesized silica were investigated using X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), and laser diffraction particle size analysis. The synthesized silica exhibited an amorphous structure, with a median particle size (D50) of approximately 88.9 μm. Subsequent annealing at 1200 °C led to crystallization of the silica, resulting in the formation of quartz and cristobalite phases. FTIR analysis revealed characteristic absorption bands associated with Si–O–Si stretching vibrations in the 1000–1100 cm^{−1} region, as well as bands attributed to hydroxyl groups. The obtained crystalline silica materials demonstrate potential for use as powder materials in thermal spraying technologies.

Keywords: Silicon dioxide (SiO₂), rice husk, chemical treatment, annealing, particle size, FTIR analysis

Introduction

In recent years, particular attention has been paid to the development of new materials based on recycled raw materials, driven by environmental and economic factors. One promising area is the use of agricultural waste as a source of functional materials. Rice husk, in particular, is of considerable interest because its ash content contains up to 90–96 % silicon dioxide (SiO₂), making it an accessible and affordable raw material [1, 2].

Traditional methods for producing silicon dioxide, such as sol-gel synthesis and the use of organosilicate precursors (tetraethoxysilane, tetramethoxysilane), are characterized by high cost, process complexity, and environmental limitations [3, 4]. Therefore, the development of alternative methods for obtaining SiO₂ from natural and man-made sources is a pressing issue.

One effective approach is the acid-thermal treatment of rice husks, which allows for the production of high-purity silicon dioxide. Acid treatment removes metallic impurities (Fe, Ca, K, etc.), while subsequent heat treatment removes the organic component and forms an amorphous or crystalline SiO₂ structure, depending on the temperature [5–7].

Despite the significant amount of research devoted to obtaining SiO₂ from rice husks, comprehensive analysis of its structure, morphology, phase composition, and dispersion remains relevant [8, 9]. Furthermore, the use of such SiO₂ as a powder material for the formation of functional and protective coatings has received limited research.

Of particular interest is the use of the resulting silicon dioxide in thermal spraying technologies, where powder materials are used to create wear-resistant and corrosion-resistant coatings [10–12]. The use of agricultural waste can significantly reduce the cost of coatings while simultaneously addressing environmental waste disposal issues. It should also be noted that research into the use of rice husks to produce SiO₂ is actively developing, but its integration with bionanocomposite and thermal spray coating technologies remains understudied [13–15].

Therefore, the aim of this study is to obtain silicon dioxide from rice husks, study its structure and properties, and evaluate its potential for use in protective coating technologies.

Experimental

Rice husks were used as the starting material. Before the experiment, the raw material was pre-cleaned: washed with distilled water three times to remove mechanical and water-soluble impurities, and then dried at room temperature until completely dry.

For chemical treatment, a hydrochloric acid solution with an initial concentration of 33 % (reagent grade) was used, from which a 5 M solution was prepared by diluting with distilled water. The volume of the starting solution was calculated using the formula:

$$C_1V_1 = C_2V_2, \quad (1)$$

where, C_1 — concentration of the initial solution; V_1 — volume; C_2 — required concentration; V_2 — final volume of the solution. The process flow for obtaining silicon dioxide (SiO_2) from rice husk is shown in Figure 1.

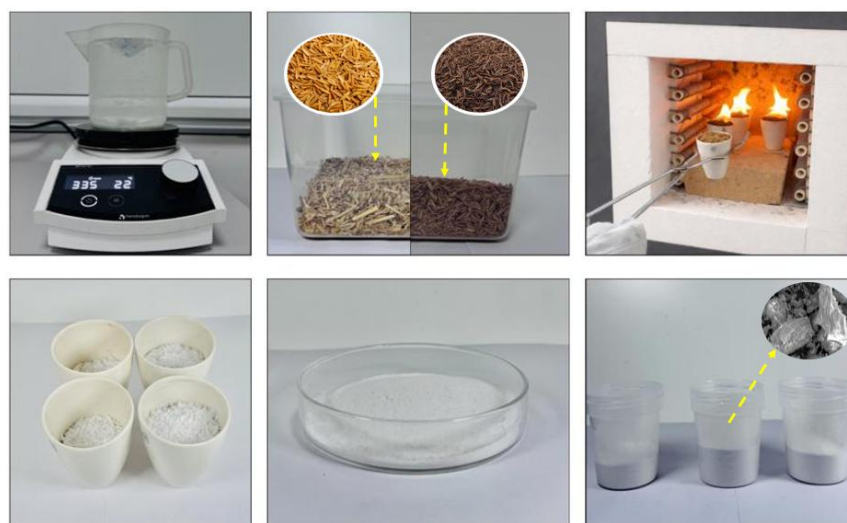


Figure 1 — Process flow for producing silicon dioxide (SiO_2) from rice husks: raw material preparation, thermal annealing, formation of silica-containing ash, and production of dispersed SiO_2 powder.

The rice husks were chemically treated in a 5 M HCl solution to remove mineral impurities. The samples were kept at room temperature for 72 hours with periodic stirring. After treatment, the material was washed with distilled water to a neutral pH and dried at room temperature. Thermal treatment was carried out in a muffle furnace at 800 °C for 3 hours using porcelain crucibles, which ensured the removal of the organic component and the formation of silicon dioxide (SiO_2).

The resulting silicon dioxide (SiO_2) had an amorphous structure. To crystallize the sample, it was annealed at 1200 °C in a muffle furnace (SNOL), which resulted in the formation of a crystalline phase.

X-ray diffraction (XRD) analysis of the samples was performed using a diffractometer with $\text{CuK}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$). Diffraction patterns were recorded in the 2θ range of 20–90° with a scan step of 0.02° and a recording speed of ~0.5 s/step [16]. The phase composition of the samples was determined using the PDF-4+ database, and the diffraction patterns were processed using HighScore Plus software. Infrared spectra of the samples were recorded using a Fourier transform infrared (FTIR) spectrometer in the wavenumber range of 500–4000 cm^{-1} . Measurements were performed at room temperature using the standard technique (in transmission mode). The obtained spectra were processed using the spectrometer's built-in software. Particle size distribution analysis was performed using a Winner 2005 NanoSizer particle size analyzer [17]. Measurements were performed at room temperature under standard conditions. The microstructure and surface morphology of the resulting silicon dioxide (SiO_2) were examined using scanning electron microscopy (SEM-3200).

Results and Discussion

X-ray diffraction analysis was performed to study the phase composition and structural state of the obtained silicon dioxide (SiO_2) before and after heat treatment. The diffraction pattern of the sample annealed at a temperature of 800 °C (Fig. 2) is characterized by the presence of a wide diffuse maximum (halo) in the

region of $\sim 22^\circ$ (2θ), indicating an amorphous structure of the material. The absence of clearly defined diffraction peaks indicates the absence of long-range crystalline order in the SiO_2 structure. After additional annealing at a temperature of 1200°C (Fig. 2), significant changes in the diffraction pattern are observed: narrow and intense diffraction peaks appear, indicating the formation of a crystalline structure. The recorded reflections correspond to the characteristic peaks of crystalline modifications of SiO_2 (quartz, cristobalite), confirming the transition of the material from an amorphous to a crystalline state. The observed transformation is due to a thermally induced rearrangement of the amorphous silica matrix, accompanied by ordering of the atomic structure and the formation of crystalline domains. Thus, the XRD analysis results demonstrate that increasing the processing temperature to 1200°C leads to an amorphous $\rightarrow$ crystalline SiO_2 phase transition. This transition is important for controlling the material's properties, particularly by enhancing its thermal stability and mechanical properties, which is of interest when using SiO_2 in thermal spraying technologies.

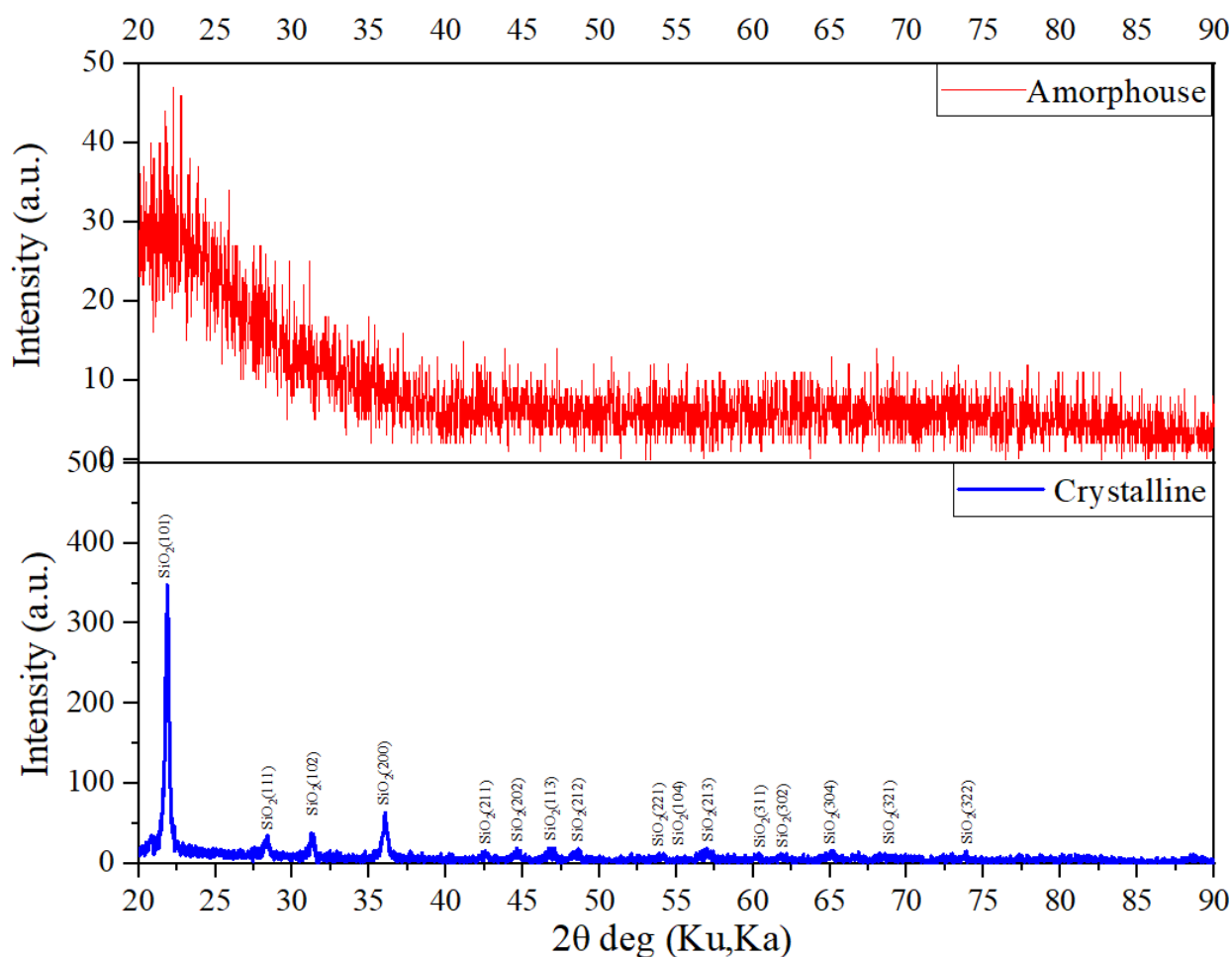


Figure 2 — X-ray diffraction patterns of silica (SiO_2) obtained from rice husk: amorphous silica; crystalline silica after thermal annealing.

FTIR spectra of silica obtained from rice husk ash before and after high-temperature annealing are shown in Figure 3. The spectra of the amorphous sample (Fig. 3) and the crystallized sample (Fig. 3) exhibit characteristic bands consistent with the silica structure. The spectrum of amorphous SiO_2 (Fig. 3) exhibits a broad band in the region of $\sim 3200\text{--}3600\text{ cm}^{-1}$, corresponding to the stretching vibrations of O–H groups, which is associated with the presence of adsorbed moisture and hydroxyl groups on the particle surface. The band near $\sim 1600\text{ cm}^{-1}$ is due to the deformation vibrations of water molecules (H–O–H). The most intense and broad band in the range of $\sim 1000\text{--}1100\text{ cm}^{-1}$ corresponds to the stretching vibrations of Si–O–Si bonds, a characteristic feature of an amorphous silica structure. The width of this band indicates the absence of long-range order in the material's structure.

After annealing at 1200 °C (Fig. 3), changes in the spectral characteristics are observed. The Si–O–Si band becomes more pronounced and partially narrows, indicating an increase in the degree of structural order and the formation of a crystalline phase. Furthermore, the intensity of the bands associated with hydroxyl groups (O–H) and adsorbed water (H–O–H) decreases, which is due to their removal during high-temperature treatment. Thus, the FTIR analysis results confirm that heat treatment at 1200 °C leads to a structural transformation of silicon dioxide from an amorphous to a crystalline state, which is consistent with the X-ray diffraction data.

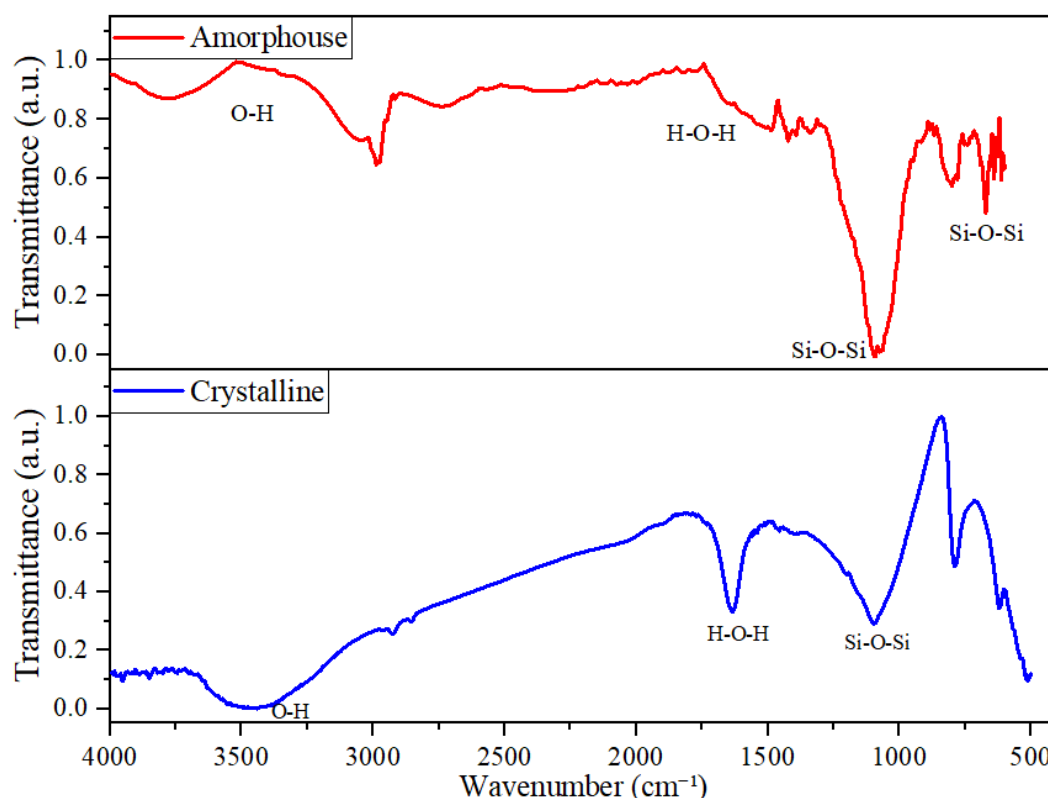


Figure 3 — FTIR spectra of silica (SiO_2) obtained from rice husks: amorphous silica; crystalline silica after thermal annealing

Figure 4 shows differential histograms and cumulative particle distribution curves for silica (SiO_2) obtained from rice husks. The amorphous sample (Fig. 4) exhibits a relatively narrow particle distribution with a pronounced maximum in the range of $\sim 90\text{--}110\ \mu\text{m}$. The majority of particles are concentrated in the range of $50\text{--}150\ \mu\text{m}$. The cumulative curve is characterized by a sharp rise, reaching almost 100 % in the range up to $180\text{--}200\ \mu\text{m}$. This indicates low polydispersity and high powder homogeneity. This distribution pattern is typical of amorphous materials, the structure of which facilitates more uniform destruction during milling and the formation of particles of similar sizes. In contrast, crystalline silicon dioxide (SiO_2) (Fig. 4b) exhibits a significantly broader and more asymmetrical distribution with a distinct “tail” in the region of large particles. Although the main fraction is also in the $\sim 150\text{--}200\ \mu\text{m}$ size range, the system also contains a significant number of particles up to $800\ \mu\text{m}$ and larger. The cumulative curve increases more smoothly and reaches saturation only at significantly larger particle sizes. This indicates high polydispersity of the sample.

Thus, amorphous silicon dioxide (SiO_2) has a more uniform particle size distribution, which can positively impact its processing properties, such as flowability, solubility, and reactivity. Crystalline silicon dioxide (SiO_2), on the other hand, is characterized by high polydispersity, which can lead to inhomogeneous properties and require additional processing (e.g., classification or regrinding).

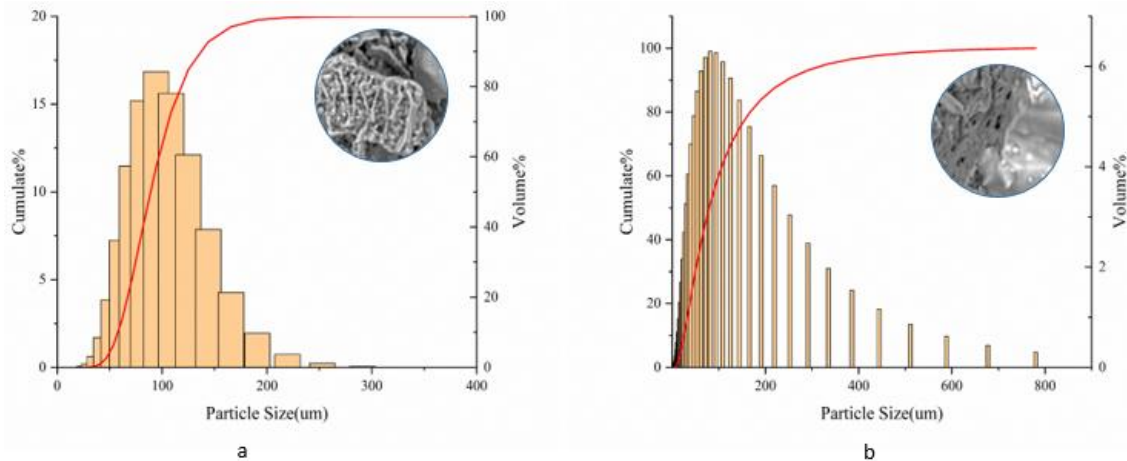


Figure 4 — Differential histograms and cumulative distribution curves of silica (SiO_2) particles obtained from rice husk: a — amorphous silica; b — crystalline silica after thermal annealing

Figure 5 shows SEM images and elemental analysis of amorphous (Fig. 5a) and crystalline (Fig. 5b) silica (SiO_2) particles obtained from rice husk. Amorphous silica (SiO_2) is characterized by a dispersed, disordered morphology consisting of small agglomerated particles with a developed surface area. EDS analysis reveals the presence of only the essential elements Si and O, indicating the high purity of the obtained material and the effective removal of impurities during acid treatment. Crystalline silicon dioxide (SiO_2) exhibits a denser and partially ordered structure with larger particles and pronounced porous fragments inherited from the original raw material. In addition to Si and O, the EDS spectrum also displays a carbon (C) peak, which may be due to residual organic components or surface contamination. Thus, crystallization is accompanied by particle coarsening, increased structural density, and the possible retention of residual impurities, whereas amorphous SiO_2 is characterized by higher purity and homogeneity.

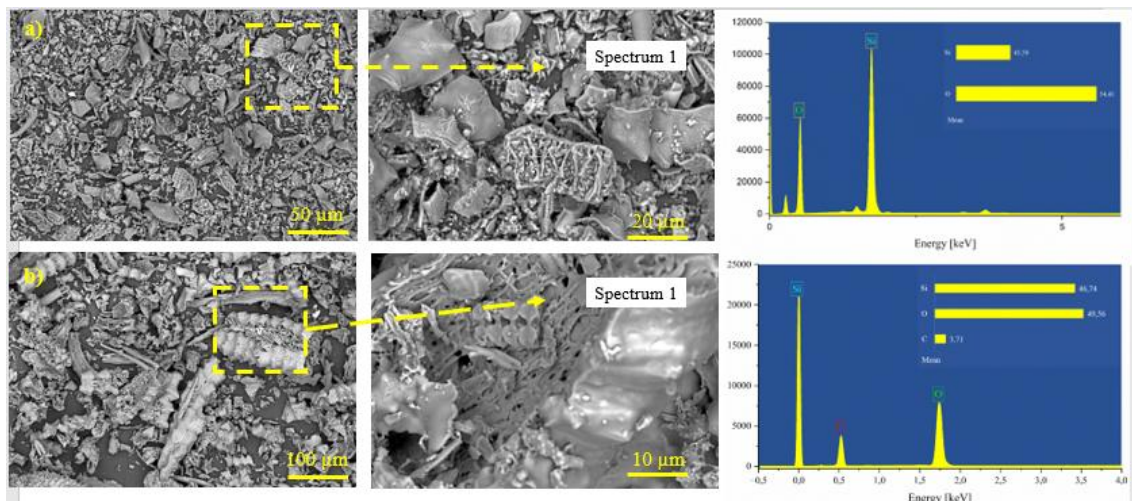


Figure 5 — SEM images and EDS analysis of silicon dioxide (SiO_2) particles obtained from rice husk: a — amorphous silica; b — crystalline silica after thermal annealing.

Conclusion

— During the study, silicon dioxide (SiO_2) was successfully synthesized from rice husk ash through acid leaching followed by thermal treatment. It was established that three-day leaching in an HCl solution and subsequent calcination at 800 °C enable the production of high-purity amorphous silica with an SiO_2 content exceeding 96 %.

— X-ray diffraction (XRD) analysis revealed that thermal treatment at 800 °C promotes the formation of an amorphous silica structure, whereas increasing the temperature to 1200 °C results in the crystallization

of the material with the formation of quartz and cristobalite phases. FTIR analysis confirmed the presence of characteristic Si–O–Si bonds and hydroxyl groups typical of silica.

– Particle size distribution analysis showed that the median particle size of the synthesized powder was $D_{50} = 88.9 \mu\text{m}$. The obtained results confirm the effectiveness of the proposed rice husk recycling approach and demonstrate the potential of the synthesized SiO_2 as a powder feedstock material for thermal spray coating technologies.

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Газотермиялық бүрку үшін күріш қабығының күлінен кремний диоксиді негізіндегі аморфты және кристалды ұнтақ композиттерінің синтезі

Жұмыста күріш қабығының күлін термиялық және химиялық өңдеу әдістерімен 800 °C температурада 3 сағат ішінде өңдеп, кремний диоксидін (SiO_2) синтездеу ұсынылған. Алынған материалдың құрылымдық және физика-химиялық қасиеттері рентген дифракция (XRD), инфрақызыл спектроскопия (FTIR) және лазерлік дифракциямен бөлшектердің өлшемдік таралуын талдау әдістерімен зерттелді. Алынған кремний диоксиді (SiO_2) бөлшектерінің орташа мөлшері $d_{50} \approx 88,9$ мкм және құрылымы аморфты. Кремний диоксидін (SiO_2) кристалдау үшін 1200 °C температурада күйдірілді және нәтижесінде кристалдық фаза (кварц, кристобалит) пайда болды. Инфрақызыл спектроскопия (FTIR) талдауы 1000-1100 см⁻¹ аймағында интенсивті сіңіру жолағы, сондай-ақ гидроксил топтарына сәйкес келетін жолақтарға тән Si–O–Si байланыстарының барлығын растады. Жүргізілген зерттеулер нәтижесінде газотермиялық бүрку технологияларында ұнтақ материал ретінде пайдалануға жарамды күріш қабығының күлінен синтезделген кремний диоксиді негізіндегі кристалды композиттер алынды.

Кілт сөздер: кремний диоксиді (SiO_2), күріш қабығы, химиялық өңдеу, күйдіру, бөлшектердің мөлшері, FTIR-талдауы

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Синтез аморфных и кристаллических порошковых композитов на основе диоксида кремния из золы рисовой шелухи для газотермического напыления

В работе представлен синтез диоксида кремния (SiO_2) из золы рисовой шелухи, осуществлённый методом химической обработки с последующей термической обработкой при температуре 800 °C в течение 3 часов. Структурные и физико-химические свойства полученного материала исследовали методами рентгенодифракционного анализа (XRD), инфракрасной спектроскопии (FTIR) и анализа распределения частиц по размерам методом лазерной дифракции. Полученный диоксид кремния (SiO_2) со средним размером частиц $D_{50} \approx 88,9$ мкм характеризовался аморфной структурой. Для его кристаллизации был проведён отжиг при температуре 1200 °C, в результате которого сформировалась кристаллическая фаза (кварц, кристобалит). FTIR-анализ подтвердил наличие характерных связей Si–O–Si с интенсивной полосой поглощения в области 1000–1100 см⁻¹, а также полос, соответствующих гидроксильным группам. В результате проведённых исследований получены кристаллические композиты на основе диоксида кремния, синтезированного из золы рисовой шелухи, пригодные для использования в качестве порошкового материала в технологиях газотермического напыления.

Ключевые слова: Диоксид кремния (SiO_2), рисовая шелуха, химическая обработка, отжиг, размер частиц, FTIR-анализ

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