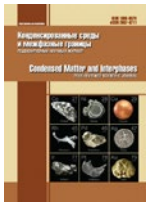

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Silicon in carboxymethylated rice husk: electron microscopy and photoelectron spectroscopy studies

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Abstract

Objectives: The work is dedicated to studies of the morphology and composition of particles obtained from rice production waste – carboxymethylated husk of rice grains, which contains a significant amount of silicon for crop products. The processing of rice production waste, as a rule, involves various technological processes, which may result in the composition changes of the final product up to the complete removal of silicon. An important task for research and subsequent industrial use is to establish the presence of silicon in the composition of processed rice husks.

Experimental: The preliminary milled rice husk was subjected to ultrasonic treatment followed by carboxymethylation. The materials were studied using scanning electron microscopy and energy dispersive microanalysis as well as X-ray photoelectron spectroscopy. Data on the morphology of particles formed as a result of processing rice husks have been obtained. The presence of silicon atoms on the particle's surface and in the bulk of the rice husk milling product, as well as after its processing, has been investigated.

Conclusions: Scanning electron microscopy, energy dispersion microanalysis and X-ray photoelectron spectroscopy have been successfully applied to study rice husk processing products. The treatment included milling, ultrasound exposure and carboxymethylation. The morphology of the treated particles with sizes up to units and tens of micrometers has been studied. Silicon was detected in the studied objects both before and after processing. On the surface and within the bulk of the materials, silicon atoms were found in charge states ranging from Si^0 to Si^{4+}. Neither ultrasonic treatment nor subsequent carboxymethylation had any noticeable effect on the presence of silicon atoms or their charge states.

Keywords: Rice grain husks, Ultrasound, Carboxymethylation, Scanning electron microscopy, Energy dispersion microanalysis, X-ray photoelectron spectroscopy, Morphology and composition

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Научная специальность ВАК – 1.3.8. Физика конденсированного состояния

Кремний в карбоксиметилированной рисовой шелухе: исследование методами электронной микроскопии и фотоэлектронной спектроскопии

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Аннотация

Цель работы: Работа посвящена исследованиям морфологии и состава частиц, полученных из отходов рисового производства – карбоксиметилированной шелухи рисовых зерен, которая содержит в своем составе значительное для продуктов растениеводства количество кремния. Переработка отходов рисового производства, как правило, сопряжена с различными технологическими процессами, результатом которых могут быть изменения в составе конечного продукта, вплоть до полного удаления кремния. Актуальным вопросом для исследований и последующего промышленного использования является установление присутствия кремния в составе переработанной рисовой шелухи.

Экспериментальная часть: Предварительно измельченная рисовая шелуха подвергалась ультразвуковой обработке с последующим карбоксиметилированием. Материалы изучались методами растровой электронной микроскопии и энергодисперсионного микроанализа, а также рентгеновской фотоэлектронной спектроскопии. Получены данные о морфологии частиц, формирующихся в результате переработки рисовой шелухи. Исследовано присутствие атомов кремния на поверхности и в объемной частиц продукта измельчения рисовой шелухи, а также после ее переработки.

Выводы: Растровая электронная микроскопия, энергодисперсионный микроанализ и рентгеновская фотоэлектронная спектроскопия успешно применены для изучения продуктов переработки рисовой шелухи. Обработка включала измельчение, воздействие ультразвуком и карбоксиметилирование. Изучена морфология обработанных частиц с размерами до единиц и десятков микрометров. Установлено, что кремний входит в состав изученных объектов до и после обработки. На поверхности и в объемной части изученных материалов атомы кремния находятся в зарядовых состояниях от Si^0 до Si^{4+}. Обработка ультразвуком и последующее карбоксиметилирование не оказывают заметного влияния на наличие атомов кремния и их зарядовые состояния.

Ключевые слова: шелуха рисовых зерен, ультразвук, карбоксиметилирование, растровая электронная микроскопия, энергодисперсионный микроанализ, рентгеновская фотоэлектронная спектроскопия, морфология и состав

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Introduction

Agricultural crop production waste is used in various modern technologies and rice production is no exception. It is known that the husk of rice grains which is one of the main components of rice cultivation waste is rich in silicon [1–2]. Numerous studies are presented in the scientific literature, which are devoted to the physico-chemical nature of silicon in rice husks as well as various ways of its application [1–5]. One of the most promising applications of rice husk and its processed products is the synthesis of composite superabsorbents [6], hydrophilic polymer materials capable of absorbing and retaining water in quantities hundreds of times greater than the polymer's own mass, as well as the isolation of silicon for the needs of the microelectronic industry [7].

Today, various approaches to processing rice husks are based on physical or chemical methods of its modification as well as their various combinations. These approaches, as a rule, lead to a change in the morphology, structure and composition of these raw materials. For example, during mechanical milling or hydrophilization by introducing carboxymethyl groups into the polysaccharide fraction of finely dispersed raw materials obtained from rice husks. In this regard, the analysis of the silicon content in the modified material and possible physico-chemical changes in the environment of its atoms during processing remains an important task.

Scanning electron microscopy (SEM) in combination with energy dispersive microanalysis (MA) is one of the most universal approaches to studying the morphology and elemental composition of microstructures including those of natural origin [5, 8–10]. X–ray photoelectron spectroscopy (XPS) is a high precision method for determining the physico-chemical state of the material's surface sensitive to the local surrounding of given atoms [11–13].

This paper presents the results of the morphology studies of particles formed as a result of milling and carboxymethylation of rice husks. The presence of silicon atoms on the surface and in the bulk particles of the rice husk milling product as well as after its carboxymethylation has been investigated.

Experimental

Rice husk crushed in a ball mill to a particle size of no more than 1000 μm was used in this work (sample «Init»). The carboxymethylated husk sample («Treat») was obtained as follows. Rice husk (3 g) was dispersed in 20 mL of a 10% aqueous NaOH solution for 1 h, then 40 mL of a 20% solution of sodium chloroacetate (>98%) in ethyl alcohol was added. The resulting mixture was exposed to ultrasound for 40 min using the UZG13-0.1/22 disperser at a nominal power of 0.1 kW. After ultrasonic treatment the reaction mass was kept at room temperature for 8 h. The target product was isolated by centrifugation (5000 rpm, 5 min), washed with an aqueous alcohol solution to neutral pH, and dried in a vacuum dryer at 45 °C to constant weight.

A JEOL JSM-6510LV scanning electron microscope equipped with a energy dispersive microanalysis unit was used. The secondary electron registration mode was applied. To estimate the particle sizes after milling the Image J software package was used. Energy dispersion microanalysis data were obtained from characteristic areas of the studied materials with a diameter of ~ 10 microns while estimating the depth of the probed layer at least 1 micron.

The «XPS» data were obtained on the ESCA module of the Kurchatov synchrotron ultrahigh vacuum experimental station NANOPES of the National Research Center «Kurchatov Institute» (Moscow), equipped with a SPECS Phoibos 150 hemispherical electron energy analyzer and a monochromatized X-ray source (Al Ka, 1486.61 eV) [14]. The depth of the informative layer was ~ 2-3 nm [15]. Survey spectra in the binding energy range of 0-1000 eV were recorded. A standard approach based on independent pure gold foil (Au 4*f*) signal detection was used to normalize and calibrate the data. To identify the features of the survey spectra well-known databases were used from which the most relevant data were selected [15–17]. To evaluate the selected components of the high-resolution XPS spectra recorded (decomposition into components was performed using the Lorentz and Gauss functions) a standard procedure for removing the Shirley spectral background was used [11]. In the XPS experiments a focused source of argon ion etching was used to remove the upper

layers of the material's surface under *in situ* study which was used in the «soft» mode adopted for samples of natural origin. The ion-accelerating voltage was 1 kV with an accumulated etching duration of 35 minutes which led to the removal of a surface layer with a thickness of at least 50 nm. The area of the etching site was selected in excess of the surface area from which the XPS data were recorded.

Results and discussion

An image of a native surface of rice grain husk fragment before milling is shown in Fig. 1a and the result of milling is shown in Fig. 1b. The result of milling is the fragmentation of the initial bio-raw material elements into individual particles measuring ~100 microns and constituting the «Init» sample. The surface of such particles does not retain the ordered macrostructure inherent in the source material as regular sequences in the form of ~50 microns formations separated by grooves of 10–25 microns. Morphological elements of the surface of particles after milling (Fig. 1b, sample «Init») are chaotic and blurry.

The results of SEM for the material after ultrasonic treatment and the introduction of carboxymethyl groups are shown in Fig. 1c (sample «Treat»). It is noticeable that the chosen processing method leads to even more noticeable and significant fragmentation and the formation of particles with sizes significantly smaller than in the source material: numerous particles have sizes less than 10 microns and can be collected in agglomeration. An estimate of the number of particles composing the treated material («Treat») and their size (maximum length) is shown in Fig. 1d and confirms fragmentation of the processed material with the formation of a significant number of microparticles measuring 10-20 microns and possibly less. The elemental composition (at. %) of the initial and processed materials (Fig. 1e) has the same content of silicon atoms. The presence of sodium atoms (and to a small extent chlorine) is the result of chemical modification – the formation of the sodium salt of the rice husk carboxymethylation product as well as the presence of residual amounts of the modifying agent sodium chloroacetate.

Thus, carboxymethylation does not significantly remove silicon atoms from the resulting particles. At the same time, it is obvious that the specifics of the local surrounding of silicon atoms in the surface layers and in the «bulk» part of the studied material may differ.

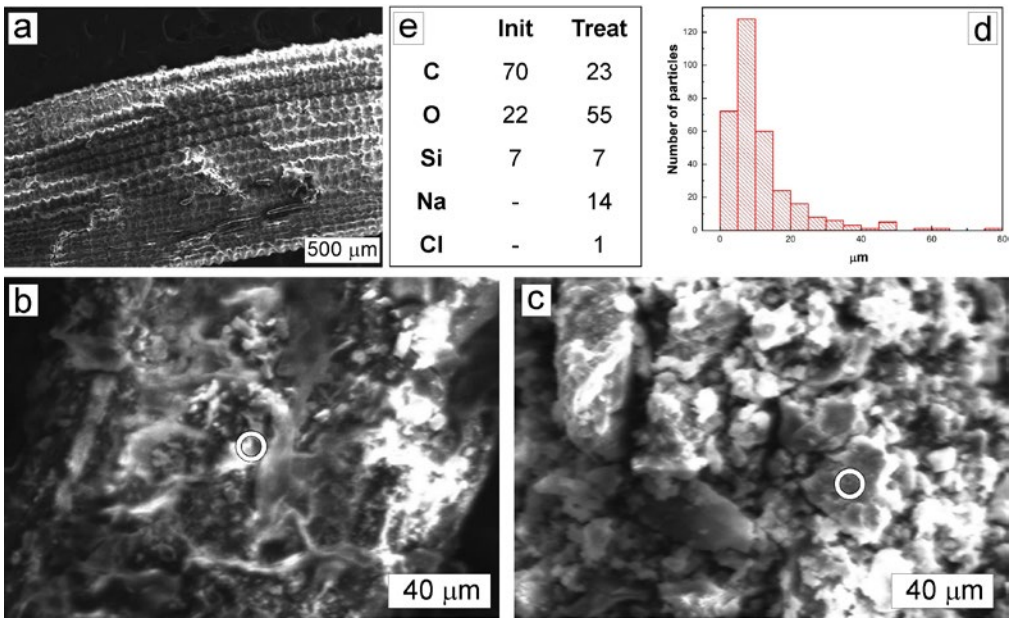

Fig. 1. Morphology of particles formed from rice husks as a result of milling and carboxymethylation. a: fragment of native rice husk; b: milled rice husk («Init»); c: carboxymethylated rice husk («Treat»); d: estimation of the number and particle sizes of the «Treat» sample after carboxymethylation; e: composition of the studied particles according to the MA data (at. %), the area of receiving MA data is highlighted by a circle.

Moreover, the degree of fragmentation of the material shown during processing indicates a noticeable increase in the contribution of the surface to the properties of the resulting material. The XPS method was used to determine the physico-chemical state of the surface and near-surface layers of the materials under study and to determine the nature of the local surrounding of silicon atoms that affects their charge state.

In Fig. 2a the XPS survey spectra for the «Init» and «Treat» samples are presented containing all the characteristic features including after removal of the surface layer as a result of *in situ* treatment (etching) with an ion beam. The

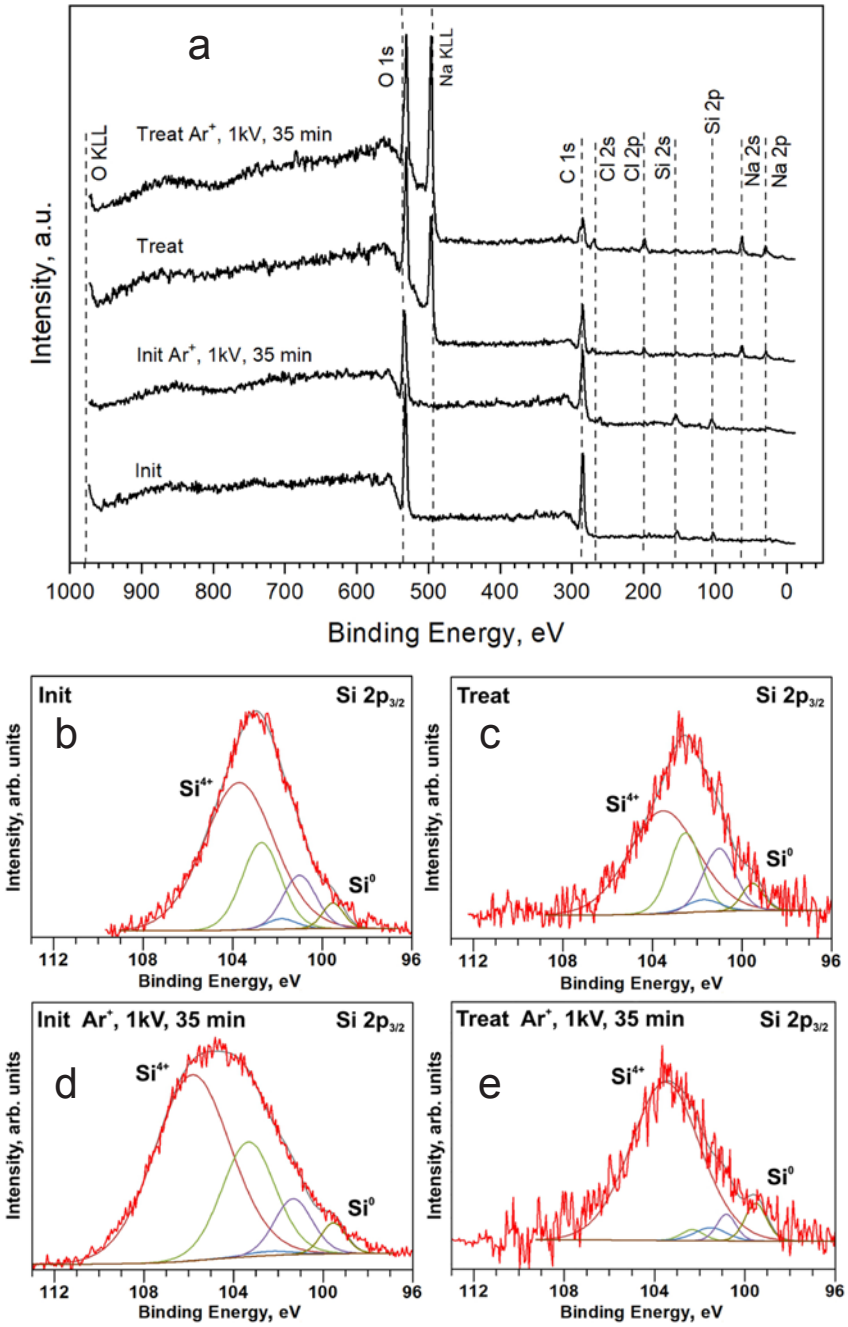

Fig. 2. XPS for particles formed from rice husks as a result of milling and carboxymethylation. a: survey spectra; b and d: Si 2*p* spectra and their modeling for particles after milling, before and after ion etching, respectively; c and e: Si 2*p* spectra and their modeling for particles after milling and carboxymethylation, before and after ion etching, respectively.

characteristic lines include «light» oxygen and carbon atoms as a result of the natural origin of the studied objects. And the characteristic lines of sodium and chlorine are the result of chemical modification of the milled particles. The source material (sample «Init») also contains a signal from silicon atoms. Observation of Si 2*s* and Si 2*p* lines at binding energies of ~150 eV and ~100 eV respectively (Fig. 2), regardless of the MA method, confirms the noticeable presence of silicon atoms in the surface of the studied object. For the «Treat» sample the signal in the region of the indicated lines of silicon atoms was observed at extremely low intensities in the survey spectra which probably indicates additional surface coverage as a result of the chosen processing method. Nevertheless, for both studied materials an attempt was made to record the spectra of the Si 2*p* line with high resolution to establish the charge states in which silicon atoms are located. The small number of silicon atoms led to a significant broadening of the detected spectral lines. To decompose the spectra into components corresponding to silicon atoms in charge states from Si^0 to Si^{4+} we used the method of accounting for the spin-orbit splitting of the Si 2*p* core level described earlier and demonstrated its application [18, 19]. This technique involves isolating the Si 2*p*3/2 part of the experimental spectrum to refine the energy positions of the components of a complex XPS line when decomposing it into components. Fig. 2b-e shows high-resolution spectra and the results of their modelling. Despite the significant broadening of the spectral lines, it seems possible, on a qualitative level, to identify the characteristic features of the 2*p* silicon spectra fine structure corresponding to atoms in the Si^0 and Si^{4+} charge states. These charge states are considered the most stable including during prolonged storage of rice husk processing products in the laboratory [4, 5]. The main contribution to the composition of the studied materials is made by silicon atoms in the Si^{4+} state regardless of the processing conditions. The most noticeable broadening of the Si 2*p* spectral line for the «Init» sample after ion etching is associated with additional surface charging as a result of such an effect on an object with complex morphology. Finally, the set of decomposition components of the recorded spectra suggests the possible presence of silicon atoms in the «intermediate» charge states of Si^{1+}, Si^{2+}, and Si^{3+} which were also previously observed [4] with various methods of processing rice husks.

Conclusions

A combination of scanning electron microscopy, energy dispersion microanalysis and X-ray photoelectron spectroscopy methods has demonstrated effectiveness in applying to rice husk processing products including after exposure to ultrasound and carboxymethylation. The proposed processing method makes it possible to obtain particle sizes up to units and tens of microns. The contribution of silicon atoms to the composition of the studied objects is shown. It has been established that silicon atoms are located on the surface and in the bulk of the studied materials in charge states from Si^0 to Si^{4+}. Ultrasound treatment and subsequent carboxymethylation have no noticeable effect on the presence of silicon atoms and their charge states in general, which can be used as part of the proposed approach to processing rice production waste to obtain functional materials based on natural origin raw – rice husks.

Contribution of the authors

The authors contributed equally to this article.

Conflict of interests

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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