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Development of a composite material based on a processed product of agricultural crop

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ABSTRACT

Today, soil contamination with heavy metals (HMs) and various wastes is one of the main problems on a global scale, which affects the food and environmental security of the population of Kazakhstan. Anthropogenic influence on the environment contributes to an increase in the content of heavy metals in soils. This is facilitated by the developed industrial sector, high population density and large-scale growth of urban areas. The areas are mainly contaminated by municipal, construction, and industrial waste landfills and rock dumps from the coal mining industry. The legislation of the Republic of Kazakhstan is focused on the transition from resource relations to relations aimed at rational use of natural resources, one of the main components of which is the preservation of environmental quality. To improve the soil content, peat, manure compost, and local loosening materials should be applied annually. One such material that holds great promise is rice husk (RH). When growing rice, a large amount of waste, primarily rice husks, accumulates annually. The content of organic matter in it reaches 82%. When processing 1 ton of rice grains, the share of rice husks is about 160 kg. Despite a significant number of developments in methods for recycling husks, most of them are not recycled, which increases the burden on the environment. However, the potential of rice husks in soil remediation is significant. The silicic acid gel present in the husk makes it possible to obtain valuable silicon-containing products. A significant part of scientific work is devoted to the production of silicon compounds from rice straw (RS). However, the most effective seems to be complex processing of RS, allowing the use of its inorganic and organic components. The result of processing can be composite materials that combine low density and sufficient strength of the cheap organic part of the RS, durability and temperature stability of the inorganic processed product. The development and implementation of new resource-saving technologies for the restoration of technogenic soil pollution, aimed at preventing the adverse impact of human economic activities on the environment, are currently a priority and relevant.

Keywords: Rice husk, Heavy metals, Erosion, Compost.
Article type: Research Article.

INTRODUCTION

One of the most important places in the system of agrotechnical measures that ensure stable crop yields and increased soil fertility is the use of organic and mineral fertilizers, which influence the soil's chemical, physical, and biological properties. Taken together, organic and mineral fertilizers and pesticides form the basis of the chemicalization of agriculture (Golubeva 1990; Lipin 2013). Soil degradation, environmental pollution, deterioration in the quality of agricultural products, global climate change, and energy shortages require the

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widespread introduction of new, environmentally friendly and at the same time highly effective methods of farming. The diversity of soils, most of which have a low level of natural fertility, suggests the need to develop farming systems that consider the maximum extent of the soil-ecological conditions of a particular region, that is, meeting the requirements of adaptive agriculture (Sergienko 2004; Workman 2018; FAO 2018; Nelson 2018; Lysenko 2019). More promising raw materials for producing amorphous silica on an industrial scale are still large-scale renewable waste from rice production, particularly fruit shells (husks), which are concentrated in grain cleaning enterprises. Waste is a potential raw material for various industries in rice production. First of all, attention is drawn to the high silicon content in the microporous organic matrix, which determines the wide possible areas of application of the waste as a raw material for obtaining materials with pronounced sorption properties, manifested after heat treatment (high-ash activated carbons), and elemental silicon. Rice is one of the most valuable food products in the world, ranking first in gross harvest and second in cultivation area in the world after wheat (Voronkov 1978; Zakharov & Vlasov1991, 1995; Zakharov 1995; Vinogradov & Bylkov 2000; Zelenkov 2004; Yu 2015; Kholomeydik 2016). In Kazakhstan, rice processing plants annually produce about 60 thousand tons of rice waste, which is sent mainly to landfills, leading to some serious environmental difficulties. In other rice-growing countries, the problem of processing rice waste has practically not been solved and has yet to lose its relevance for many years (Ma & Takahashi 2002). In this regard, the development and study of the chemical composition of rice husk and the production of domestic fertilizer of plant origin, characterized by low bulk density and high adsorption capacity, is a pressing topic.

MATERIALS AND METHODS

Rice husks were used as the starting material for obtaining samples. The rice husk is chosen because this raw material contains cellulose, lignin, and mineral ash, consisting of 92-97% silicon dioxide, which are useful substances for the human body. Rice husk raw material is a rapidly renewable source and is environmentally friendly. The work also used hydrogen (grade A GOST 3022-80), H_2 - 99.92%, (O_2, CH_4, CO_2, CO) - 0.08%, H_2O - 0.004%; Propane; Argon (high purity GOST 10157-79); Ar - 99.992%, O_2 - 0.0007%, N_2 - 0.006%; Nitrogen N_2 – 96.30%, O_2 – 3.70%. The sample synthesis was carried out under isothermal conditions. The samples were modified in a rotating reactor in an inert environment at a temperature of 650 ± 10 °C, an argon supply rate of 50 cm^3 min^{-1}, and a contact time of 20-30 min. The installation diagram is shown in Fig. 1. The activation process was as follow: the activator gas (CO_2) was supplied starting from 300 °C up to the selected activation temperature (800 °C, 450 °C), at which the sample was kept for a certain time. The AAS method was carried out to determine the sorption of heavy metal ions by phytosorbent based on plant fiber, based on measuring the degree of sorption on an atomic absorption spectrometer. It is a device designed for carrying out quantitative elemental analysis (up to 70 elements) using atomic absorption spectra, primarily for determining the content of metals in solutions of their salts: in natural and waste waters, in mineralized solutions of consistency products, technological and other solutions.

Fig. 1. Carbonization installation diagram.

The operating principle of an atomic absorption spectrometer is based on measuring the absorption value of a beam of light passing through the atomic vapor of the sample under study. An atomizer is used to convert the test substance into atomic vapor. Various narrowband light sources are used as a light source.

Leaching and acid treatment process

The leaching process is carried out to remove impurities and obtain a high cellulose content. The leaching process involves the fact that raw materials of enterosorbing dietary fiber are subjected to alkaline treatment with 10%

NaOH. A 500-mL of 10% NaOH solution was added to rice husks (200 g sample). Boiling was carried out for 3 hours, twice, then washed and dried. The acid treatment process was carried out to open the pores and increase the adsorption capacity. Acid treatment was carried out as follows: 5% nitric acid was added to 350 g of sifted, washed, and dried rice husks in a ratio of 1: 3-7 rice husks and nitric acid in the total mass. Then it was placed in a 3-L measuring glass, stirred with a glass rod, and left for 14 hours. The resulting composition was boiled at a temperature of 75-85 °C for 3 hours, cooled to room temperature, washed, and neutralized with distilled water to pH 7. The resulting enterosorbing dietary fiber was dried and crushed for carbonization and activation of enterosorbing dietary fiber raw materials. The process of carbonization and activating samples was carried out under isothermal conditions. The modification of the samples took place in a rotating reactor in an inert environment at a temperature of 650 ± 10 °C, an argon supply rate of 50 cm^3 min^{-1}, and a contact time of 20-30 min. The activation process was carried out as follows: the activator gas (CO_2) was supplied starting from 300 ℃, up to the selected activation temperature (800 °C, 450 °C), at which the sample was still kept for a certain time.
The reactor is made of heat-resistant chromium steel. It is equipped with a heater and a rotation mechanism. The temperature in the reactor was maintained with precision ± 5 °C. They are loading up to 500 catalysts. The volume of the reaction chamber was 3000 cm^3. The carbonization time was constant. The gas mixture supply rate was 50 mL per minute—contact time: 60 minutes. Carbonization was carried out at temperatures of 650-750 °C with an interval of 25 °C. Propane was used as a carbon source. The electric furnace was heated using a temperature controller, which maintained the required temperature in the rotating reactor. Hydrocarbon was supplied through the gas supply system at a predetermined speed. The hydrocarbon vapors entrained in this process were introduced into the reactor by the carrier gas. Next, the effect of carbonization temperature on the change in carbon content was studied using elemental analysis. It was found that the maximum carbon content for the samples is observed at 800 ºC and reaches a value of 51.1 wt%. at 850 ºC (Fig. 1). The carbon content in the initial sample of the substance after drying is 35.4 wt (%). Based on the uneven growth of carbon content, it can be assumed that the carbonization process takes Place in several stages. At relatively low heating temperatures, predominantly pyrolytic elimination of water occurs; at higher temperatures, the elimination of low molecular weight carbon-containing products occurs.

RESULTS AND DISCUSSION

Rice husk was used as a basis for determining the chemical composition of rice husk and its value when used as a fertilizer for soil remediation. It is known that over the past half-century, the humus content in chernozems in the southern regions of Kazakhstan has decreased by 25-35%. The soil cover is subjected to desertification processes due to an increase in temperature of almost 2 ℃ in this zone. After the development of virgin lands in the 1950s of the last century, the cultivated land area decreased due to wind erosion and soil deflation. It is difficult to overestimate the importance of soils in the biosphere and the population's life. Soil is the main natural resource, practically non-renewable within the historical period, the greatest, irreplaceable national treasure of the people, and the nation's gold fund. This heritage is intended by nature for all generations - now living and subsequent ones. It should be inherited not in a deteriorated state but in an improved form. In this regard, we studied rice husks to study the physicochemical processes of the relationship and mechanisms occurring with the EcoSoil fertilizer and the soil as well as the influence of these processes on the structure formation and strength of the soil cover. The rice husk structure has an inner and outer surface. The outer surface consists of jagged, rectangular elements with a high silica content, covered with thick cockpit and hair. The inner surface consists of elongated subcutaneous cellulose fibers. The middle region is transitional and also contains little silica. During rice growth, unique nanoporous silica layers are formed in the RH, providing access to air and moisture to the grain. The main characteristics of the feedstock (RF) depend on the chemical composition of carbon and mineral components, weather conditions of cultivation and storage, and the region where rice is grown. The outer surface of rice husks has a unique "lumpy" structure. The inner surface is quite smooth and fibrous. For many varieties of rice, the husk length is 2 - 5 mm of the length of the rice grain, and the bulk density is approximately 0.1 g cm^{-1}. The volume of the husk can be reduced by almost half by grinding, which is important when transporting and storing this material. The hardness of RS on the Mohs scale is 5.5 - 6.5 units. The true density of RS is 0.735 g cm^{-1}, and the bulk density is only 0.1 g cm^{-1}. RS crushed to varying degrees has a bulk density - from 0.19 - 0.21 to 0.38 - 0.40 g cm^{-1}. The average bulk density of RS after grinding is 83 - 125 kg m^{-3}, while the specific gravity of the original RS is approximately 0.075 g cm^{-3}. Grinding RS to different particle fineness increases its average density by 2 - 5 times.

RH in an undisturbed state has a thermal conductivity of about 0.056 W/m-K, and in a ground state approximately 0.067 W/m. The article shows an assessment of the effectiveness of using sorbents of plant origin for the neutralization of oil-contaminated soils. Rice husk, a large-scale waste that pollutes the environment, was used as a sorbent. Under laboratory conditions, the cellulose-lignin structure of the husk was destroyed. In fact, the RS has a variable composition. It depends on the type of rice, the presence of bran and fragments (scrap) of rice grains, geographical indicators, harvest season and other factors. The rice husk's chemical composition and basic properties are known in the literature. The rice husk was chosen because it contains cellulose, lignin, and mineral ash, which consist of 92-97% silicon dioxide, and useful substances for the human body. Rice husk raw material is a rapidly renewable source and is environmentally friendly. The work also used: (i) Hydrogen (grade A GOST 3022-80); H_2 - 99.92% (O_2, CH_4, CO_2, CO) - 0.08%, H_2O - 0.004%; (ii) Propane; (iii) Argon (high purity GOST 10157-79); Ar - 99.992%, O_2 - 0.0007%, N_2 - 0.006%; (iv) Nitrogen (N_2) – 96.30%, O_2 – 3.70%. The data is presented in Table 1.

Table 1. Chemical composition of rice husk according to the results of X-ray spectral analysis (wt.%).

Component	Content (% of mass)
Water	3.75-24.08
Ash	11.86-31.78
Pentosan	4.52-37.0
Cellulose	34.32-43.12
Lignin	19.2-46.97
Protein	1.21-8.75
Fats	0.38-6.62

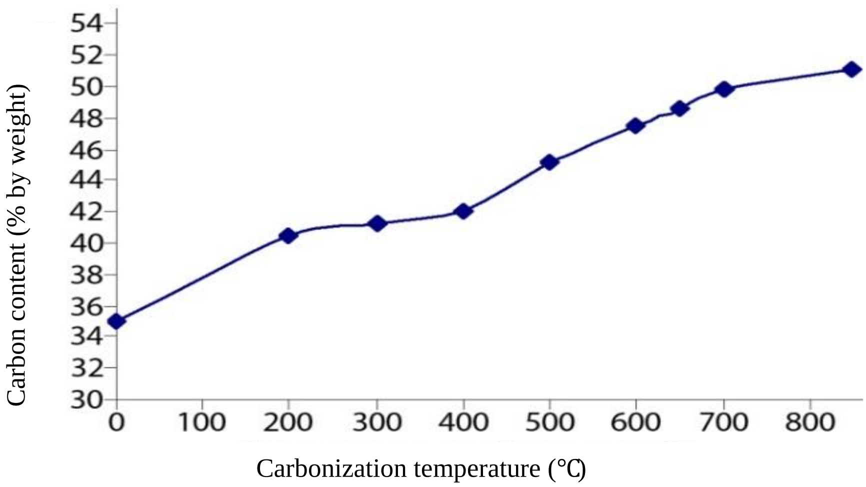

Fig. 2. Dependence of carbon content (wt.%) on the carbonization temperature of the sample.

Samples carbonized at different temperatures differed significantly in appearance. Thus, samples carbonized at low temperatures (300-500 ºC) were visually distinct from those carbonized at higher temperatures, with dark brown rather than black color. From this, we can conclude that the carbonization process up to a temperature of 500 ºC did not occur entirely but only partially. To obtain EPV with positive properties, it is advantageous to use a temperature of 700 ºC. Using a Quanta 200i 3D scanning electron microscope (FEI Company, USA), the elemental composition of the component of carbonized EPV samples was determined.

Table 2. elemental composition of samples.

Name of analysis	results
Sample 1 (carbonated, activated rice husk)	C – up to 74%, O – up to 13%, Si – up to 13%

As a result of the study, it was revealed that the components that make enterosorbing dietary fiber in a ratio of 1:10 (rice husk and carbonized rice husk) contain C, Si, and cellulose. The toxic elements were determined using the atomic absorption method, and it was shown that the EPV composite does not contain arsenic and mercury; lead and cadmium are contained within acceptable limits (Table 2). As a result, studying the carbonization of rice husks using an intensive heating pyrolysis oven without access to oxygen make it possible to obtain gaseous, liquid and solid products. As a result of the work, an experimental installation was created for complex waste-free thermochemical processing of rice husks to produce environmentally friendly, and safe carbon materials for wide industrial use. During the complex processing of rice husks, many chemical reactions occur, ultimately determining the possibility of obtaining certain products in a certain range of thermodynamic parameters. The chemistry of the process is very complex: many homogeneous and heterogeneous reactions take place. Thermodynamic analysis will allow us to outline ways to optimize the complex processing of rice husks. Various organic substances' pyrolysis processes (thermal destruction) underlie many technological processes (production of activated carbon, etc.). In addition, pyrolysis processes accompany the combustion and gasification of natural solid fuels (coal, etc.). Pyrolysis of organic substances is a complex physics-chemical process in which so-called volatiles are released from solid fuel in the form of gaseous and liquid products: hydrocarbons, resins, acids, etc. It is known that the total amount and composition of released volatiles, to a certain extent, depend on the thermal and other conditions under which the destruction process is carried out - the heating rate of particles, maximum temperature, etc. We obtained rice husk carbonate using an intense heating pyrolysis furnace without access to oxygen. We used dried RS, which was subjected to a carbonization process due to thermal-oxidative destruction or pyrolysis. It has been established that the solid carbonate residue contains carbon-containing and mineral parts, including components sufficient to produce activated carbon material and silicon dioxide. It was revealed that carbonization is an object of study for the processes of releasing silicon dioxide from the mineral component and activating the carbon-containing component. A black carbonized product was obtained, retaining its original morphological shape. The material after carbonization was subjected to grinding in a ball mill, followed by classification of particles by size; the highest content (83.98 wt.%) in the carbonizate after grinding is characterized by a fraction of particles with a size of < 0.1 mm. The need for grinding is due to the further operation of silicon leaching, the speed of which depends on the active surface of the solid phase. The pyrolysis process most fully satisfies the requirements for producing activated carbon (AC). In this case, heating the starting material to a temperature of 520 °C leads to a weight loss of 60%, which gives reason to believe that this method is economically possible for producing activated carbon (Fig. 2).

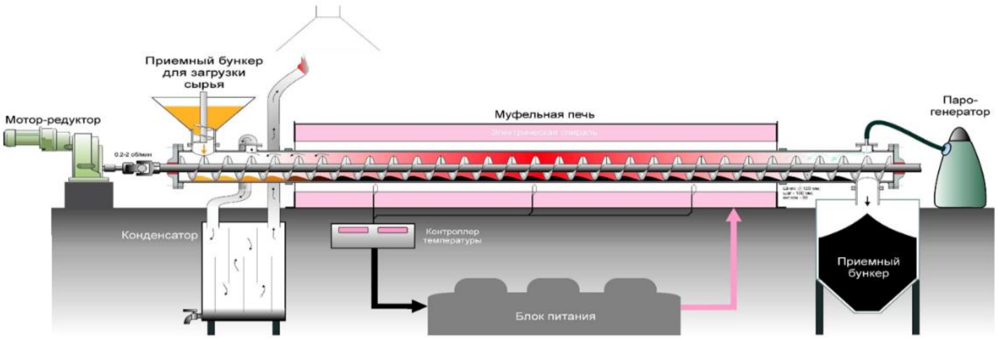

Fig. 3. Furnace installation diagram.

At the same time, when heated in argon, the composition of the exhaust gases changes: the CO content significantly decreases compared to combustion in air, as does the content of CO_2 and H_2O. For the production of activated carbon, we used rice husk, a raw material containing carbon. Thus, a typical process can be characterized as follows: the initial biomass is pre-crushed, dried, and then fed into the reactor; the necessary energy is provided by burning part of the biomass or using heat carriers (for example, sand). A system of cyclones and condensers allows the concentration of solid and liquid products. Ineffective pyrolysis technologies and gaseous products are used to generate the energy necessary for drying the initial biomass, maintaining the primary process, and using it by external consumers; some are returned to the reactor, which makes it possible to regulate the conversion parameters. The article determines the chemical composition of rice husk (RH). The value of rice husk when used as a fertilizer for soil rehabilitation has been established. It was determined that organic matter includes 34.3% cellulose, 19.2% lignin, 4.5% pentosans, and a few other substances. The primary biomass of RS consists of 3 polymers - cellulose, hemicellulose, and lignin. It has been proven that the main chemical elements of RS are oxygen, nitrogen, sulfur, and hydrogen. Mineral ash, consisting of 92-97% silicon dioxide, has beneficial properties for the human body and is environmentally friendly and cost-effective. A high concentration of silicon dioxide was found in RS on surfaces, the inner and outer epidermis, and intercellular epidermal cells. Used: Hydrogen (grade A GOST 3022-80); H_2 - 99.92% (O_2, CH_4, CO_2, CO) - 0.08%, H_2O - 0.004%; Propane; Argon (high purity GOST 10157-79); Ar - 99.992%, O_2 - 0.0007%, N_2 - 0.006%; Nitrogen N_2 – 96.30%, O_2 – 3.70%. It is known that replenishment and structuring of the soil requires the annual use of organic matter: peat, manure compost, local loosening materials. One of these materials is RS, where the organic matter content is 82%. Rice husks were added to the surface layer of soil and used for mulching, which helped reduce the evaporation of water from the soil and, therefore, save water, preventing drying out and overheating of the root layer. It has been proven that RSH is organic matter, which, when decomposing, forms humic substances. At the same time, soil biota and suppressiveness are restored. Free humic acids formed in the process are biologically active substances that not only stimulate the development of the root system and the plant as a whole as growth regulators and increase its adaptation to unfavorable environmental conditions but also have a fungicide effect, reducing the viability of fungal conidia and their development in soil. The Republic of Kazakhstan is currently faced with the problem of recycling large quantities of rice husks. Rice is one of the most important agricultural food products in the world. At the same time, the result of industrial rice processing is large volumes (up to 20% wt.) of husk. In major rice-producing countries such as China, India, and Vietnam, rice waste amounts to millions of tons. The bulk of rice husk (RH) is burned, which causes environmental damage, especially in countries with high population density (Ma *et al.* 2002; Ma & Yamaji 2006; Kaerlek 2012; Lim *et al.* 2012; Fernandes 2014; Sahebi 2015; Agostinho *et al.* 2017; Soerono 2018). Our article shows the disposal of large husks from rice processing as a result of firing the source material at a temperature of 600 ℃ led to a significant decrease in oxygen content from 41.81 to 25.16 wt.% and an increase in silicon content from 6.00 to 24.00 wt.%. The C/H ratio naturally increased to 29. The elemental composition and parameters of the porous structure of rice husk, carbonate, and material after separation of SiO_2 and activated carbon were studied. Activation of the resulting material at a temperature of 900 ℃ using potassium carbonate containing 9% wt. The mixture produced activated carbon with a specific surface area of 1218 m^2 g^{-1} and an adsorption volume of 0.710 cm^3 g^{-1}. It has been shown that due to the operations performed, the contribution of the micropore surface to the specific surface area increased to 50%.

CONCLUSIONS

1. It has been shown that the composition of rice husk, carbonation product, and material after separation of silicon dioxide differs significantly in the content of carbon, oxygen, hydrogen, and silicon.
2. It was revealed that after carbonization, the oxygen content significantly decreases from 41.81 to 25.16% wt., the silicon content increases from 7.00 to 25.00% wt., and the C/H ratio naturally increases to 30. This is a consequence of firing the initial material at a temperature of 600 °C.
3. Research has shown that the pyrolysis process can potentially be used to produce activated carbon because it technologically and cost-effectively produces activated carbon from rice husks.
4. The utilization of large amounts of husks resulting from rice processing is indicated. The elemental composition and parameters of the porous structure of rice husk, carbonate, and material after separation of SiO_2 and activated carbon were studied.

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