

# Ecological aspects of study of carbon adsorbents and their characteristics depending on rice husk processing conditions

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**Abstract.** The industrial production and use of adsorption materials, especially carbon adsorbents, is steadily increasing every year worldwide. Natural coals and plant waste are used to produce activated carbon. One of the promising raw materials in the Republic of Uzbekistan for the production of activated carbon is rice husk, because of its renewability and cheapness. The paper highlights the results of the study of processing of rice husks to separate the main substances - activated carbon. Pre-carbonization of PW at 600°C with subsequent impregnation with KOH solution and further thermal activation at 1000°C is found to increase the specific surface of coal to 1286 m<sup>2</sup>/g, whereas for coal sample obtained by thermal activation of carbonizate. The specific surface is 1108 m<sup>2</sup>/g after impregnation with a KOH solution. In all temperature ranges of activation of the surface, the characteristic is greater in the first sample. Therefore, rice husks should be processed in a sequence: carbonization at 600°C, 20% carbonization of KOH solutions at 120°C within 2 hours, thermal and activation with water vapor at temperatures above 850°C, which ensures higher surface characteristics of the coal produced.

## 1 Introduction

Worldwide, the industrial production and use of adsorption materials is steadily increasing every year. This is especially true for carbon materials, the number of annual use of which is more than 1 million ton. According to the analytical forecast, the volume of consumption increases every year by 9-10%. Non-renewable resources account for more than 85% of coal production, i.e. coal and petroleum products.

Popular raw materials such as coal, wood waste, coconut shell, peat, petroleum, and polymers are used to produce activated carbon. Despite its extensive use in the adsorption process, the biggest problem with activated carbon on an industrial scale is its high cost and difficulty in regenerating adsorbents [1-6]. In this connection, there is a need to find new

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renewable and inexpensive materials as an alternative to adsorbents in solving the problem of water pollution and recovery of waste gases from various processes.

It is known, however, that porous carbon materials can be derived from plant waste: tree measles and sawdust, logging residues and agricultural products [7, 8].

Such renewable materials also include rice straw and husks. According to data available in the Republic of Uzbekistan, rice production is about 600 thousand tons. As a result of processing of this amount of rice, about 20% of the mass is produced. rice husk. Therefore, research and introduction of developments in the field of production of adsorption-active materials based on rice husk [9] are considered relevant.

Carbon adsorbents are now widely used in adsorption cleaning, separation and concentration processes in liquid and gas systems [10]. Particularly relevant are the processes of drinking water treatment, sewage treatment of various industries in order to reduce environmental damage and return them to the production cycle. Without carbon adsorbents, there is no substitute for the pharmaceutical and medical industries. Widely used for cleaning and decontamination in the food industry, incl. the production of alcoholic and non-alcoholic beverages.

Rice husks contain about 75% of organic matter, of which about 25% are ash when burned. The organic substance of husks mainly contains cellulose, lignin and hemicellulose, as well as other components such as nitrogen and inorganic compounds. Lignin is about 25-30%, and cellulose about 35-40% [11].

Rice husk contains in its mineral part a significant amount of silicon dioxide, the nature of which has been explained in detail in some works [12]. According to research results, silicon is formed during the natural evolution of rice husk, distributed as monosilic acid, which moves to the outer husk shell, where it becomes cellulose by evaporation and concentrationsilica. Silicon dioxide in rice husks differs from other popular silicon-containing raw materials (quartz, cristobalite and others) in that it is amorphous, contains less metal impurities and is chemically more active.

Activated charcoal can be produced from rice husks in two ways: steam or chemical activation. Compared to steam gas, chemical activation has two main advantages: low activation temperature and higher product yield [13]. Activated carbon is also produced in a two-stage process that combines chemical and steam activation to produce a product with the required quality and performance.

When steam gas activation, that is, heat treatment of carbon-containing materials by oxidizing agents, part of the carbon is removed with volatile gases from the carbon matrix, developing the surface area. Oxidative gases such as carbon dioxide, water vapor, air and oxygen are often used as activators.

Activated carbon is produced by steam-gas activation of carbonated rice husk in operation [14]. According to studies, activated carbon was obtained by activating rice husk carbonization products with carbon dioxide, water vapor or wet carbon dioxide. The rice husk was washed with deionized water and then carbonized at 900 °C in the atmosphere for five hours of nitrogen, then treated with ozone. The results showed that in all cases, ozone treatment before activation produces more activated carbon with a specific surface area than raw samples. The maximum surface area has reached 417 m<sup>2</sup>/g when wet carbon dioxide is activated.

In [15] the carbonized rice husk was leached by sodium carbonate to separate silicon dioxide. The carbon product produced after the release of silicon dioxide from carbonization was activated using carbon dioxide and water vapor at 800 °C. This resulted in activated carbon with a specific surface area of 1514 and 1365 m<sup>2</sup>/g, respectively.

The aim of the research is to study the processes of PW processing and the influence of conditions on the output and change of the specific surface of activated carbon.

## 2 Materials and methods

Samples of rice husks selected in the Khorezm Oblast (October 2020) were selected as feedstock. Potassium hydroxide was used as a chemical modifier.

Before testing the composition and further heat treatment, the raw material passed the preparatory stage in the form of washing with water and drying at a temperature of at least 100°C. The husks were heat and chemical treated to produce coal. Chemical treatment was carried out both after (1-way) and before (2-way) heat treatment. The carbonization temperature was 600°C, because the possibility of high ash extraction (silicon oxide and various metals) and coal was established. The temperature of the carbonization was determined to be 600°C [16]. Pyrolysis and thermal activation were performed in the inert atmosphere of the argon feed to the chamber.

According to 1 method, the carbonizer produced at 600°C was chemically treated with a 20% potassium hydroxide solution at a ratio of carbonizate and activating reagent (1:1). The processing temperature was 120°C, and the process lasted 2 hours. The resulting mixture was then filtered. The solid residue after leaching mainly contains carbon (AU1).

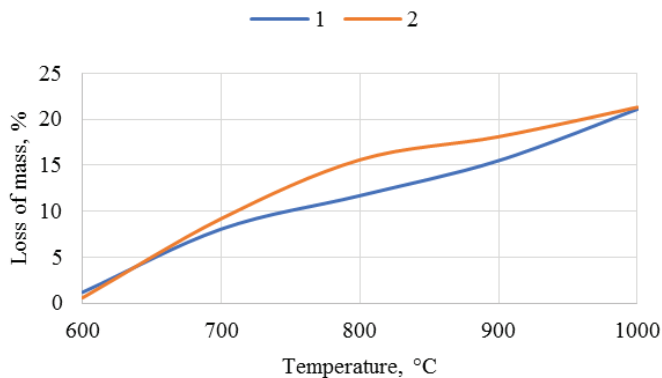
According to the 2-method rice husk was treated with a solution of potassium hydroxide in such a quantity that the first method. The system obtained after alkaline treatment was filtered and washed with water until it was completely neutralized. The solid phase was heat treated at 600°C for 2-3 hours, which mainly contains carbon (A2).

After the carbon containing material was produced, physical activation (600-950°C) was performed using water vapor.

The characteristics of the porous coal structure (surface area -  $S_{ud}$ , total pore volume -  $V_{\Sigma}$ , pore diameter -  $d_{pore}$ ) were determined by low temperature adsorption of nitrogen at 77 K at the static adsorption plant Quantachrome Nova 1000e. The adsorption-to-residual pressure curves were processed using the BET method.

## 3 Result and discussion

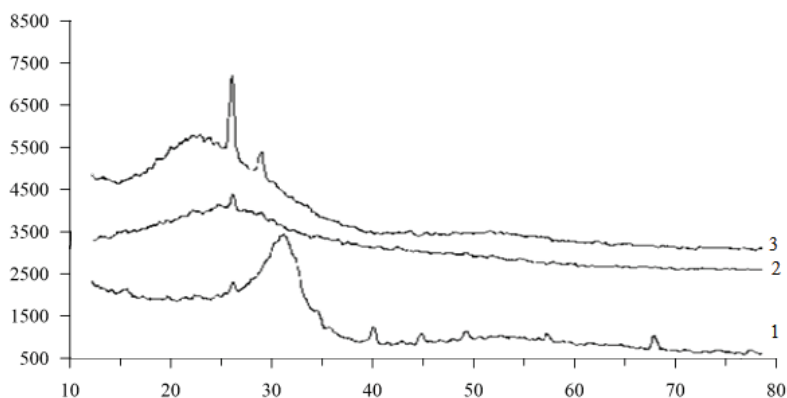
The carbon materials produced by the 1 and 2 methods were subjected to heat treatment at temperatures above 600°C. The change of mass in this process was investigated, the results of which are given in Figure 1.



**Fig. 1.** Coal mass change by thermal activation in inert atmosphere: 1) AU1; 2) AU2.

From the curves in Figure 1 it is possible to observe different thermal behaviour of coal materials obtained from the first (A1) and second (A2) methods. Heat treatment at a temperature of 600°C results in a decrease in the mass of AU1 and AU2 by 1.2 and 0.8%,

respectively. These differences are mainly related to the constituents of coal samples. The separation of carbon containing components, i.e. cellulose and lignin, probably prior to carbonization is associated with the destruction of more stable organic compounds in the original RW, which, when treated with an alkali solution, then the acids change and are separated with the ash part into silicon dioxide. This assumption is very valid when taking into account the large quantities of calcium, iron, phosphorus, sulphur and nitrogen components in the composition of 2DC compared to 1DC. However, when the calcination temperature rises to 700°C and vice versa, it helps to remove more mass from AU2. Therefore, the smaller change in mass at a temperature of less than 700°C is also due to the above-quoted components with a higher content of lignin containing substances that are more stable and at a given temperature form a large amount of resin substances, which are retained in the coal pores previously formed. A further increase in the treatment temperature leads to the removal of the resin data, which is mainly responsible for the mass change. The total mass change at temperatures above 950°C has the same values as already at 850°C the processes related to the removal of functional surface groups in the coal material are coming to an end, and further mass change is associated with the combustion of amorphous carbon and the formation of a more orderly graphite structure [17].



**Fig. 2.** The X-ray of a heat treated at different temperatures AU1 (°C): 1) 600; 2) 850; 2) 1000.

The X-rays of the AU1-600 and AU1-850 samples are typical amorphous materials. Besides the main lines characteristic of amorphous coal, lines for calcite, silica and graphite are found. Graphite reflexes ( $2\theta=26.6; 44.5; 50.8$ ) become more intense when the treatment temperature increases (Figure 2-2). Data recorded reflexes of crystallized carbon are previously described in the work [18].

As can be seen from the X-ray graph, the peak intensity of 002 is noticeably lower than that of typical natural coals, due to the less ordered coal structure based on vegetable raw materials obtained at 600°C. The further increase in the treatment temperature significantly increases the intensity of the peak, which is moved to a low angle area. However, the shape of the peak 002 remains wide to 950°C, probably due to the different size of the carbon particles rather than the degree of carbon amortization. Currently, existing scientific evidence has shown that active coal is a small crystalline carbon particle with a size of about 1-3 nm [19]. In contrast to the structure of tightly oriented graphite, individual planes in activated corners may not be in strict sequence. Therefore, X-rays exhibit broad peaks characteristic of amorphous compounds. The distance between the contacting particles, i.e. the planes is 3.355 Å, which is larger than graphite (3.650). The calculated size of the crystallites 1AU-950 and 2AU-950 according to the Sherrer equation is about 10-12 nm, so these crystallites can contain from 3 to 4 layers.

The effects of water vapour activation on the output and structure of coal samples were investigated. The mass loss during vaporization is slightly higher than without it, due to the additional oxidation and reaction with the activator molecules of some components of the coal material. The difference in mass loss is particularly noticeable at 650-900°C. At higher temperatures, the difference between these values is reduced. Consequently, the processes associated with polar water molecule change occur precisely within a temperature range of 650-900°C. However, some authors argue [20] that it is possible to obtain higher process values for the process of vaporization of carbon-containing raw materials within a temperature range of 700-950°C.

The main criterion in practice is considered to be the yield of the carbon product. The results of a study of the effect of the vapour vaporization process on the output showed that when using water vapor for activation within 1 hour at 75 ml/min, the porosity of 1AU and 2AU increases by 11 and 13%, respectively. The further activation process over the next 2 hours slightly increases the porosity of the samples under examination. However, after 4 hours of steam activation, there is a decrease in porosity. Also, when the temperature rises to 750-800°C, the porosity and the specific surface for both coal samples rise sharply, and further temperature increases also reduce the yield and specific surface of coal materials. This increase in duration is primarily due to the destruction of the porous structure already formed by deep reaction with water molecules at high temperatures.

The specific surface of the coal material based on the low-temperature adsorption isotherm of nitrogen vapour was calculated.

**Table 1.** Impact of activation conditions on output and specific surface\*.

Sample	Activation temperature, °C	Coal yield *, %	S, m <sup>2</sup> /g	Activation temperature, °C	Coal yield *, %	S, m <sup>2</sup> /g
	Water without vapor			Water with vapor		
1AU	600	1.2	895	600	1.3	1091
	700	8.07	605	700	5.1	989
	800	11.7	886	800	12.0	1101
	900	15.5	912	900	19.6	1206
	1000	21.1	998	1000	20.8	1286
2AU	600	0.6	701	600	0.7	961
	700	9.2	542	700	11.2	716
	800	15.6	756	800	18.1	892
	900	18.1	895	900	20.9	956
	1000	21.3	906	1000	21.5	1108

\*-duration of process 3 h.

As the table data show, under the same temperature conditions, the use of water vapours slightly increases the values of the specific surface, while the yield of the product is significantly lower at a temperature of up to 900 C. Thus, the best output and specific surface values for both samples were obtained during the process at a temperature of 800°C and a water consumption of 75 ml/min for 1.5-2 h. The mass loss is 12.0 and 18.1%, respectively, for 1AU and 2AU, and the specific surface values are 1101 and 892 m<sup>2</sup>/g.

## 4 Conclusion

Processes of thermal activation and their influence on porous coal structure based on rice husks have been investigated. Heat treatment at 600°C for 2 hours has been found to reduce the mass of 1AU and 2 AU by 1.2 and 0.8%, respectively. Further increasing the temperature to 700°C and more vice versa contributes to greater mass removal from A2. Therefore, the

smaller mass change at temperatures below 700°C is due to heteroatomic components, as well as a higher content of lignin, which is more stable and at a given temperature forms a large amount of resinous substances, which are retained in the coal pores previously formed.

The peak intensity of 002 is markedly lower than that of typical natural coals, due to the less ordered coal structure based on vegetable raw materials obtained at 600°C. Further increase in treatment temperature, significantly increases the intensity of peak. However, this peak remains wide to 950°C, due to the different size of the carbon particles rather than the degree of carbon amorphous.

Thus, the best yield and specific surface values for both PW-based coal samples were obtained from a process of 800°C and a water discharge of 75 ml/min for 1.5-2 h. The mass loss is 12.0 and 18.1%, respectively, for 1AU and 2AU, and the specific surface values are 1101 and 892 m<sup>2</sup>/g.

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