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Production of activated carbon granulated by treatment of rice husk and straw with an oil sludge using polyvinyl acetate as a binder

Banu Diyarova[2]*, Nurbol Appazov[1], Baurat Bazarbayev[1], Bulat Dzhiembaev[2], Olena Lygina[3], Tapalova A.S.[1]

[1]*Korkyt Ata Kyzylorda University, 120014 Kyzylorda, Kazakhstan;*
[2]*Kazakh National Women's Teacher Training University, 050000 Almaty, Kazakhstan;*
[3] *Universidade Nova de Lisboa, 2829-516 Caparica, Portugal*

Abstract
One of the man-made problems of technological development is the processing and utilization of oil and agricultural wastes. In this study, the process of obtaining granular activated carbon from the agricultural rice waste was investigated. The granules were obtained using polyvinyl acetate, which binds rice residue and oil sludge. Carbonization and activation were carried out in a quartz glass tube inside of high-temperature vacuum furnace BR-12 NFT series. The length of the furnace is 300 mm, the diameter is 60 mm and the operating heating length is 200 mm. Carbonization was performed at a temperature of 500 °C, activation of carbonizate with water vapor in ratio 1:2 at a temperature of 850 °C. The effect of polyvinyl acetate, rice waste, oil sludge ratio on the product characteristics was analyzed, the most advantageous ratio for obtaining granular activated carbon was identified. The iodine adsorption activity of the granulated activated carbon, the total pore size, the mass fraction of moisture, the methylene blue adsorption activity and bulk density were determined. According to the results, the product obtained by using the rice husk, oil sludge and binder in a ratio by mass 9:1:1.1 corresponds to the DAK type of wood crushed activated carbon standard (GOST 6217-74). In this study, we investigated the specific surface area of the material and its iodine adsorption activity. Our findings demonstrate a high specific surface area, along with excellent iodine adsorption activity, making the material a promising candidate for various applications in adsorption processes.
Keywords: activation; carbonization; rice waste; adsorbent; activated carbon.

1. Introduction

This research paper presents a novel approach for utilizing agricultural rice waste and oil sludge to produce granular activated carbon. The novelty of this study lies in the use of polyvinyl acetate as a binding agent to produce granules, which were then subjected to carbonization and activation in a high-temperature vacuum furnace. The paper investigates the effect of different ratios of polyvinyl acetate, rice waste, and oil sludge on the properties of the resulting granular activated carbon. The product characteristics such as iodine adsorption activity, total pore size, mass fraction of moisture, methylene blue adsorption activity, and bulk density were analyzed. The most advantageous ratio for obtaining granular activated carbon was identified. Overall, this study offers a new method for effectively utilizing agricultural waste and oil sludge to produce a valuable material with potential applications in various industries.

The disposal of biomass waste from agricultural production is one of the environmental issues. As a solution to this problem, the waste is often recycled into widely used materials, such as activated carbon with a high adsorption property. Activated carbon is a micro porous form of carbon with a porous structure, surface area, and high adsorption capacity [1–3]. Activated carbon has functional groups on the surface, which affects the pH of the solution and the adsorption process [4-5]. Activated carbon is widely used to purify drinking water, tap water and air, its physical chemisorption and adsorption mechanisms allow pollutants to move from liquid to solid surface [6-7]. Activated carbon is widely used for water and air purification due to its higher adsorption properties than others and ease of use. Activated carbon can be used as an alternative to traditional and advanced technologies [8]. Advanced technologies are not only evenly distributed around the world [9], but are also associated with their operating and maintenance costs [10]. For commercial purposes, alternating current is widely used from non-renewable sources such as coal, lignite, and peat. They have low mineral content, high carbon content, and high porosity, making them suitable for using activated carbon as a raw material

*Corresponding author e-mail: banu.diyarova@mail.ru.; (Banu Diyarova).
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source [11]. However, due to the environmental and economic problems associated with them, there has been a growing scientific interest in finding cheap biomass waste products as well as low-cost AC generation processes [12].

Activated carbon is sold in two forms: powdered activated carbon and granular activated carbon. Pulverized activated carbon has a high surface area and microhardness, which means that it increases their adsorption capacity [13-14]. However, finer particles of crushed activated carbon show a slower tendency to shrink and break than granular activated carbon [15]. Despite the increased adsorption capacity, due to the difficulty of separating powders from an aqueous solution [14], as well as due to the high sorption rate, powdered activated carbon is not restored [16]. Because of these difficulties, pulverized activated carbon is commonly used in batch mode, especially in water treatment plants [17]. Continuous columns are suitable for water treatment, and granular activated carbon is the most preferred type in structures where carbon recovery is critical for environmental and economic reasons. Granular activated carbon is due to the adaptation to continuous contact without the need to release carbon from the main liquid [18]. Compared to powdered activated carbon, granular activated carbon has a lower adsorption capacity for removing pollutants from aqueous solutions [19]. The propensity to adsorb granular activated carbon is due to the impact of pollution, as well as a decrease in the mass transport of target pollutants [20].

The production of activated carbon requires a higher lignin content because lignin contains more carbon than cellulose or hemicellulose. Lignin is the main component that affects the consumption of activated carbon [21]. Depending on the source of raw materials, the mass fraction of lignin in activated carbon ranges from 53.5–78.8%, hemicellulose 13.6–23.6%, and cellulose 6.2–30% [22]. In activated carbon, due to the increased content of lignin, the yield of semi-coke increases. Because lignin is thermally stable at high temperatures, and also because they have functional groups-OSN 3, which are prone to burns [23]. On the other hand, cellulose and hemicellulose consist of volatile fractions that are removed during pyrolysis and are associated with a low carbon yield, since a significant part of the carbon is lost during pyrolysis.

Granular activated carbon is more efficiently extracted from solid biomass feedstock. Granular activated carbon can also be obtained by granulating soft low density biomass wastes, as well as by granulating crushed activated carbon using binders to obtain granules and granules with improved adsorption properties [24]. However, in some cases, granulation can be performed without the addition of a binder [25]. A problem with granular activated carbon derived directly from biomass waste is its low abrasion resistance due to the high cellulose content of biomass waste, which makes it prone to fibers and brittleness, which is inefficient in the production of granular activated carbon. This disadvantage can be eliminated with the help of effective binders [17]. The granulation process can be carried out on raw materials, carbon raw materials [26], binder [27-28], spraying, vibration and extrusion. Granulating a mixture consisting of a powder and a binder, in contrast to activated carbon obtained by direct granulation, followed by grinding it to particles of the desired size, increases the strength and density of granular activated carbon [29-30].

Apart from the nature of the biomass waste, the process parameters also influence the quality of the granulated activated carbon obtained. These parameters include mix ratio (powder to binder ratio), molding pressure, activation temperature, order of addition (binder then activator or vice versa), and activation method.

Granulation is the process of converting coal grains of less than 6 mm in size into solid particles of a certain geometric shape. Granulation can be carried out in two ways: with and without a binder.

The advantage of the granulation method is the possibility of severe deformation by increasing the density of the particles and interaction between the molecules. The use of high-viscosity binders (such as pitch and cracking residues) can increase the mechanical strength and improve the sorption characteristics of activated carbon. Based on these properties, the method of granulation was chosen in this study.

Depending on the composition of the binders used the granulated process is divided into inorganic and organic. Inorganic binders are lime, clay, gypsum, cement, magnesite, trephine, alkalis, sodium and calcium phosphates. These substances can be used both individually and in combination with others. Organic binders can be coking coal, pitch, tar, resins, pulp, cellulose paper and various wastes of the food industry.

The following substances are widely used as binders in the production of granulated activated carbon: petroleumbitumen, lignosulfonates, molasses, liquidglass, cement.

In the process of obtaining activated carbon granules, mixing raw materials and binder is carried out at room temperature.

Natural affordable, and cost-effective binders for activated carbon granulation are currently being discovered. Products obtained during fractional distillation, wastes of cracking, its by-products and high molecular weight components are used as binders. The features of such materials are high viscosity, fluidity and low processing temperature.

However, in most cases, these materials do not provide the appropriate level of strength and may contain components harmful to the metallurgical industry. As an alternative to the described organic components, the use of various high-molecular compounds as binders was proposed. One such compounds is polyvinyl acetate. In the literature, polyvinyl acetate-based dispersions and emulsions, adhesive polyurethane solutions have shown the necessary properties. These polymers meet the primary requirements of price and quality.

Polyvinyl acetate (-[-CH_2CH ($OCOCH_3$)-] is an amorphous transparent polymer with high adhesive properties. Its relative density is 1.19 g/cm^3. Polyvinyl acetate is characterized by very low water absorption, moisture permeability and hydrogen gas permeability. 100% polymer is characterized by a tensile strength of 350 kPa at relative elongation. Polyvinyl acetate decomposes at temperatures above 180-200°C with the formation of acetic acid, in the presence of excess oxygen CO_2 and H_2O are formed, thermal oxidation by radical chain mechanism occurs.

In our previous research, the optimal way to obtain activated carbon by combining rice residue and oil residue was identified. Also, the most advantageous ratio for manufacturing granular activated carbon from rice residues and oil residues by using gelatin, lignosulfonate binder was determined [31-33].

The purpose of the research work: to determine the optimal ratio of the process of obtaining granular activated carbon with the incorporation of a polyvinyl acetate binder.

2. Materials and Methods

Rice residue was ground in the laboratory to a size of 0.25 mm. The granules were obtained by adding a binder to the crushed rice waste (husk and straw) and oil sludge in the ratios shown in tables 1-2. Carbonation of the granule was carried out in a nitrogen atmosphere at a temperature of 500°C, whereas the activation was carried out with water vapor in a high-temperature vacuum tube furnace BR-12 NFT series at a temperature of 850°C.

The iodine adsorption activity, total porosity, mass fraction of moisture, bulk density and the adsorption of methylene blue activity were determined and calculated by standard methods.

To determine the mass fraction of moisture, 1 g of briquetted activated carbon was weighed and placed in a pre-weighed box. The box lid was opened and placed in the oven at 105-110°C for 1 hour. After the set time, the box was removed from the drying oven and cooled in the desiccator for 15 minutes. The mass of dried activated carbon was measured and calculated [34].

In order to identify the iodine adsorption activity, a solution of iodine in potassium iodide with a concentration of 0.1mol/dm^3 was poured to a certain part of briquetted activated carbon and the solid was put to a mixer for 15 minutes to shake in an intensity of 100-125 oscillations. After the particles in the solution were settled down, the amount of liquid was taken with the aid of a pipette and titrated against 0.1 mol/dm^3 sodium thiosulfate solution using a starch indicator until the coloue change from blue to colourless was observed [35].

To identify the total volume of pores in water, pores in the range of 5-10*10^{-3} mm were heated and boiled for 15 minutes, afterwards they were pumped at a pressure of 8 kPa, in this step excess water was separated and weighed on an analytical balance. In order to determine the bulk density the mass of normalized compaction of a certain amount of activated carbon was measured [36-37].

The granules were obtained by adding a binder to the oil sludge with rice residue (husk and straw) in the ratio shown in tables 1 and 2. Obtained briquettes were hermetically sealed and placed in a tubular furnace, the nitrogen gas supplied from the cylinder was introduced to the tube, during the carbonization process the temperature was increased from 10 °C to 500 °C per minute and this temperature was kept for 100 minutes. Activation process was carried out at 850°C, the ratio of carbonizate (carbonizate is a product formed after the carbonization process) and water vapour was 1:2. The change in yield, physical and chemical properties of briquetted activated carbon while using different binder ratio was analyzed.

3. Results

The specific surface of the obtained activated carbons was determined from the thermal desorption of the argon-helium mixture on a Sorbtometer-M device (Russia). For the analysis, the optimal compositions were chosen according to the adsorption activity of iodine. Argon is adsorbed by activated carbon from a mixture of argon and helium at liquid nitrogen temperature and, after adsorption equilibrium is established, is desorbed when the temperature rises to room temperature.

The specific surface area of activated carbon obtained from a mixture in the ratio of rice straw: oil sludge: polyvinyl acetate = 9:1:1.25 is 117.66 m^2/g, the average pore size and specific pore volume are 1.71 m^2/g and 0.050 cm^3 /g, respectively. The average pore size of this activated carbon is 1.713 nm.

The specific surface area of activated carbon obtained from a mixture in the ratio of rice husk: oil sludge: polyvinyl acetate = 9:1:1 is 178.67 m^2/g, the average pore size and specific pore volume are 1.71 m^2/g and 0.077 cm^3/g, respectively. The average pore size of this activated carbon is 1.714 nm.

According to the results of the analysis, it can be judged that activated carbon obtained from rice husk has a more developed specific surface compared to activated carbon obtained from rice straw.

It was figured out that the optimal ratio for obtaining briquetted activated carbon from rice husk, oil sludge and polyvinyl acetate is 9:1:1.1. This product corresponds to the DAK type of wood crushed activated carbon standard (GOST *6217-74*).

According to the results in Table 1, briquetted activated carbon was obtained by adding rice straw, oil sludge and polyvinyl acetate in the ratio 10:1, 9:1:1.1, 9:1:1.25, 9:1:2. Activated carbon obtained with the ratio 9:1:1.25 showed the highest results: iodine adsorption activity was 41.91 %, total pore volume was 0.939 cm^3/g, bulk density was 204.06 g/dm^3 and methylene blue adsorption activity was equal to 240.11 mg/g.

According to Table 2, briquetted activated carbon was obtained by using rice husk, oil sludge and polyvinyl acetate in the ratio 10:1, 9:1:1.1, 9:1:1.25, 9:1:2. Activated carbon obtained with the ratio 9:1:1.1 showed the highest results: iodine adsorption activity was 44.45 %, total pore volume was 0.519 cm^3/g, bulk density was 357.99 g/dm^3 and methylene blue adsorption activity was equal to 245.57 mg/g.
The specific surface area of granular activated carbon was studied on a Sorbtometer - M. Table 3 shows the result of the specific surface area of granular activated carbon.

Table 1. Characteristics of granular activated carbon obtained from rice straw and oil sludge using polyvinyl acetate as a binder.

Indicators	Experimental results			
	Rice straw: Polyvinyl acetate	Rice straw: Oil sludge: Polyvinyl acetate		
Ratio	10:1	9:1:1.1	9:1:1.25	9:1:2
Temperature of carbonization, °C	500			
Carbonizate yield, wt. %	73.99	74.16	71.4	70.1
Temperature of activation, °C	850			
Mass ratio of water and carbonizate	2:1			
Activated carbon yield, %	30.4	25.6	21.9	38.1
Iodine adsorption activity, %	40.64	39.37	41.91	38.1
The total volume of pores, cm^3/g	0.928	0.819	0.939	0.945
Mass fraction of moisture, %	0.2	0.2	0.8	0.1
Bulk density, g/dm^3	226.24	220.36	204.06	225.87
Methylene blue adsorption activity, mg/g	242.51	252.46	240.11	180.41

Table 2. Characteristics of granular activated carbon obtained from rice husk and oil sludge using polyvinyl acetate as a binder.

Indicators	Experimental results			
	Rice husk: Polyvinyl acetate	Rice husk: Oil sludge: Polyvinyl acetate		
Ratio	10:1	9:1:1.1	9:1:1.25	9:1:2
Temperature of carbonization, °C	500			
Carbonizate yield, wt. %	70.1	75.6	89.6	83.2
Temperature of activation, °C	850			
Mass ratio of water and carbonizate	2:1			
Activated carbon yield, wt. %	26.2	27.8	31.1	21.5
Iodine adsorption activity, %	38.1	44.45	39.37	36.83
The total volume of pores, cm^3/g	0.511	0.519	0.519	0.629
Mass fraction of moisture,%	1.8	1.3	2	1
Bulk density, g/dm^3	386.51	372.49	357.99	487.38
Methylene blue adsorption activity, mg/g	228.75	254.57	245	171.21

Table 3 - Specific surface of samples

№	Name	Specific surface, m^2/g	Average pore size, nm	Specific pore volume, cm^3/g
1	Rice straw: Polyvinyl acetate	117,66	1,71	0,050
1	Rice husk: Polyvinyl acetate	178,67	1,71	0,077

Fig.1. Granular activated carbon obtained from rice husk, oil sludge and polyvinyl acetate in a mass ratio of 9:1:1.1.

Discussion.

The comprehensive characterization of the produced granular activated carbon using various analytical techniques in this study yielded valuable insights into its properties and potential applications. The FT-IR analysis revealed the presence of functional groups such as carboxylic, hydroxyl, and aromatic groups, which are known to enhance the adsorption capacity of activated carbon materials. The TGA results indicated that the granular activated carbon exhibited high thermal stability, a crucial factor for its suitability in high-temperature applications, ensuring its durability and performance under harsh conditions. The SEM analysis provided visual evidence of the material's porous structure and high surface area, confirming its potential for efficient pollutant adsorption, making it a promising candidate for water and air purification processes. Notably, the granular activated carbon produced in this study demonstrated comparable or even superior properties when compared to existing literature data on activated carbon derived from rice husk and straw. The enhanced iodine adsorption activity further supports the material's high adsorption capacity and underscores its utility in addressing environmental pollution challenges. The innovative use of polyvinyl acetate as a binder and oil sludge as a precursor in the preparation of granular activated carbon represents a novel approach that has not been extensively explored in the literature. This novel method opens up new possibilities for the utilization of agricultural waste and oil sludge, contributing to the sustainable management of these abundant waste resources while concurrently yielding high-quality activated carbon materials. Overall, the findings of this study contribute significantly to the advancement of knowledge regarding the valorization of agro-industrial waste and oil sludge for the production of efficient and eco-friendly activated carbon materials. The outcomes hold great promise for developing environmentally friendly and cost-effective solutions for waste management and pollution control, benefiting both the industrial sector and the environment. Future research can build upon these results to further optimize the production process and explore additional applications of the produced granular activated carbon in various environmental and industrial settings.

5. Conclusion.

In conclusion, this study holds significant implications for sustainable waste management and economic development in the agro-industrial sector. The findings pave the way for the development of innovative methods to harness solid waste from this complex, unlocking its potential for value-added applications. The utilization of polyvinyl acetate binder in the treatment of rice residue and oil sludge showcases a promising and optimal alternative for the preparation of granular activated carbon, which can further enhance the efficiency and cost-effectiveness of adsorption processes. These insights contribute to the growing body of knowledge on waste-to-resource approaches and offer actionable solutions for mitigating environmental impacts while promoting regional growth. Moving forward, this research opens avenues for further exploration and implementation of sustainable practices in the agro-industrial domain.

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