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Analysis of Methods for Activating Carbon-Containing Materials to Obtain Carbon Adsorbents

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ABSTRACT: This paper presents the specific principles of physical activation of various coals, chemical activation using acids, alkalis and salts. The raw materials for steam-gas activation are usually charred natural materials in the form of charcoal, peat coke, materials such as coal or brown coke, products of pyrolysis of coconut shells. The ability of such materials to activate depends on the proportion of volatile components in them. Its low values make activation difficult or practically exclude its very possibility. The reactivity of the named raw material significantly depends on the presence of macropores in it. So, for example, olive seeds - waste products of olive oil production are treated with 10% sulfuric acid and water, then subjected to pyrolysis at about 830°C, obtaining a product with a value of about 500 m^2/g, which after activation of the carbonizate increases to almost 1500 m^2/g. The oxygen content in such coals is 3-5%. The properties of activated carbons during their preparation are regulated by the choice of raw materials and activation method, as well as by changing the duration and conditions of activation, and some properties of the target products may depend on a number of parameters. The method of preparation of activated carbon includes: 1) pretreatment of plant materials in water tanks communicating with air for 24 days and subsequent air drying for two months at 25-30ºC until cracks appear on the surface of the materials; 2) carbonation of dried raw materials at 300-350ºC in a nitrogen stream (12 nl/h per 1.5 kg of raw materials); 3) activation of primary coal (carbonizate) in a rotating tubular reactor at 750-850ºC for 3-4 hours with a mixture of water vapor (2.5-4.5 nl/h) and nitrogen (12 nl /h) per 0.5 kg of primary coal. The characterized method is free from the use of synthetic and mineral additives and binding materials. It allows the production of high-quality (highly active and durable) active carbons for use as sorbents for purification of drinking and process water from trace elements, extraction from solutions of rare and precious metals, removal of toxins from blood plasma, as well as carriers of salt catalysts for the synthesis of vinyl chloride and vinyl acetate.

KEYWORDS: adsorbent, thermal activation, adsorption, charcoal, peat, peat coke, some hard and brown coals, semi-coke of brown coals, lignite coals, supermikropor, chemical adsorption, physical adsorption.

I. INTRODUCTION

Active carbons are a kind of microcrystalline carbon. Microcrystallites, which are the main structural element, are mainly characterized by alternating condensed aromatic rings. The three valence electrons of each carbon atom of this system are connected to neighboring atoms by strong, completely unsaturated σ-bonds, and the fourth π-electron is delocalized and is able to move freely through the system of conjugated bonds [1-3].

Unlike naturally oriented graphite structures, where carbon atoms are in the sp^2-hybrid state, and the interplane distance d_{002} is 0.3354 nm, there is no strict three-dimensional ordering of elementary hexagonal mesh layers in active carbons.

Active coals are characterized by a randomly layered (turbostratic) structure. The dimensions of graphite-like aggregates - microcrystallites of activated carbons determine the conditions for their production: the length is 2.0-2.5 nm, the height is 1.0-1.3 nm. A single microcrystallite can include up to four parallel planes (layers) randomly displaced relative to each other, unlike graphite, and the distance between the layers is not constant and is in the range of 0.34-0.37 nm. Another component of active carbons is represented by amorphous carbon [2,3].

The use of active coals is steadily increasing with the development of their industrial production, which dates back to the beginning of the last century.

Today, activated carbon is used in a large number of chemical processes. A huge number of water treatment processes, waste gas and wastewater treatment are based on the adsorption of pollutants with activated carbon. Modern means of individual and collective respiratory protection cannot do without active coals [2,4,5].

The uniqueness of activated carbon lies in the fact that it is the only industrial sorbent with a non-polar (electroneutral) surface. Its saturation with moisture is an extremely slow process: equilibrium is established within a few months. As a result, in most real technological processes, the humidity of the medium practically does not affect the efficiency of extraction of impurities from the gas or liquid medium by activated carbon [6].

Along with this, it should be noted the negative feature of activated carbon as an adsorbent – its combustibility. In the atmosphere of air, the oxidation of coal begins at temperatures above 250°C, although fires at carbon adsorption plants occur at lower temperatures.

II. SIGNIFICANCE OF THE SYSTEM

The raw materials for the production of activated carbons are a variety of widely available carbon-containing materials (including industrial waste) in non-carbonized form or in the form of coals and cokes. The most important raw materials are wood (in the form of sawdust), charcoal, peat, peat coke, some hard and brown coals, semi-coke of brown coals, lignite coals and petrochemical products [7]**.**

For anti–gas equipment and solving a number of special tasks, for example, the gold mining industry requires granular active coals with increased strength and a large volume of thin pores, which in world practice are obtained from coconut shells, shells of various types of nuts, fruit seeds, asphalt, metal carbides, soot, carbon-containing waste of various kinds - garbage, sewage sludge, fly ash, worn rubber tires, waste from the production of polyvinyl chloride and other synthetic polymers (for example , phenolic resins). These materials have not yet found wide application in the industrial production of activated carbon [7]. The study of methodology is explained in section III, section IV covers the experimental results of the study, and section V discusses the future study and conclusion.

III. METHODOLOGY

The basic principle of activation is that the carbon-containing material is subjected to selective heat treatment under certain conditions, leading to the formation of numerous pores, cracks and cracks, accompanied by an increase in the pore surface per unit mass of the target product. Chemical and steam-gas activation methods are used in the technique [8].

As a raw material for chemical activation, such non-carbonized products are usually used, such as peat or sawdust, a mixture of which with inorganic activating agents is subjected to high-temperature treatment. The activating agents are dehydrating and other substances in the form of acids, alkalis and salts [2,4,5,8].

The raw materials for steam-gas activation are usually charred natural materials in the form of charcoal, peat coke, materials such as coal or brown coke, products of pyrolysis of coconut shells. The ability of such materials to activate depends on the proportion of volatile components in them. Its low values make activation difficult or practically exclude its very possibility. The reactivity of the named raw material significantly depends on the presence of macropores in it [2].

Gas activation is usually carried out with oxygen (air), water vapor and carbon dioxide. Activation of the first of these agents is selective in nature and is associated with the danger of external over burning of carbonizate grains, and therefore preference is given to water vapor and carbon dioxide [2,4,5]. The technically acceptable reaction rate when using these gases is provided by temperatures of the order of 800-1000°C.

At the carbonation stage, the framework of the future activated carbon is formed – primary porosity and strength [3]. As a result of thermochemical activation, coarse-porous activated carbon is formed, which is usually used for discoloration of liquid process media and streams. As a result of steam activation, finely porous activated carbon is formed, which is used for fine (deep) cleaning [3]. Furnaces of various types and designs are used for the production of activated carbon. The most common are multipole, shaft, horizontal and vertical rotary kilns, as well as fluidized bed reactors [3]. Rotary kilns are universal aggregates for pyrolysis and activation processes, therefore they are used most often.

The determining influence on the pore structure of activated carbons is exerted by the raw materials from which they are obtained. Coconut shell-based activated carbons are characterized by a greater proportion of micropores, and

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activated carbons based on coal are characterized by a greater proportion of mesopores. A large proportion of macropores is characteristic of wood-based activated carbons [9].

As a rule, all kinds of pores are present in the active carbon, and the differential curve of their volumetric size distribution has 2-3 maxima. Depending on the degree of development of supermicropores, active coals with narrow (these pores are practically absent) are distinguished and a wide (they are significantly developed) distribution [3].

Van der Waals forces act on the walls of the pores of activated carbon, causing intermolecular attraction. By their nature, they are akin to the force of gravity, with the only difference that they act on a molecular, and not on an astronomical level. It is these forces that keep molecules of organic pollutants in water or gas streams on the surface of activated carbon. Such absorption, which is not complicated by the chemical interaction between the extracted substance and the adsorbent, is called physical adsorption. Chemical interactions may also occur between the adsorbed substances and the surface of the activated carbon. Such processes are called chemical adsorption or chemisorption. They are widely used in industry to solve problems of gas purification, degassing, separation of metals in solutions. During chemisorption, the adsorbed substance changes its chemical properties. For this reason, chemisorption is irreversible [3].

Chemically bound oxygen in activated carbons is part of the surface compounds of a basic or acidic nature, which significantly affect the adsorption properties of these absorbers [3,10].

Sufficiently large-tonnage plant waste in the form of nut shells (coconut, walnut, cedar, forest, etc.), seeds of fruits and fruits (apricot, peach, grapes, olives, etc.), as well as seed husks, bonfires and stems of some, mainly cultivated, plants are excellent raw materials for producing very good quality active coals [7,11,12,15].

So, for example, olive seeds - waste products of olive oil production are treated with 10% sulfuric acid and water, then subjected to pyrolysis at about 830 ° C, obtaining a product with a value of about 500 m^2/g, which after activation of the carbonizate increases to almost 1500 m^2/g. The oxygen content in such coals is 3-5%.

The properties of activated carbons during their preparation are regulated by the choice of raw materials and activation method, as well as by changing the duration and conditions of activation, and some properties of the target products may depend on a number of parameters [4].

Fragments of hollow bamboo trunks at least 5 years old in China have been used since the XV century for the production of crushed activated carbons with a transient porosity and a value of about 600 m^2/g, subjecting them to pyrolysis at temperatures of 800-1200ºC. Carbon fibers with unique properties are produced from threads obtained by spinning bamboo microfibrils by carbonation at 1200ºC. Bamboo carbon fibers are durable, have high absorption rates (in particular, with respect to odors and harmful substances) and excellent gas permeability. They are hypoallergenic, resistant to washing, have antibacterial and antifungal properties [16].

Crushed to fragments of 2.0–8.0 mm in size and dried to a constant mass at 105°C, the shell of pine nuts was subjected to two variants of pyrolysis and activation by excess water vapor in a tubular reactor placed in an electric furnace. In the first variant, steam was supplied at the beginning of the pyrolysis process to remove air from the reactor and at the end to activate the resulting coal. Activation was carried out for 1-3 hours at temperatures of 600, 650 and 700°C. In the second variant, steam was supplied continuously to combine the pyrolysis and activation processes. Pyrolysis was carried out until the release of liquid and gaseous products ceased. The volume of the latter served as a criterion for the completeness of the process. The target product was cooled to 40°C in the reactor before unloading. The quality of the resulting activated carbon was evaluated by its total porosity and adsorption activity by methylene blue.

The results of the study of a variant of the classical technology indicate that with an increase in temperature and the duration of the activation process, the quality of the target product generally increases, but its yield decreases. In the area where the value of adsorption activity reaches the corresponding GOST value (225 mg / g), the yield is 20-23%, which the authors hypothetically associate with the burning of raw materials during pyrolysis and the destruction of its pores due to the oxygen of the air in the pores of the fresh shell. The negative influence of this factor can be suppressed, according to the authors, by continuous steam supply during the combined pyrolysis and activation process. The experimental results presented in the paper indicate that under comparable heat treatment conditions, in particular at an activation temperature of 650°C and a process duration of 1-2 hours, there is an increase in the yield (by 4-6%) and the adsorption activity of the product, compared with the variant of the classical technology. A greater increase in the temperature or duration of the activation process in the characterized mode gives a slight increase in product performance, but leads to an increase in energy consumption and steam consumption. With a decrease in the temperature and duration of the activation process, the quality of the resulting product does not meet the requirements of GOST.

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IV. EXPERIMENTAL RESULTS

The source [14, 17] indicates that the activated carbon obtained from the cedar nut shell by traditional pyrolysis technology and subsequent activation by water vapor is characterized by a specific surface area of 870 m^2/g.

A peculiar technology of processing the same raw materials into active coals by chemical activation is characterized in [18,19]. In accordance with it, the raw materials were impregnated with a solution of phosphoric acid, ensuring its mass content in the impregnated material of 8% in terms of an absolutely dry shell, and dried. Then, in an experimental vertical batch reactor and an original design, the resulting impregnate was subjected to carbonation combined with air activation. The process was initiated by igniting a stationary layer of fragments of material loaded into the reactor with an open flame from below when air was injected into it. After that, it proceeded spontaneously for 15-20 minutes. when moving from the bottom up the thermal oxidation front created by the air blast. Upon cooling of the obtained material, it was washed with distilled water from phosphoric acid, assessing the completeness of washing using molybdenum-vanadium reagent.

The results of the comparison carried out in [18] on a number of technical characteristics of activated carbon (SCOAU) obtained from the shell of pine nuts with activated carbon of industrial production of the BAU brand allowed us to state that in terms of mechanical strength and brightening ability for methylene blue, SCOAU coal surpasses BAU coal by 12 and 33%, respectively, determining the prospects of its use for water purification from organic pollutants. The original method of obtaining activated carbon from the shell and meal – waste from the processing of pine nuts is described in RF Patent No. 2154603 [20]. The authors of this patent point to the method described in [21] for obtaining activated carbon from the shell of fruit seeds by grinding the seeds with a moisture content of 12-20% of apricots, plums, peaches to a particle size of 1.0-1.5 mm and processing in a sealed reactor at 350-400ºC and a pressure of 15-22 MPa, followed by processing the pyrolyzed mass with a vapor-gas mixture. After pressure relief and cooling, the discharged activated carbon is subjected to rolling and packing. The degree of coal burning according to the proposed technology is 60-75%. The authors of the patent [20] consider the need for deep preliminary grinding of raw materials and high pressure in the reactor to be disadvantages of this method. Along with this, the same authors note a method known according to [22] for producing activated carbon from carbon-containing waste from processing plant raw materials - grape meal and grape oil production coabstock, including mixing the waste with a binder, granulating the mixture, carbonizing the granules and activating them with water vapor at a temperature of 830-870ºC. Its disadvantage is the complexity of the technological process, due to the need for pre-processing of raw materials associated with the use of binder and granulation.

The proposed invention [20] provides an opportunity to solve the problem of creating a new resource-saving, waste-free technology, expanding the raw material base for the production of a scarce product - activated carbon with high sorption characteristics. The technical result is achieved by the fact that in a known method for producing activated carbon, including heat treatment of raw materials, activation, according to the invention, shell and meal – waste from processing pine nuts (Pinuc sibirica Du Tour nut) are used as raw materials, heat treatment is carried out at a temperature of 750-850°C with a heating intensity of 9-14°C / min, activation at the final temperature is carried out by water vapor with a mass ratio of coal and steam equal to 1: (2-6).

Activation by water vapor of the resulting coal is not accompanied by the formation of harmful emissions. The resulting pyrolysis gases (methane, ethylene, hydrogen) are environmentally friendly fuels. The proposed technology eliminates the formation of solid, liquid or gaseous waste.

Activated carbon from the shell and meal from the processing of pine nuts is black scales, without mechanical inclusions. It is characterized by the activity of methylene blue 270 - 310 (coal from the shell) and 110-140 mg/g (coal from the meal), as well as the absorption capacity of iodine 90-100 and 70-80% (for coal from the shell and meal, respectively).

Pine nut shells can also be a source for the production of bifunctional sorbents capable of simultaneously absorbing nonpolar substances and heavy metal ions by van der Waals and ion exchange interaction mechanisms, respectively [23,24].

An interesting method of preparing activated charcoal from vegetable raw materials (fruit and berry bones, coconut shells, sugar cane stalks, birch and pine wood, fossil coals) is described in [25,26]. The method makes it possible to obtain highly active, durable coal with a given set of properties, eliminate the need for the introduction of special additives and increase the adsorption capacity and porosity of coal due to a significant increase in the volume of mesopores.

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Referring to Japanese patent No. 1317113 dated 12/21/1989, which protects the method of preparing activated carbon by oxidation of raw granular coal in a reactor at 160-320ºC with a gas containing 2-10% O_2, carburization at 600ºC and activation of the resulting carbonizate at 900-950ºC with a mixture of CO_2 and H2O, the authors point to the disadvantages of this method due to the complexity of the technology of using mixtures of CO_2 and H_2O at high temperatures and the design of equipment in conditions of corrosion of reactor walls.

RF Patent No. 2064429 provides a method for obtaining activated carbon from fruit seeds and / or nut shells, including their treatment with 2-5 normal alkali solution for 2-8 hours at 95-100ºC, then with HCl solution (3-4 n) and concentrated phosphoric acid at the same temperature. The disadvantages of the method are: 1) reduced absorption capacity of activated carbon in organic matter vapors due to the destruction of 60-70% of the frame of the raw material already during carbonization and even more so when activated at 700-900ºC, which causes a low yield of the target product (no more than 5-7%) and its low abrasion strength (no more than 9-10%); 2) the complexity of processing and its high energy consumption; 3) thermal decomposition of high-molecular poly alcohols, aggravated by residues of metal hydroxides and acids; 4) a narrow spectrum of action of the resulting carbon adsorbents.

According to [25], the method of preparation of activated carbon includes: 1) pretreatment of plant materials in water tanks communicating with air for 24 days and subsequent air drying for two months at 25-30ºC until cracks appear on the surface of the materials; 2) carbonation of dried raw materials at 300-350ºC in a nitrogen stream (12 nl/h per 1.5 kg of raw materials); 3) activation of primary coal (carbonizate) in a rotating tubular reactor at 750-850ºC for 3-4 hours with a mixture of water vapor (2.5-4.5 nl/h) and nitrogen (12 nl /h) per 0.5 kg of primary coal. The characterized method is free from the use of synthetic and mineral additives and binding materials. It allows the production of high-quality (highly active and durable) active carbons for use as sorbents for purification of drinking and process water from trace elements, extraction from solutions of rare and precious metals, removal of toxins from blood plasma, as well as carriers of salt catalysts for the synthesis of vinyl chloride and vinyl acetate.

The best coals for sorption of metal ions and use as catalyst carriers were obtained by the authors by carbonization of raw materials in the temperature range of 300-350ºC and activation of carbonizates at 750ºC at a water vapor and nitrogen consumption of 3.0-3.5 and 12 ml/h, respectively.

V. CONCLUSION AND FUTURE WORK

The source [13,27] contains information about the developed automatic line LU-AU-750, which provides the possibility of producing high-quality activated carbon. The technology implemented on this line provides for the crushing of the shell of the bones and the classification of the crushed product with the separation of fragments of the required size, forming a homogeneous raw material mass. This mass is transported to the drying unit through a magnetic separator to separate metal chips - the product of wear of the working bodies of the crushing mechanism. In the drying unit with hot air, the humidity of raw materials is reduced from 40 to 4-5%. Dry raw materials are transported to the loading zone of carbonation furnaces, where they are placed on special pallets-baking trays with a solid layer of a certain thickness on tables made by the coal producer from improvised materials and loaded on these pallets into the carbonation furnace. In this furnace, pyrolysis of raw materials is carried out under specified conditions. Upon completion of pyrolysis, baking trays with partially sintered carbonized fragments are removed from the furnace and their contents are unloaded. The resulting carbonizate is transported to a crusher, and a fraction of the required size is isolated from the crushing product on a vibrating screen, returning larger fragments for repeated crushing. The conditioned fraction of carbonizate is transported to the tables of its packing described above on pallets-baking trays, on which it is placed in the activation furnace. Activation is carried out by water vapor under the conditions provided by the technology. Upon its completion, the baking trays are removed from the oven and emptied by means of a special lift. When sintering fragments of the resulting activated carbon caused by a violation of the activation mode, it is subjected to grinding, the product of which is packed into paper bags with a capacity of 0.5-10 kg, sewn up and sent to the warehouse.

In the sources [13,27] on the LU-AU-750 line, there is also technical plan information, including information about the operating mode, productivity, energy and water consumption, occupied areas, equipment, necessary maintenance personnel, consumption coefficients, product standards, installation conditions, etc.

Thus, there are numerous methods and technologies for the activation of carbon-containing raw materials that contribute to the production of carbon adsorbents. These activation methods differ in the stages and conditions of the process, which primarily depends on the requirements for coal adsorbents.

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