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DEVELOPMENT OF TECHNOLOGY FOR MAKING NANO SORBENT FROM WOOD PROCESSING WASTE

Abstract. 100–120 g of the test drug is placed on a smooth, clean white surface and examined by gently stirring in natural light. 20–30 g of the test drug is poured into a clean porcelain container and the odor is determined organoleptically. If it is necessary to enhance the odor, the porcelain dish is covered with a bottle, along with the medicine inside, and is placed in a water bath heated to boiling and heated for 5–7 minutes, after which the odor of the drug under test was detected again. ***SEM*** of ***Phenom G2 pro*** model was used to study the surface structure of the objects studied in the study, this allows images up to 25 nm in magnification in the range of 80 x to 45000 x. Grinding rate and granule size were evaluated and controlled using ***ANALYSETTE22 MicroTec plus*** laser diffraction particle size analyzer. This analyzer allows the determination of granulometric (mechanical) content in the range from 80 nm to 2000 μm. A ***SARTORIUS MA 150*** moisture meter was used to determine the moisture content of the samples studied in the study. The samples were dried to a constant mass at a temperature of 103 ± 2 °C.

Keywords: hydrolytic lignin, porosity, analyzer, filtrate, centrifuge, mycotoxin.

Introduction

To date, machinery and technology, including pharmaceuticals, petroleum, cosmetology, oil and gas refining industries, as well as high selectivity in various sectors of the economy, the demand for effective environmentally safe adsorbents is increasing [1–5]. Preparation of adsorbents that meet such requirements, the study of colloidal chemical properties of adsorbents. The study of the mechanism of adsorption processes in them poses new challenges to new scientific approaches, as well as to scientists and researchers in the field.

The influence of various factors on the synthesis of nanocarbon from the walnut peel, apricot kernel, methane, natural gas, and propane-butane fractions was studied and the texture and sorption characteristics of the obtained nanocarbon were examined. In addition, the effect of various factors on the rate of formation of nanocarbon obtained from methane, natural gas, and propane-butane fractions was studied and optimal conditions for the process were proposed [6–10].

Currently, activated charcoal is used in the food industry, medicine, and other fields for the treatment of gases and effluents, and other water [11–12]. The use of charcoal, for example, to purify water, allows drinking water to meet basic requirements [13–16], and the use of them as electrodes of supercapaci-

tors allows the creation of inexpensive rechargeable electrochemical devices with high power and energy properties [17–19]. Therefore, the study of the raw material base for obtaining activated charcoal and improving its physical and technical characteristics is of particular interest [20]. Its black ash, obtained by burning walnut shells in the absence of oxygen, has sorption activity [21].

Experimental part

100–120 g of the test drug is placed on a smooth, clean white surface and examined by gently stirring in natural light. 20–30 g of the test drug is poured into a clean porcelain container and the odor is determined organoleptically. If it is necessary to enhance the odor, the porcelain dish is covered with a bottle, along with the medicine inside, and is placed in a water bath heated to boiling and heated for 5–7 minutes, after which the odor of the drug under test was detected again. ***SEM*** of ***Phenom G2 pro*** model was used to study the surface structure of the objects studied in the study, this allows images up to 25 nm in magnification in the range of 80 x to 45000 x.

Methods for determining the granulometric content. Grinding rate and granule size were evaluated and controlled using ***ANALYSETTE 22 MicroTec plus*** laser diffraction particle size analyzer.

Figure 1. ANALYSETTE 22 MicroTec plus: a unit for determining the unit of measurement for dry and wet samples using a laser

This analyzer allows the determination of granulometric (mechanical) content in the range from 80 nm to 2000 μm. A dispersion block in a liquid medium is used to prepare a sample of hydrolytic lignin for analysis. In determining the granulometric composition of the finished product form of a complex nano sorbent (micro granules), the preparation of the sample is carried out in a dry dispersion block.

A ***SARTORIUS MA 150*** moisture meter was used to determine the moisture content of the samples studied in the study.

Figure 2. SARTORIUS MA 150: Moisture measuring device

Robust design, along with low space requirements and convenient operation, is key features of these analyzers. Fully automatic drying of the sample until a constant weight is achieved eliminates the need to program the endpoint removal parameter. It is possible to weigh from 1 mg to 150 g of product. The operating temperature is in the range of 40 °C to 220 °C at a temperature acceptable. The samples were dried to a constant mass at a temperature of 103 ± 2 °C.

Method for determining the ash content of raw materials. The porcelain crucible is fired in a muffle furnace to a constant mass, cooled in a desiccator, and weighed.

A portion of lignin weighing 2–3 g is weighed, the result of weighing is written with the accuracy of the third fraction in grams, and is placed in the crucible. The sample crucible is placed in a hood over an electric stove, burned without a flame, then baked in a muffle furnace at a temperature of 800–850 °C for 2–3 hours. After roasting, the crucible is cooled in the air for 5 minutes, then cooled in a desiccator, after which

it is pulled. Bake for another 30 minutes until the mass is less than 0.001 g. The mass fraction of ash (X_1) as a percentage is calculated according to formula 1:

$$X_1 = \frac{m_1}{m_1 \cdot \left(1 - \frac{W}{100}\right)} \cdot 100 \quad (1)$$

where, m is the mass before drying, g,

m_1 – mass after drying, g,

W – humidity of the sample under study,%.

The result of the analysis is taken as the arithmetic mean of two parallel determinations, the allowable differences between them should not exceed 0.01%. A test sample of 20 g of hydrolytic lignin is placed in a beaker with a capacity of 600 cm^3 and weighed to the second fraction. Add 100 cm^3 of hot distilled water, mix well with a glass rod and leave for 5–10 minutes. The precipitated solution is filtered under a vacuum in a porcelain funnel through a paper filter.

Another 100 cm^3 of hot distilled water is added to the sediment of the sample being tested in the beaker, mixed, and filtered under vacuum. This wash is repeated 3–4 times (according to the methyl orange indicator) until the water is neutral. The volume of washing water is transferred to a measuring tube with a volume of 500 cm^3, the volume is adjusted to the mark with distilled water and mixed. 25 cm^3 of the resulting solution is titrated with 0.1M sodium hydroxide solution in the presence of methyl orange indicator.

The mass fraction of acids (in terms of sulfuric acid) is calculated as a percentage according to formula 2.

$$X_2 = \frac{V \cdot K \cdot T \cdot V_2}{V_1 \cdot m \cdot \left(1 - \frac{W}{100}\right)} \cdot 100 \quad (2)$$

Here, V is the volume of 0.1M NaOH solution used for titration, cm^3

K – correction factor for the titer of 0.1 M NaOH solution

0.0049 – a mass of sulfuric acid per 1 cm^3 of solution with a net concentration of 0.1mol/dm^3, g

V_1 – the volume of washing water obtained for titration, cm^3

V_2 – total volume of washing water, cm^3

m – the mass of sample, g

W – humidity of the detected sample,%

The result of the analysis is taken as the arithmetic mean of two parallel determinations, the allowable differences between them should not exceed 0.01%.

Approximately 10 g of the analyzed lignin sample is weighed (by absolute dry matter) (the result of weighing in grams is written to the second decimal place), into a flask, add 100 cm^3 of water, and boil for 3 minutes. Stop the flask with the return current capacitor. The substances in the tube are then quickly filtered under vacuum through a paper filter, and the first parts of the filtrate are discarded. The filtrate is cooled and its pH is determined. A ***pH –150 MI*** millivoltmeter was used to determine the pH. The result of the analysis is taken as the arithmetic mean of the results of two parallel determinations, the absolute discrepancy between them does not exceed the allowable difference of 0.2.

A portion of the airdry lignin (approximately 2 g) is poured into a 250 ml conical flask and filled with 100 ml of distilled water. A return flow condenser is attached to the flask and placed in a boiling water bath and the water level in the bath should be slightly higher than the water level in the tube. Extraction is carried out for 3 hours. The lignin suspension is then filtered by vacuum in a porous glass filter dried to a constant mass and washes the lignin from the tube to the filter with hot water. The lignin filter is dried and weighed to a constant mass in an oven at 103 ± 2 °C at a constant weight. The mass fraction of water-soluble substances ($Э_{р.в.}$) and the fraction of absolute dry lignin are calculated according to formula 3.

$$Э_{р.в.} = \frac{m_2 - (m - m_1)}{m_2} \cdot 100 \quad (3)$$

Here, m is the filter mass with lignin residue, g

m_1 – empty filter mass, g

m_2 – absolute dry lignin sample mass, g.

The result of the analysis is taken as the arithmetic mean of two parallel determinations, the allowable differences between them should not exceed 0.02%.

A portion (2–3 g) of airdrying lignin is poured into a liner wrapped in filter paper. A lignin sleeve is placed in the nozzle for removal and the level of lignin in the sleeve should be 1 … 1.5 cm below the level of the siphon pipe. Pour 200 ml of solvent (alcohol: benzene mixture in a 1 : 2 ratio) into the flask. The instrument for extraction is collected and placed in a water bath. The bath temperature is set at 5–10 °C above the boiling point of the solvent. Water is fed to the refrigerator at a rate that ensures complete condensation of the solvent vapors.

Extraction is continued for 4–5 hours, after which the apparatus is removed from the bath, and the nozzle is separated from the tube and the refrigerator. The extract is poured into a dried flask to a constant mass (pour into a flask dried to constant weight) and the solvent is distilled in a water bath. The tube with cubic residues is dried in an oven at 103 ± 2 °C to a constant mass and weighed.

The mass fraction of soluble substances in organic solvents ($Э_{o.p.}$) is the proportion of absolutely dry lignin calculated by formula 4.

$$Э_{o.p.} = \frac{m - m_1}{m_2} \cdot 100 \qquad (4)$$

Where m is the mass of the flask together with the extract substance, g

m_1 – the mass of empty tube, g;

m_2 – the mass of absolutely dry lignin, g.

The result of the analysis is taken as the arithmetic mean of two parallel determinations, the allowable differences between them should not exceed 0.2%.

The device is assembled for testing, the general view of which is shown in Figure 13.

When a vacuum network is available, pump 1 is removed and the rest of the system is connected to the vacuum line. Clamp 3 serves to introduce air into the evacuation system and uses a front vacuum pump. It also serves to regulate the vacuum measured with a pressure gauge 4. When operating with a water flow pump, the vacuum is regulated by changing the water flow rate with a water tap. Tube 2 acts as a buffer tank. Valve 5 is used to shut off tube 7.

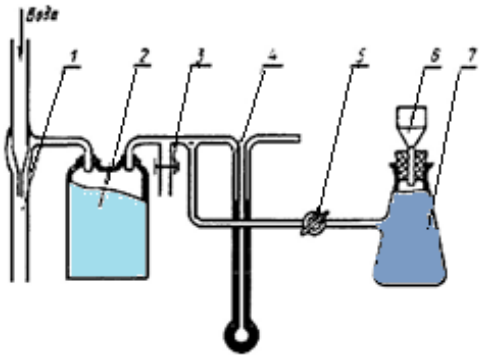

Figure 3. Device diagram for detecting open porosity: 1 – water flow pump, 2 – double-necked tube, 3 – clamp, 4 – pressure gauge, 5 – connecting tap, 6 – funnel, 7 – suction tube

The test sample is averaged and reduced to a volume of about 25 cm^3, is dried in a layer of not more than 5 mm for 1 h (105 ± 2) °C. 10 ± 0.1 cm^3 of the dried sample is poured into the measuring cylinder with a slight shaking, transferred to a pre-weighed weighing bottle, closed with a lid and weighed with an error of not more than 0.01 g. The sample is poured through a funnel into a conical flask,100 cm^3 of water is poured into it and its level is recorded.

The contents of the flask are boiled for (15 ± 1) minutes, after which cold distilled water is added to the initial volume and the outer surface of the tube is cooled to room temperature with tap water. A paper filter is placed on the bottom of funnel 6 and moistened with water. When valve 5 is closed, a vacuum (60 ± 5) mm of mercury is generated in the system, then a vacuum is created in the tube by turning the tap 5, after which the tube is turned off again. During assimilation of the filter, care must be taken to insure that it is firmly attached to the bottom of the funnel and that the connecting hoses are not twisted. The contents of the conical flask are poured into the suction funnel without loss and the sample is flattened with a spatula on the filter surface. Turn the tap to (60 ± 5) mmHg (mercury column) maintaining the vacuum, and assimilation begins. The stopwatch was started at the same time. After 3 minutes, the test sample is transferred to a weighing bottle before weighing. The rest of the sample

in the filter is removed with a spatula, transferred to the vial without loss, and closed with a lid. The wet sample weighing bottle is weighed with an error of not more than 0.01 g no later than 3 minutes after the end of suction. The tap is turned off, the filter is removed from the funnel, water is poured from the tube and the device is prepared for the next test. V_{Σ} – the total volume of pores (cm^3/g) is calculated using formula 5.

$$V_{\Sigma} = \frac{m_1 - m}{m \cdot \rho} \cdot 100 \qquad (5)$$

Here, m is the dry mass, g,

m_1 – wet mass, g;

ρ – water density, g/cm^3.

The density of water is calculated as 1 g/cm^3 for any room temperature up to 35 °C. The test result is taken as the arithmetic mean of two parallel determinations, the allowable differences between them should not exceed 4% of the smaller value.

The method of sorption-desorption of molecular nitrogen allows the dead analysis of micro-and meso cytes, their size distribution, and determination of surface area.

Figure 4. ASAP 2020 Micromeritics: a device for studying the nano-porous structure of hydrolytic lignin

To study the nano-porous structure of hydrolytic lignin, we analyzed the surface area using physical sorption methods **ASAP 2020 Micromeritics** (USA) and we used an automated system for to study of the porous structure of materials. This system allows to determination of the surface area, as well as a complete analysis of micro-and meso cytes, distributing their volume. Hole diameter measurements range from 0.35 to 100 nm.

Build a calibration graph. Comparative solutions are prepared to construct the calibration curve. To do this, 0.5 is added to 10 measuring tubes, each with a capacity of 50 cm^3; 1,0; 1,5; 2,0; 3,0; 4.0; 5,0; 6,0; 7,0; 8,0 cm^3 of indicator solution (1500 mg / dm^3) is then added to the volumes with water at the specified temperature (20 ± 2 °C). The resulting solutions contain 1 dm3, the density of the indicator is 15, respectively; to equal 30; 45; 60; 90; 120; 150; 180; 210; 240 mg/dm^3. The optical density of the reference solutions is measured on a blue light filter photo electro calorimeter with a wavelength (l) of 400 nm in cuvettes with a light-absorbing layer thickness of 10 mm. Distilled water is used as the element. Based on the obtained data, a calibration graph is constructed because the optical density of the solutions depends on the mass concentration of the reference solutions. ***Analysis.*** Approximately 0.1 g of the test drug, previously dried to a constant mass, is weighed (the weighing result is recorded

to the nearest third decimal place). The weighed part of the drug is poured into a 50 cm^3 conical flask, 25 cm^3 of indicator solution is added, is closed with a stopper, and shaken in a device to shake the liquid in the container for 20 minutes. After shaking, the suspension is transferred to centrifuge tubes and centrifuged for 15 minutes, 5 cm from the clarified solution is carefully pipetted and its optical density is determined. If the optical density of the clarified solution exceeds 0.8 optical units, it is transferred to measuring tubes of 25 or 50 cm^3, depending on the optical density of 5 cm^3 of the solution. The solution in the flask was diluted with distilled water to the specified mark. After dilution, the optical density of the solution should be 0.1 to 0.8 optical units. In this case, the dilution coefficient is 5 or 10.

Based on the obtained value of the optical density, the residual mass concentration of the indicator in the detected solution is determined using the calibration curve. ***Processing of results.*** The adsorption activity on the indicator (X_3) is calculated using formula 6 in milligrams per 1 g of product.

$$X_3 = \frac{(C_1 - C_2 \cdot K) \cdot 0.025}{m} \qquad (6)$$

Here, C_1 – mass concentration of initial indicator solution, mg / dm^3;

C_2 – mass concentration of the solution after contact with the test sample, mg/dm^3.

K – the dilution coefficient of the solution obtained for analysis after contact with the sample;

m – the weight of test sample, g;

0.025 – the volume of indicator solution obtained for determination, dm^3

The result of the analysis is taken as the arithmetic mean of the results of two parallel determinations, the absolute discrepancy between them shall not exceed a difference of 10 mg per 1 g of the permissible test sample.

Preparation of a standard solution of mycotoxin at a concentration of 1000 μg/cm³. The standard 5.0 mcg dry mycotoxin by net weight should be dissolved in 5 ml of methanol.

Preparation of a working solution of mycotoxin. Approximately 10 ml of 0.1 M phosphate buffer solution pH = 6.5 is required to prepare the working solution of mycotoxin and a 50 μl standard solution of the selected toxin in methanol should be added at the adjusted concentration. A 50 cm^3 volumetric flask is labeled with 0.1 M phosphate buffer solution pH = 6.5 to calculate that the toxin concentration in the working solution is 1000 μg / dm^3, and mix well.

Analysis. 0.001 g of the weighed sample (clearly weighed) is poured into an Eppendorf-type solution with a volume of 2 cm^3. To the same solution is added 1 cm^3 of the working solution of the toxin. The sorption process is carried out for one hour at a temperature of 37 °C in a thermos shaker at a speed of 1400 rpm. The resulting suspension is centrifuged for 5 minutes at a speed of 10.000 rpm. The obtained supernatant and starting solution used "RIDASCREEN" test systems for in vitro analysis according to the method proposed by the manufacturer of the solution (R-Biopharm AG, Germany) case is analyzed for the number of mycotoxins in it by the method of competitive immuno enzyme analysis. For 1 mg of sorbent, the sorption capacity of the sorbent substance is calculated according to formula 7:

$$Sorption\,ability = \frac{C_{initial} - C_r}{m_н} \qquad (7)$$

Here, $C_{initial}$ – the composition of the toxin in the working solution, mkg;

C_r – the content of residual toxin in the working solution after the sorption process, mkg.

m_s – mass of sorbent sample, g

Сорбсия фоизи 8-формула билан ҳисобланади:

$$Sorption\,\% = \frac{C_{initial} - C_r}{C_{initial}} \cdot 100\% \qquad (8)$$

Here, $C_{initial}$ – the value of the toxin concentration in the working solution, mkg/cm^3.

C_r – the value of the residual toxin concentration in the working solution after the sorption process, μg/cm^3

The results of the analysis are the arithmetic mean of the three parallel determinations.

Preliminary studies of mechanic activation in wood processing wastes such as wood shaving, Cellulose lignin, and hydrolytic lignin have been conducted. Based on several characteristics obtained, hydrolytic lignin was selected as the main research object. Pre-ground and dried hydrolytic lignin is a free-flowing sawdust-like mass of brown color with a small addition of partially hydrolyzed sawdust.

The chemical analysis showed that "Seasoned" hydrolytic lignin does not contain free sulfuric acid, because of the high content of ash components (28.9%) and it is washed away for many years by atmospheric precipitation during storage in the open air.

Microphotographs clearly show the structural properties of hydrolytic lignin, which partially retains the anatomical structure of the original wood. An evolved system of capillary cavities of various sizes is observed, from the internal cavities of the cells to the pores in the walls, to the micro-cavities of the layered structure. A closer examination reveals the structure of the strongly lignified primary wall and the true median lamina protruding from it. Numerous layers of the secondary cell wall with highly developed interfibrillar porosity are also clearly distinguishable.

Conclusion

1. The use of mechanically activated hydrolytic lignin as the basis of a complex nano sorbent is most preferable.

2. Mechanical activation of hydrolytic lignin is purposefully carried out in one cycle, at a rotation speed of 5000 rpm.

3. All studied specimens are characterized by the absence of micropores, the significant size of meso cytes, and the significant outer surface.

4. Mechanically activated lignin can be the basis of a nanoporous sorbent and a carrier of other sorption systems.

5. Polysaccharide components of yeast biomass and natural aluminosilicates and others can be used as such sorption systems.

6. For various purposes, such complex sorbents can be used as entero sorbents for the purification of drinking and wastewater, the separation of heavy metal ions from multi-component solutions, and the removal of toxins from the bodies of animals and humans.

7. For each specific sorption system, the complex nano sorbent composition must be optimized to achieve maximum efficiency.

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