ISSN: 2350-0328
International Journal of AdvancedResearch in Science, Engineering and Technology
Vol. 11, Issue 6, June 2024

Nitrogen Vapor Adsorption on Plum Seed Bark and Activated Carbons

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ABSTRACT: The article analyzes the influence of temperature and water vapor on the sorption activity in the modification of the bark and activated carbon adsorbents of plum seeds grown in our republic. Activated carbons are widely used in various fields due to their properties such as activated surface, porosity, good conductivity, and stability in various environments. Research methods were carried out based on samples presented in the literature and GOST standard indicators. According to the results, the lowest value of the specific surface area of the structure of the porous material according to the BET theory was 2.0227 m2/g, corresponding to the skin of the plum kernel. Based on the analysis, it was proved that after carbonation at 500°C, the specific surface area increased to 79.1184 m2/g, and after activation with steam at 800°C, the value of the specific surface area increased to 517.5933 m2/g.

KEY WORDS: adsorption, desorption, nitrogen, isotherm, plum kernel peel, pyrolysis, steam activation, specific surface area...

I. INTRODUCTION

Activated carbon is a material with surface area, internal surface, certain pore size, chemical stability, and various functional groups on the surface. Due to its high adsorption properties, it is widely used as an adsorbent in many industrial processes, including gas separation and purification, waste water, juice and oil purification, and catalysis processes [1 - 3]. There is a lot of literature on the structure, properties, and activation conditions of dispersed and porous materials, including information on the processing of agricultural waste into activated carbon [4 - 7]. In the world, the share of non-renewable resources such as coal and oil is more than 85% of processed carbon materials. In addition, it has been proven by many studies that porous carbon materials can also be obtained based on plant and agricultural wastes [8]. Among them, active carbons obtained from the shells of cereal fruits have a special place [9 - 14]. There are many technologies for the production of carbon adsorbents. However, the methods of obtaining highly porous carbon adsorbents are not always universal. Many technologies for the production of adsorbents require the involvement of large raw materials and low-cost material resources. Therefore, it is of particular importance to improve the methods of obtaining carbon adsorbents from the waste of industrial enterprises and low-value agricultural products. Regarding this task, there are many methods of plant waste processing in world practice [15 - 17]. Among the promising methods of improving the porous structure of carbon adsorbents is the method of activation with steam. The purpose of this research work is to study the adsorption of nitrogen vapor on activated carbon materials using plum seed bark, carbonatite, and water vapor.

II. METHODS

The peel of a plum seed of a certain size was removed and carbon adsorbents were obtained by the method of thermal activation from 200 to 800°C in a pyrolysis device designed for laboratory conditions. The charring process was carried out for 2 hours in an inert environment - in the presence of argon gas. Carbons obtained at a temperature of 500°C were selected for activation with the help of steam. Temperature-dependent heating rate was changed: heating up to 200°C was 5°C/min, 200 - 700°C was 10°C/min, and subsequent heating was reduced to the previous rate. Activated with steam for 30 minutes at a pressure of 196,133 kPa when the temperature reached 800^0C. This leads to an increase in porosity in the adsorbent and an increase in its sorption properties. The moisture content of the samples was determined using MA 210. R equipment. Determination of ash content was carried out according to GOST 11022 - 95 [18 - 20]. SEM analysis (EVOMA 10 brand scanning electron microscope) was used to investigate their elemental composition. The functional groups in the samples were studied on the Perkin Elmer Spectrum IR apparatus. The characteristics of the porous structure (comparative surface area - S, pore volume - V, pore diameter - d) were determined by low-temperature nitrogen adsorption at 77.35 K in a static Quantachrome Nova 1000e adsorption unit. For this, the test samples were prepared in the form of vacuum treatment at a temperature of 100^0C for 12 hours. The curves of dependence of the amount of

ISSN: 2350-0328
International Journal of AdvancedResearch in Science, Engineering and Technology
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adsorption on the residual pressure were processed by the BET method. The t-plot method was used to determine the size of micropores. The average pore diameter was estimated by the BET method according to the formula Dmed=4V/S.

III. RESULTS AND DISCUSSIONS

Textural properties of original plum kernels, pyrolyzed carbonization at 500°C (OFUA – 500), and steam-activated samples at 800°C (OFUA – 500-800) were determined on a low-temperature nitrogen adsorption Quantachrome Nova 1000e at 77 K. The samples were brought to constant mass for 12 hours at a temperature of 100^0C before measurement. Nitrogen partial pressures were investigated in the range of 0.005 to 0.995 p/p^0 for adsorption and desorption curves. Micropores and average pore radii were determined using adsorption curves using the BET method, and mesopores were determined using the Beretta-Joyner-Halenda (BJH) method. The size and surface distribution of pores were determined by DFT (Density functional theory) and Barreta-Djoynera-Xalendy (BJH) method. The average pore diameter was determined using the BET formula Dsr=4V/S, V- in the volume of adsorbed nitrogen.

Depending on the appearance of the curves, the mechanism of adsorption on porous materials can be described. The isotherm belongs to type IV, indicating that it has a mesoporous structure. This type of isotherm is mainly suitable for materials subject to capillary condensation. The rise of the curves in the isotherm at high relative pressure indicates the presence of large macropores in the structure.

The hysteresis corresponds to type H3, which is a mesoporous material. Their pores are in the form of columnar or irregular channels. This type of hysteresis indicates that adsorption and desorption occur in pores of different shapes and sizes. In the initial part of the curve (p/p° at low relative pressure), a linear growth of nitrogen absorption was observed, which indicated the filling of the microporous structure. This part shows that it is sorbed into the micropore volume, that is, there are micropores in the structure.

The pore size and total volume of the porous material can be determined from the pore size distribution graph. From differential pore distribution curves–pore volume change depends on pore size change. The upper peaks show the common pore sizes in these curves. This peak is observed at a short band size, indicating that pores are predominant in this material. The red curve shows the distribution of total pores. The fact that this curve rises at a rate and then remains constant, most pores have a small entrance size, followed by the formation of new large pores does not affect the total pore size.

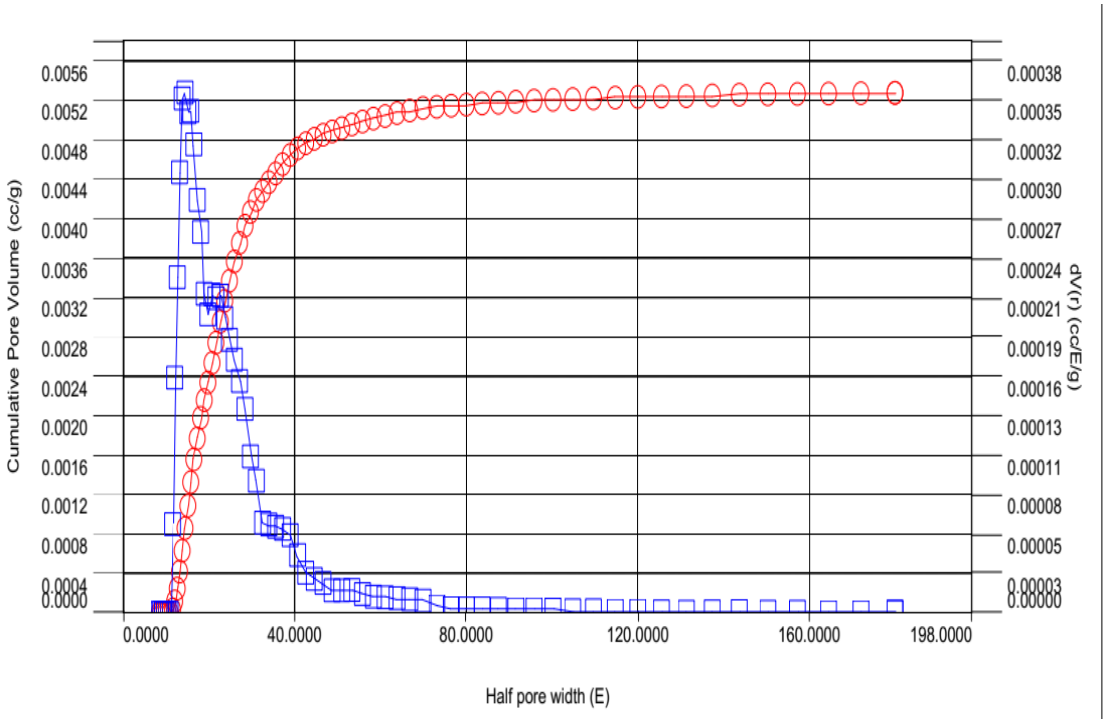

Figure 1. Pore size distribution by ODP sample volume

ISSN: 2350-0328
International Journal of AdvancedResearch in Science, Engineering and Technology
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As a result of the study of the pore size, it was found that the material has a large number of small and large-sized pores, and further increasing the pore size does not affect the total pore size. This indicates that it has two types, i.e. micro and meso-macropores. In the distribution of pores in such porous materials, mainly micropores occupy the main area on the surface, and meso- and macropores serve as transport channels.

The porous structure and surface characteristics of the material can be known from the texture parameters of the examined sample. The fact that it has a specific surface area of 11.67 m^2/g indicates the low porosity of the material, that is, the penetration of gases into the pores is limited. This shows that it is inferior in comparison to analogs of high-sorption material. The t-Plot Micropore Area reading is 8.736 m^2/g, indicating the dominance of micropores over other pores, i.e. their position on the total surface. The micropore volume is equal to -0.00425 cm^3/g, which indicates that the porous material has a low micropore content, that is, it is a high-density or low-porosity material. The indicated mean pore radius of 38.86 Å indicates the presence of mesopores in the porous material, or large-sized pores despite low overall porosity.

The hydraulic radius of the pores is 7.01, which makes it possible to study the adsorption activity as a transport channel in the effective absorption of molecules. Based on the obtained results, it was determined that the above sample has a low surface area, and the presence of a small amount of micropores, but their size consists of mesopores. Such materials can't adsorb molecules, but the presence of large pores allows large particles or molecules to enter. From the analysis results, it can be seen that the adsorption amount of ODP samples is very low.

It is possible to observe that the treatment of coals at 800ºC is due to the combustion of combustible substances and the formation of micropores, that is, the development of pores and the inner surface can be seen through isotherms. The results make it possible to use such adsorbents in monolayer adsorption processes, for example: gas and wastewater treatment.

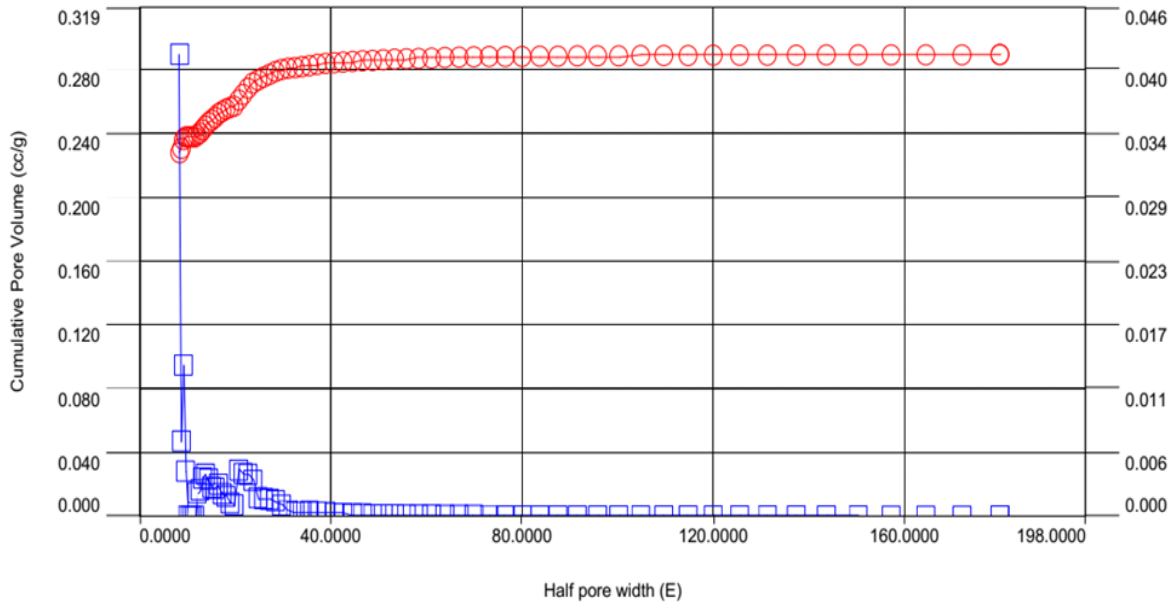

Figure 2. OFUA – 500-800 pore size distribution over sample size

From the pore size distribution graph, it can be observed that the second sample has increased large pores compared to the first sample. In the histogram, the peaks are approximately 20 Å apart but show the presence of large pores. From this histogram, it can be seen that the amount of mesopores increased as a result of high-temperature treatment. The total pore volume, especially the mesopores, is higher than the sample obtained at 500ºC.

Thus, thermal activation using water vapor at high temperatures has been found to have wider pores, i.e. increased mesopore size, compared to the sample thermally treated at lower temperatures. Steam treatment at 800ºC shows an

ISSN: 2350-0328
International Journal of AdvancedResearch in Science, Engineering and Technology
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increase in total pore size, i.e., an increase in active centers, compared to the results of heat treatment at 500ºC. The size of micropores was similar in both samples, but in the sample treated with steam at 800ºS, micropores decreased and mesopores increased

Table 1. Textural characteristics of the analyzed samples

Sample	Single point surface area, m^2/g	BET Surface Area, m^2/g	Micropore Area, m^2/g	External surface area, m^2/g	Micropore volume, cm^3/g	Adsorption average pore diameter, Å	Average pore hydraulic radius, Å
ODP	1,2329	2,0227	-	3,4344	-0,001038	94,225	15,8702
OFUA-500	83,5033	79,1184	62,4012	16,7172	0,034217	23,524	3,8591
OFUA – 500-800	542,8960	517,5933	353,5040	164,0892	0,195054	24,590	4,2023

In general, high-temperature steam treatment is widely used to improve the structure of porous material, as the above results prove. A change in such pores allows to expansion of the fields of application of the adsorbent material. The obtained results revealed that carbon materials have a complex porous structure, micropores, and a certain amount of mesopores.

IV. CONCLUSION

Thus, the physicochemical parameters of the activated high-carbon sorbent based on plum seed waste, their adsorption activity, the carbonization process, and the effect of temperature on the sorption process as a result of treatment with water vapor were studied. As a result of the SEM analysis of the obtained sorbent materials, the change of the carbon content by the activation temperature, as well as the morphology of the sorbent, was studied based on the elemental analysis. Based on IK analysis, analyses of functional groups formed during the activation of sorbents were studied. Also, adsorption-desorption isotherms of activated carbon sorbent materials were studied by the nitrogen adsorption method, and important parameters of their relative surface area and pore size were determined.

REFERENCES

1. Activated carbon. Chemical encyclopedia. M.: Publishing house "Soviet Encyclopedia", 1988, vol. 1, p. 77
2. Activated carbon [Electronic resource] Access mode: http://www.chemsystem.ru/aktivirovannyy-ugol/ (date of access: 04/17/17).
3. Mukhin V.M., Klushin V.N., Production and application of carbon adsorbents - Lambert Academic Publishing, 2018. - 350 rub.
4. A.P. Karnaukhov, “Adsorption. The texture of dispersed and porous materials.” The book is a winner of the competition of scientific publications of the Siberian Branch of the Russian Academy of Sciences. 1998.
5. Kozlov V.N., Pyrolysis of wood. - M.: Publishing House of the USSR Academy of Sciences. 1952. - 282 p.
6. Preparation of carbonized sorbents [Electronic resource], access mode: http://helpiks.org/3-96300/html (access date: 11/01/2017).
7. Carbonization of plant raw materials and study of the obtained materials by physical-chemical methods of analysis [Electronic resource] Access mode: http://helpiks.org/3-96312.html (access date: 11/01/2017).
8. Belyaev E.Yu., Production and use of wood activated carbons for environmental purposes. Chemistry of plant raw materials, 2000, No. 2, p. 5-15.
9. Our technologies. Sh. Technology for producing activated carbon from pine nut shells [Electronic resource] Access mode: http://xantib.narod.ru/catalog.htm. (date of access: 09/18/2017).
10. Active carbon from pine nut shells! [Electronic resource] Access mode: http://konspektiruem.ru/news/Aktivnyi-ugol-iz-skorlupy-kedrovogo-oreha/. (date of access: November 23, 2017).
11. Savelyeva Yu.R., Kryazhov A.N., Bogomolov M.S., et al., Preparation of active carbon from pine nut shells. Chemistry of plant raw materials, 2003, No. 4, p. 61-64.
12. Bagoev A.V., Lebedev I.A., Karchevsky D.F., et al., Preparation of active carbons from pine nut shells. Polzunovsky Bulletin, 2013, No. 1, p. 282-284.
13. Activated carbon: production from fruit seeds, sawdust, and agricultural waste. [Electronic resource] Access mode: http://www/asia-business.ru /torg/ mini factory/coal/activated/activated 1147. html. (date access: 11/27/2016).
14. Determination of physicochemical and adsorption characteristics of new activated carbon from apricot seeds [Electronic resource]. Access mode: http //7universum.com/ru/tech/archive/item/4373 (access date:
15. RF Patent No. 2111923 dated May 27, 1998 Method for producing activated carbon from fruit seeds and nut shells (Golubev V.P., Mukhin V.M., Tamamyan A.N., etc.) [Electronic resource]. Access mode: http://www.findpatent.ru /patent/211/2111923.html (access date: 12.12.2017).

ISSN: 2350-0328

International Journal of AdvancedResearch in Science, Engineering and Technology

Vol. 11, Issue 6, June 2024

16. RF Patent No. 2237013 Method of preparing activated carbon from plant raw materials [Electronic resource] Access mode: http://bankpatenttov.ru/node/96409. (access date: 12/12/18).
17. RF Patent No. 2154603 Method for producing active carbon (Poboronchuk T.N., Petrov V.S., Rubchevskaya L.P.). [Electronic resource] Access mode: http://www.findpatent.ru/patent/215/ 2154603 .html (date of access: 05/21/2017).
18. GOST 11022-95 Solid mineral fuel. Methods for determining ash content.
19. GOST 6217-74 Interstate standard crushed wood active coal technical specifications.
20. I.K.Gindulin, Yu.L.Yuryev., "Technical analysis of nanoporous materials" Yekaterinburg. 2011.
21. Salikxanova Dilnoza, Usmonova Zulfiya., Application of activated carbon from local plant waste – plums in wastewater treatment of industrial enterprises. International Journal of Current Science Research and Review ISSN: 2581-8341 Volume 06 Issue 12 December 2023 DOI: 10.47191/ijcsrr/V6-i12-62, Impact Factor: 6.789 IJCSRR @ 2023 Page No. 8079-8082
22. Yessinbek A., Satayev M., Azimov A., Activated carbon production from plum pit shells for oily wastewater treatment. Water Practice & Technology Vol 00 № 0,1 doi: 10,2166/wpt.2023.018;