

STUDYING NITROGEN ADSORPTION TO DETERMINE THE POROSITY OF THE SYNTHESIS SORBENT

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Abstract. The article studied the pores of dicyandiamide-formaldehyde resin based on dicyandiamide and formaldehyde. It was established that the study of the absorption of nitrogen vapor on samples of the DSDAF ion exchanger at a pressure of 0.950 P/P₀ was 12.4811 cm³/g.

Key words: dicyandiamide, formaldehyde, dicyandiamide-formaldehyde resin, adsorption and ion exchange resin.

Introduction.

Gas adsorption is of great importance for the characterization of a wide range of porous materials. Of all the numerous gases and vapors that are readily available and can be used as adsorbents, nitrogen still occupies a dominant position.

Thanks to user-friendly new equipment and fast data processing, nitrogen adsorption at 77 K can now be used for both routine quality control and research into new materials. Due to the importance of technology, it is interesting to trace its historical development.

The earliest studies of the adsorption of nitrogen and other gases at liquid air temperature (88 K) appear to have been carried out by Dewar and Ramsay [1] in their study of atmospheric composition and noble gas separation. This was followed by a number of other low-temperature studies of the adsorption of nitrogen and other gases by charcoal, including an extensive series of measurements over a wide temperature range by Ida Homfray [2].

Langmuir's monumental work on monolayer adsorption [3] led to renewed interest in the interpretation of adsorption data. According to the Langmuir model (now called "ideal localized monolayer adsorption"), the amount adsorbed on the plateau of a type I isotherm corresponds to complete coverage of the monolayer. In the early 1930s, it became clear that multilayer nitrogen adsorption could occur at liquid nitrogen temperature (77 K). The work of Benton and White led Brunauer and Emmett to use gas adsorption to determine surface area. They found that the adsorption isotherms of nitrogen and several other gases on the iron catalyst of synthetic ammonia had the same sigmoidal shape (later designated as type II). Empirical data showed that the transition from single-layer to multilayer adsorption occurred at the beginning of the middle,

almost linear portion of the isotherm (called point B). In 1938, the publication of the Brunauer-Emmett-Teller (BET) theory [4] appeared to provide theoretical support for this proposal, as the absorption at point B was found to agree well with the capacitance of a BET monolayer in nm.

To obtain the specific surface area a from nm, Brunauer, Emmett, and Teller followed the example of Emmett and Brunauer by assuming that the completed monolayer is in a close-packed state. Despite the perceived theoretical limitations of the BET model, the nitrogen BET method soon became a standard procedure for determining surface area.

The use of nitrogen adsorption for pore size analysis began in the late 1940s. It is based on the application of the Kelvin equation, corrected for the thickness of the multilayer layer on the pore walls. One of the first computational procedures was proposed by Shull, but the method developed by Barrett, Joyner and Halenda (BJH) in 1951 remains the most popular way of determining the pore size distribution based on the corresponding nitrogen isotherm.

2. Experimental part.

All materials used in the process of studying the pores of the sorbent, such as dicyandiamide-formaldehyde resin, were previously synthesized sorbents and were used in the sorbent for further processing.

Dicyandiamide-formaldehyde resin (DCDAF) is intended for the sorption of copper ions in wastewater, the sorbent isotherm was studied using a Quantachrome® ASiQwin™ - Automated Gas Sorption © 1994-2017, version 5.21 Quantachrome Instruments. The nitrogen vapor pressure isotherm of the sample was studied as follows. Sample weight: 0.0546 g. Approximate gas evolution time: 1.1 hours. Final outlet temperature: 120 °C, Analysis time: 2:27 h:min, Bath temperature: 77.35 K, Analysis mode: Standard, Cold zone V: 1.67808 cubic meters, Hot zone V: 16.5106 cubic meters, (d): 4.0000 mm. Liquid density: 0.808 g/cm³.

3. Results and Discussion.

As is known, the BET theory [4] is based on a simplified model of physisorption [3]. As in Langmuir's theory, the surface of the adsorbent is depicted as a set of equivalent areas on which molecules are randomly adsorbed. It is assumed that the probability of site occupancy is independent of the occupancy of neighboring sites and that there are no lateral interactions between adsorbed molecules (i.e., an ideal localized monolayer).

The molecules in the first layer act as sites for molecules in the second layer, and these in turn are sites for molecules in the third layer, and so on for molecules in higher layers. Although no lateral interactions are allowed, all layers above the first are assumed to have fluid properties. Given the artificial nature of the BET theory, it is not surprising that the range of applicability of the BET equation is always limited to the nitrogen isotherm portion. The best fit rarely exceeds P/P_0 0.30, and for some adsorbents (e.g. graphitized carbon) the upper limit is P/P_0 0.1. Obviously, the location and extent of the linear region of the BET graph depends on the adsorption system (both adsorbent and adsorption) and the operating temperature. In view of this situation, it is strongly recommended [5] that the BET monolayer capacitance, nm, be determined based on the best linear fit for the portion of the isotherm that includes point B.

In table 1, the adsorption of nitrogen vapor with the DCDAF ion exchanger was studied, at 0.05 P/P_0 (the ratio of the highest vapor pressure to the initial vapor pressure), it is clear that the main adsorption process will last up to 0.75 P/P_0 which absorbs 4.7677 cm³/g, and 0.805 P/P_0 is absorbed by 12.2354. The capillary condensation process occurs at 0.85188 P/P_0 , and at a vapor pressure of 0.851 P/P_0 , 11.4649 cm³/g of nitrogen vapor formed a bond with the ion exchanger,

the desorption process occurred at a given pressure of 0.996 P/P₀. Based on the data presented in Fig. 1 and Table 1, an isotherm of the sample was plotted and analyzed. The highest absorption of nitrogen vapor of the obtained sample of the DCDAF ion exchanger at a pressure of 0.950 P/P₀ was 12.4811 cm³/g and was presented in the form of a diagram [6].

Table 1

Results of nitrogen vapor adsorption of the obtained sample of the DCDAF ion exchanger.

Relative pressure P/P ₀	Absorption volume sm ³ /g	Relative pressure P/P ₀	Relative pressure P/P ₀	Relative pressure P/P ₀	Relative pressure P/P ₀
5.16478e-02	0.0559	5.16478e-01	3.0244	9.93056e-01	12.1916
1.00771e-01	0.3478	6.00763e-01	3.4596	9.00757e-01	11.0837
1.51069e-01	0.5983	6.50663e-01	3.9442	7.98805e-01	10.4927
2.01106e-01	0.8059	7.00630e-01	4.4696	6.99871e-01	9.3748
2.51021e-01	1.0952	7.50831e-01	4.7677	5.99827e-01	8.5594
3.01109e-01	1.3790	8.05174e-01	12.2354	4.99252e-01	7.6086
3.51236e-01	1.3953	8.51881e-01	11.4649	4.00053e-01	6.2274
4.00844e-01	1.9134	9.00427e-01	11.7694	2.98531e-01	4.5694
4.50844e-01	2.2838	9.50206e 01	12.4811	1.99330e-01	2.9332
5.00655e-01	2.8486	9.96583e-01	12.3381	9.82938e-02	1.2492

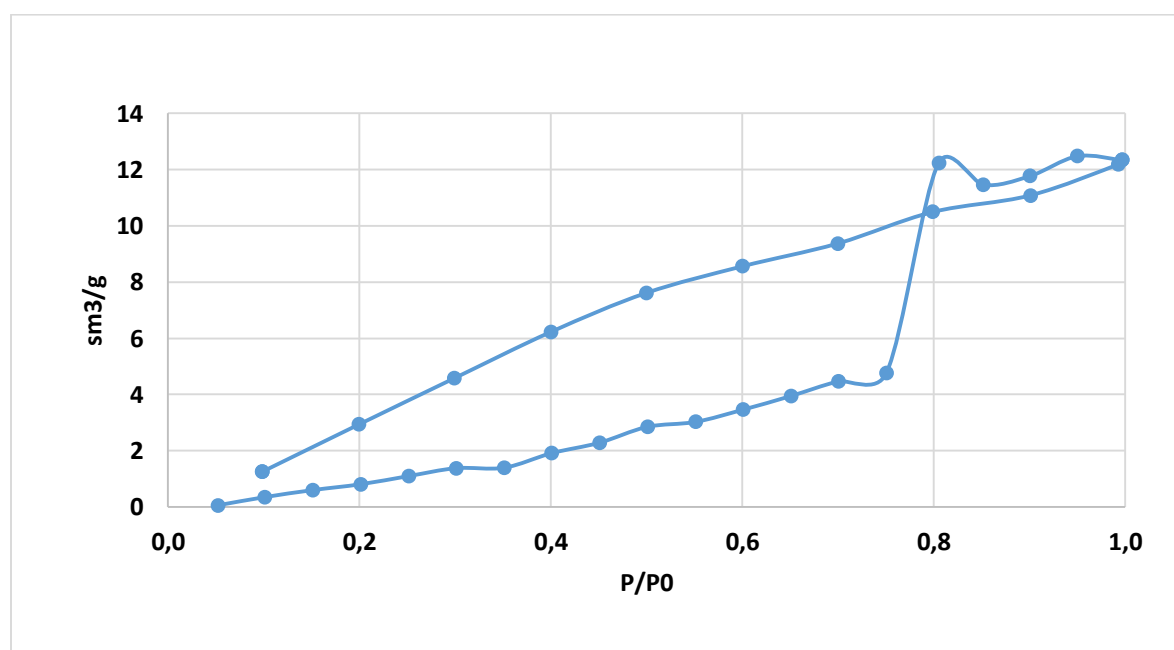


Fig.1. Isotherms of DCDAF resin.

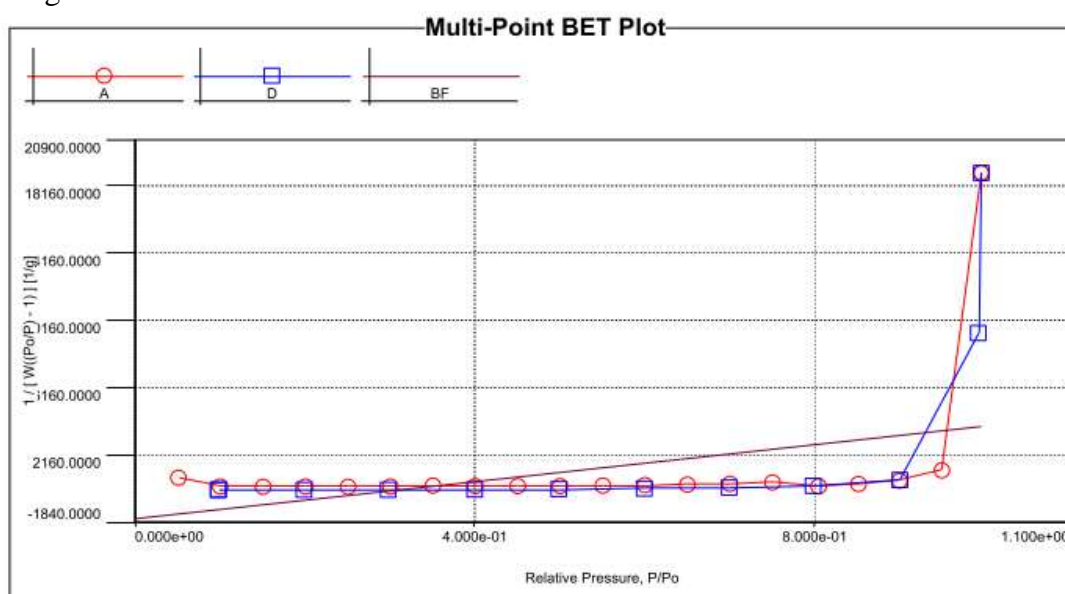
Table 2.

The actions performed by the multipoint BET method are presented.

№	Relative pressure P/P ₀	Absorption volume, cm ³ /g	1/[W((P/P ₀)-1)]
1	0,05	0.0559	778,9
2	0,1	0.3478	257,81
3	0,15	0.5983	237,97
4	0,2	0.8059	249,92

5	0,25	1.0952	244,84
6	0,3	1.3790	249,98
7	0,35	1.3953	310,45
8	0,4	1.9134	279,76
9	0,45	2.2838	287,62
10	0,5	2.8486	281,62
11	0,55	3.0244	325
12	0,6	3.4596	348,01
13	0,65	3.9442	377,83
14	0,7	4.4696	418,95
15	0,75	4.7677	505,69
16	0,8	12.2354	270,26
17	0,85	11.4649	401,37
18	0,9	11.7694	614,75
19	0,95	12.4811	1223,3
20	0,995	12.3381	18916
21	0,993	12.1916	9385,1
22	0,9	11.0837	655,2
23	0,988	10.4927	302,75
24	0,6998	9.3748	199,02
25	0,5998	8.5594	140,12
26	0,4992	7.6086	104,84
27	0,4	6.2274	85,67
28	0,298	4.5694	74,52
29	0,1993	2.9332	67,91
30	0,982	1.2492	69,82

Table 2 examines the amount of nitrogen vapor absorbed by the ion exchanger at low surface pressures and presents the results of the analysis. In this case, we have established that the correlation coefficient is $r = 0.430378$, and the constant C is -2.283 , and the surface area is 0.913 ml/g .



Rice. 2. Multi-point BET graph.

In Fig. Figure 2 shows a linear graph of the BET equation $1/[W(P_0/P - 1)]$ versus P/P_0 ; for most solids, the adsorption isotherm region has been studied when using nitrogen as an adsorbate, usually between 0.05 and 0.35 P/P_0 . This linear field is used to study pores at low relative pressures of microporous materials. Figure 2 shows the BET graph [7].

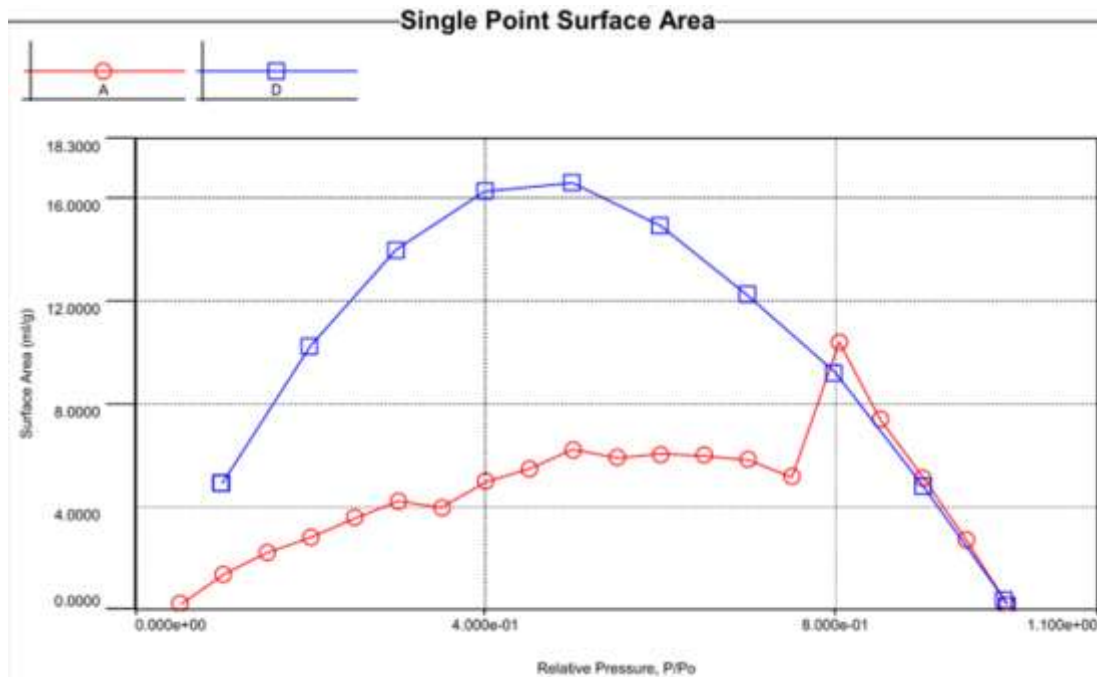


Fig. 3. Surface area at a point

For conventional surface area measurements, BET values are given separately for adsorption at each absorption point. A simplified approach using only one point on the isotherm is possible. The C value for nitrogen is usually large enough to indicate that the intercept in the BET equation is zero.

Conclusion.

Thus, the study of the absorption of nitrogen vapor on samples of the DSDAF ion exchanger at a pressure of 0.950 P/P_0 was 12.4811 cm^3/g . When absorbing nitrogen vapor, it is clear that nitrogen vapor is absorbed up to 0.750 P/P_0 and 4.7677 cm^3/g , after which capillary condensation is observed. After which it is assumed that nitrogen vapor forms bonds with the ion exchanger, then the desorption process occurs at a given pressure of 0.996 P/P_0 .

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