العدد 15 تشرين2 2024
No.15 Nov 2024
المجلة العراقية للبحوث الإنسانية والإجتماعية والعلمية
Iraqi Journal of Humanitarian, Social and Scientific Research
Print ISSN 2710-0952 Electronic ISSN 2790-1254

Synthesis of Activated Carbon from Fabaceae Astaglis Spinosal plant and study of its adsorption properties

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Abstract:

In this research, four models of activated carbon were prepared from the stems of the Fabaceae Astaglis Spinosal plant (takik plant*), where the stem of the plant was cut into pieces of equal size and it was first carbonized as it was heated without air for one hour and until the end of the rise of vapors and gases, and the pieces of carbon were soaked with different concentrations from the base (Sodium hydroxide) for 24 hours, then the samples were filtered and dried, and then the final carbonization of the samples was carried out for 30 minutes and until the vapors and gases ceased to rise. The results showed that model 4 (non-activated carbon 1:2 base) is the best in terms of absorbing methylene blue dye from its aqueous solution and model 3 (non-activated carbon 1:1.5 base) is the best in terms of absorbing iodine from its aqueous solution. The physical properties of humidity, ash and density were within the acceptable academic and commercial limits. BET measurements showed that model 3 (non-activated carbon 1:1.5 base) had the largest surface area, while model 4 (non-activated carbon 1:2 base) gave the largest pore size, as shown in the results table. Table (3). (takik plant*) = It is a common name for a plant that grows abundantly in southern Mosul, Iraq.

Key Words: Activated Carbon, Fabaceae Astaglis Spinosal plant, takik plant*, Chemical Treatment, Activation with sodium hydroxide.

تحضير الكاربون المنشط من نبات Fabaceae Astaglis Spinosal ودراسة خواصه الامتزازية

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المديرية العامة لتربية نينوى

الخلاصة:

في هذا البحث تم تحضير اربع نماذج من الكاربون المنشط من سيقان نبات الطكيك* حيث تم قطع ساق النبات الى قطع متساوية في الحجم وتم كربنتها كربنه اولية اذ سخنت بمعزل عن الهواء لمدة ساعة واحدة ولحين انتهاء تصاعد الابخرة والغازات وتم نقع قطع الكاربون بتراكيز مختلفة من القاعدة (هيدروكسيد الصوديوم) ولمدة س24 ساعة وتم ترشيح وتجفيف النماذج ومن ثم اجريت الكربنة النهائية للنماذج ولمدة 30 دقيقة ولحين انتهاء تصاعد الابخرة والغازات. واظهرت النتائج ان النموذج 4 (فحم 2:1 قاعدة) هو الافضل من حيث امتصاص صبغة المثلين الزرقاء من محلولها المائي والنموذج 3 (فحم 1.5: 1 قاعدة) الافضل من حيث امتصاص اليود من محلوله المائي. وكانت الخواص الفيزيائية الرطوبة والرماد والكثافة ضمن الحدود المقبولة اكاديميا وتجاريا. اما قياس الـ BET فقد كان النموذج 3 (فحم 1.5:1 قاعدة) يمتلك اكبر مساحة سطحية (BET Surface area) بينما النموذج 4 (فحم 1: 2 قاعدة) اعطى اكبر حجم للمسامات (pore volume , pore size) وكما هو مبين في الجدول (3).نبات

الطكيك* هو اسم شائع لنبات طبيعي شوكي ينموا بكثرة في جنوب مدينة الموصل العراقية في فصل الربيع

الكلمات المفتاحية: كاربون منشط ، نبات الطكيك، المعالجة الكيميائية ، التنشيط مع هيدروكسيد الصوديوم

Introduction:

Activated carbon is one of the most important industrial carbon and has been known for thousands of years. The ancient Egyptians used it since 2000 BC. Purification of water used for medical purposes, and the first development of this type of carbon was during the first world war and its development was accompanied with the development of the industry.(1) Porous carbon is composed of irregular, non-integral hexagonal rings of carbon especially at the edges, this composition adds some special properties to activated carbon, an example of randomness the distribution of activated carbon rings leads to the presence of holes or gaps of varying size and varying in the shape is such that the surface area of one gram of activated carbon (that is, the sum of the voids existing up to 1000 m^2) this means that 5 grams of activated carbon may be equal to it is the size of a football field. Incomplete activated carbon rings at the edges lead to the presence of some atomic groups on the surface (acidic , basic, neutral).The nature of the raw material used to prepare the activated carbon, in addition to the method of activation, is one of the most important factors affecting the quality and quantity of surface pores. Activated carbon absorbs pollutants through the pores spread on the surface, where chemical bonds are formed between the atoms or molecules of the unsaturated adsorbent (activated carbon). Electronically, the atoms or molecules of the adsorbed material (impurities), then the adsorption is called an adsorption chemical, or that adsorption takes place through the voids and pores where the adsorbent material travels through the voids reach the inner surfaces of the pores and are automatically connected to them by vender’s vals forces, and then the adsorption is called physical adsorption, since the adsorption force in this type of the adsorption depends on the type of pores and the activated carbon in addition to the size of adsorbed molecules.

The raw material used to produce activated carbon, as well as the production method, play a role. It is important to determine the size and shape of the pores, and this is the reason for the difference in porosity between types of activated carbon. During the activation process, the space between the carbon crystals is cleaned from various carbon materials and from irregular carbon. As carbon is removed from between the graphitic layers, the voids resulting from this cleaning process are called pores, and studies indicate the presence of several forms of pores connected to the internal structure (its shape resembles the shape of an ink bottle), and there are open capillary-like pores on both ends or closed on one side, and pores that take the form of cracks that connect between layers the sample has V-shaped pores, pointed pores, and pores of other shapes. (2)

The pore size and internal pore structure formed during carbon activation processes by the activator affects its adsorption properties. (3-5)

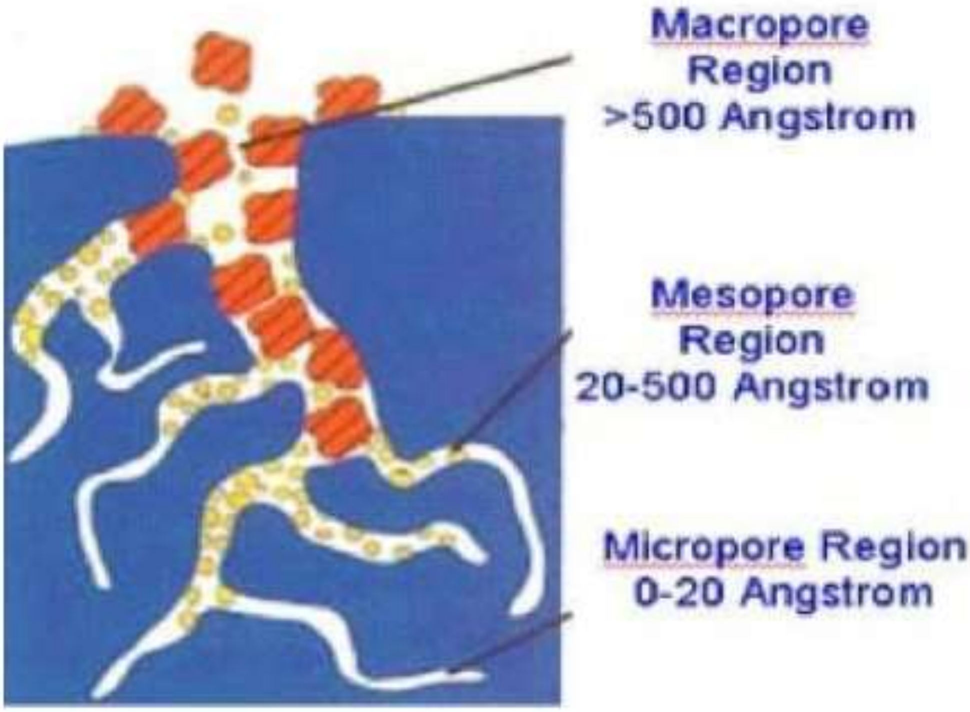

Figure (1) The porous structure of activated carbon

Activated carbon with well-developed porosity can be prepared by activation with sodium hydroxides by changing the experimental conditions. However, from the application point of view, fine porosity conditioning (not only pore size and surface area but also pore size distribution) is the most important aspect of the activated carbon process. Thus, the distribution of the pore size on the activated carbon samples more accurately makes them have high adsorption properties. (6,7)

Activated carbon is considered a unique material due to its large specific surface area, porous structure, adsorption properties, and high surface reactivity. Among the most important industrial applications of activated carbon are:

1- Removing odor, color and taste, in addition to removing unwanted organic impurities from the treated water resulting from the treatment of domestic and industrial water.

2- Solvent recovery.

3- Purify the air in crowded areas such as restaurants and hospitals.

4- Purifying the air by removing harmful gases and vapors and removing some complex pollutants such as pesticides and mineral oils.

5- Comb carbon is used to remove ammonia from acetic acid.

6- It is used to remove volatile organic compounds from dry cleaning and paint rooms, and to clean and rehabilitate polluted sites. (8)

Experimental

1- Preparing Samples of wood for Carbonization Purpose:-

A stem of the Fabaceae Astaglis Spinosal plant was taken from the perennial parts and left in the air till they dyed, then, it was cut into homogeneous pieces, then the pieces were put in a 110-130Co furnace until they had were an equilibrium weight thereafter. The pieces were made smaller to get the carbon able size, by using the base NaOH.

2- Primary Carbonization by Soaking Method:-

The primary material was taken (the non-activated carbon), by various ratios (0.5, 1, 1.5, 2) from the base (NaOH), (the Carbonizing) and activating material for 48 hours, then all the samples were filtered in a normal way without using any filtering papers. All the models were dried and their weights were calculated (dried at 110-130Co), and all dried samples were kept in for final carbonizing.

3- Final Thermal Carbonization and the Final Activation of the Various Samples:-

The resultant material from the primary carbonization has been taken and warmed up to a temperature of about (550±50Co) by direct flame for a 3 hour period of time. Then the samples were cooled off to room temperature. (9,10)

4- The Carbon Activation and Purification:-

1- The samples of prepared carbon were washed by distilled water for the purpose of removing the inexperienced sodium hydroxide, and for the confining of the equivalence of the resultant water of the washing process by using sunflower paper as evidence. Then all the samples dried out in a furnace at (110-130Co) for a period of 3 hours to obtain a constant weight for the sample.

2- After finishing the washing process and drying, the carbon sample is put in a round flask. Then a quantity of 10% hydrochloric acid was added. The solution was boiled for two hours, and after that, the carbon was infiltrated and washed by the distilled water until the equivalence of the washing water. After this process, the sample is dried at (110-130Co) until the weight is constant, then the sample is kept in a container, sealed until the next usage in measurements.

5- Determination of the Internal and External Pores in Activated Carbon:-

5.1- Measurement of Internal area for Activated Carbon using Iodine adsorption from aqueous solution:-(11,12)

The measurement of the adsorbed iodine amount by the activated carbon samples, is regarded as an easy and quick method to obtain information about the inner surface dimension, and the determination of the amount of the adsorbed iodine by milligrams of one gram of the activated carbon, and the procedure implies the following:-

1. Transfer one activated carbon gram in a conical flask with 250ml capacity, an amount of 10ml of 10% HCl acid .

2. Boil the mixture for half hours, then the solution left to cool off to the laboratory temperature.
3. 100 ml of iodine solution (0.1N), transferred to a flask, then sealed by a light stopper the flask put in the electrical agitating device for 30 minutes, then all its contents filtered material and remainder collected in dry flask.
4. 50 ml of the filtered transferred, and was put in a conical flask of 250ml capacity, then diluted with a standard solution of the sodium thiosulfate dihydrate (0.1N), till it becomes of pale yellow color.

Finally we added one milliliter of starch pilot the color of the solution will change the blue color and according to the size of the sodium thiosulfate dihydrate (0.1N), till it acquires the yellow pale color, then one ml of starch pilot is added (the solution color will shift to blue), and the process of admixing is continued until the blue color disappear according to the consumed sodium thiosulfate dihydrate through the sizes differences.

5- The following formula has been applied for the account of adsorbed iodine weight by the activated carbon:-

$X = A - [2.2B \times \text{ml volume of sodium thiosulfate dihydrate}]$

$A = N1 \times 12693$…..

$B = N2 \times 126.93$….

Whereas:-

X = the adsorbed iodine weight in ml, by the activated carbon

N1 = Iodine solution concentration

N2 = sodium thiosolfate dihydrate concentration which (N1 = N2) equivalent to (0.1N)

As for the Iodine number, it is calculated by the following equation:-

I.N = $\frac{X}{M} D$ …..

M = the activated carbon sample weight used (1gm)

D = correction factor

5.2- Measurement of External Surface Area For Activated Carbon Using Adsorption of Methylene Blue From Aqueous Solutions:-(13,14)

The study of methylene blue adsorption from its aqueous solution, gives an idea about the molecules which have a high molecular weight, this method expresses the outer surface area for the activated carbon. We can define this value as the number of the milligrams of methylene blue which removed from its aqueous solution, by its adsorption on the outer surface of the activated carbon by one gram of the activated carbon, whereas (0.1) gram of the activated carbon was taken after adding a certain amount of methylene blue (20 ppm) and put in a dry conical flask, then the flask attached to the electrical shaker device for (24) hours at the laboratory temperature until the color is disappeared, at this case, an additional amount of the pigment solution is added, and the shaker continues until the color is fixed, then a certain amount of the solution is taken and put in a centrifugal apparatus for (3-5) minutes to get rid of the carbon particles, after that the purified solution is situated in an absorption cell, then the absorption is measured, and the value of the absorption is determined concerning the pigment solution at (665 nm) wave length (the wave length where the pigment is being absorbed).

The calculation of the removed pigment concentration, from the aqueous solution is done by the standard curve, which is prepared for this purpose, by taking various standard concentrations from pigment solution (5, 10, 15, 20, 25 ppm), and by measuring the absorption of these solutions at the wave length (665 nm) and by drawing a diagrammatic lines between the absorption lines and the concentrations.

6 - Measurement of Some Activated Carbon physical Properties:-

6.1- Determination of Humidity Content:-(15,16)

One gram of the wet activated carbon samples has been weighed and put in an oven at 150 Celsius for three hours ,The percentage of the humidity has been measured according to the difference in the weight.

%Humidity=

$$\frac{weight\ of\ the\ wet\ activated\ carbon - weight\ of\ the\ dry\ activated\ carbon}{weight\ of\ the\ dry\ activated\ carbon} \times 100$$

6.2- Percentage of Ash:- (17,11)

One gram of the activated carbon was taken and put in a crucible and the crucible was put in an electric oven at 1000C° for three hours, Then it was left to be cool for weight of different material which include the ash for each sample of the greenish activated carbon samples and finally to measure the percentage of ash in each sample.

$$\%Ash = \frac{weight\ after\ born}{weight\ befor\ born} \times 100$$

6.3- Determination of apparent Density:- (18,19)

The density is measured by putting any amount of activated carbon (after being crashed and sieved in specific sieves size 80 mmash) in volumetric bottle is weighed by using a sensitive scales and the density is measured as follows:-

$$Density = \frac{mass}{volume} \quad gm./cm^3$$

Results and Discussion

Samples	Ratio non-activated carbon : NaOH	Weight non-activated carbon before finally carbonization gm.	The weight of the final activated carbon after activation with the base gm.	Reducing the weight of activated carbon gm.	The percentage of activated carbon produced %
1	1 : 0.5	10	9.8	0.2	98
2	1 : 1	10	9.2	0.8	92
3	1 : 1.5	10	9.1	0.9	91
4	1 : 2	10	8.9	1.1	89

Table (1) :- change in weight Activated Carbon sample after Final activation with the base

When the initial carbonation of the stems of the Fabaceae Astaglis Spinosal plant (takik plant), 100 grams of the wood of the stems of the Fabaceae Astaglis Spinosal plant was taken and it was carbonized as a primary carbonation in isolation from the air. The resultant result of non-activated carbon was 67 grams. That is, the percentage of non-activated carbon was 67%.

Taking 10 grams of non-activated carbon produced from the primary carbonization with different proportions of the base were taken and the weight of the activated carbon produced after chemical treatment with the base was measured. A decrease in the weight of the activated carbon samples was observed, which indicates that the diameter of the internal and surface pores size increased and the prepared activated carbon became more adsorption; As shown by the results of the examination of methylene blue dye (M.B) and iodine adsorption (iodine number I.N). Table (2)

samples **measurements**	**S 1**	**S 2**	**S 3**	**S 4**	**BDH***
Density gm./cm^3	0.29	0.23	0.17	0.14	0.35
Humidity %	0.1	0.1	0.1	0.2	0.8
Ash %	2.8362	2.4377	2.1148	2.1502	3.2
M.B mg/g	168	202	330	441	90
I.N mg/g	283	292	713	697	908

Table (2):- The value of the measurement of activated carbon obtained by soaking with different concentrations of NaOH and the heat carbonization weight of each model before soaking is 10 gm.**BDH* =** Commercial Model (20)

In the table (2) above, we find that the values of **M.B** and **I.N** increased with the increase in the percentage of base added in the chemical activation process on the base. It was noticed that model S4 gave the best absorbance for methylene blue dye, and model S3 gave the highest absorbance for iodine.

We note that model S3 gave higher iodine absorption than model S4, and this indicates that some of the pores collapsed and disappeared when using a high percentage of base (non-activated carbon: base) model S4.

As for the physical properties of density, humidity and ash, they were within the acceptable academic limits.

Samples / measurements	S 1	S 2	S 3	S 4
BET Surface area M^2/g	198.4908	305.7128	481.5743	327.1606
Pore Volume cm^3/g	0.027665	0.042455	0.081025	0.115302
Pore Size (width) nm	2.17392	2.22033	2.24403	2.77310

Table (3):- Value BET Surface area, Pore Volume and Pore Size (width) For activated carbon models.

Analysis by using (BET) Technique:-

The BET method is one of the traditional methods that has a clear advantage in facilitating analysis, calculating the surface area, and evaluating the size of pores and channels (21). This technique relies on the use of nitrogen gas N2 and it is possible to use other gases such as CO_2 , Ar and water, However, nitrogen is preferred because it has a boiling point of (77°k , -195.79 °C) (22), as the results showed that the prepared activated carbon models possess large pore size and good surface area, especially the S3 , S4 Samples.

The results of the BET analysis can be seen in Table (3) , Figures (2) and Figures (3).

In Table (3), we notice that the surface area of the sample S3 gave the largest area, which proves that the pores on the surface of the sample S3 are larger and more spacious than the rest of the samples. When using a larger proportion of the base (more than non-activated carbon 1: 1.5 base), we notice the destruction of these pores and thus a decrease in the surface area of the activated carbon, as is the case in the S4 sample.

We also note that the size of the pores increases with the increase in the percentage of the added base upon chemical activation with sodium hydroxide for the prepared samples; The width of the pores also increases with the increase in the percentage of added base.

```
( S 1 )                         Summary Report

                                Surface Area
Single point surface area at p/p° = 0.300000000:  199.1324 m²/g
                          BET Surface Area:  198.4908 m²/g
                    Langmuir Surface Area:  320.6174 m²/g
                   t-Plot Micropore Area:  169.3194 m²/g
             t-Plot External Surface Area:  29.1714 m²/g
BJH Adsorption cumulative surface area of pores
    between 1.7000 nm and 300.0000 nm width:  19.170 m²/g
BJH Desorption cumulative surface area of pores
    between 1.7000 nm and 300.0000 nm width:  7.7946 m²/g
D-H Adsorption cumulative surface area of pores
    between 1.7000 nm and 300.0000 nm width:  12.064 m²/g
D-H Desorption cumulative surface area of pores
    between 1.7000 nm and 300.0000 nm width:  7.3065 m²/g

                                Pore Volume
Single point adsorption total pore volume of pores
less than 40.4123 nm width at p/p° = 0.950000000:  0.107876 cm³/g
Single point desorption total pore volume of pores
less than 40.4123 nm width at p/p° = 0.950000000:  0.116121 cm³/g
                     t-Plot micropore volume:  0.087494 cm³/g
BJH Adsorption cumulative volume of pores
    between 1.7000 nm and 300.0000 nm width:  0.034052 cm³/g
BJH Desorption cumulative volume of pores
    between 1.7000 nm and 300.0000 nm width:  0.021277 cm³/g

                                Pore Size
Adsorption average pore diameter (4V/A by BET):  2.17382 nm
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( S 2 )                         Summary Report

                                Surface Area
Single point surface area at p/p° = 0.300000000:  308.3195 m²/g
                          BET Surface Area:  305.7128 m²/g
                    Langmuir Surface Area:  494.2648 m²/g
                   t-Plot Micropore Area:  251.6677 m²/g
             t-Plot External Surface Area:  54.0450 m²/g
BJH Adsorption cumulative surface area of pores
    between 1.7000 nm and 300.0000 nm width:  45.718 m²/g
BJH Desorption cumulative surface area of pores
    between 1.7000 nm and 300.0000 nm width:  36.0244 m²/g
D-H Adsorption cumulative surface area of pores
    between 1.7000 nm and 300.0000 nm width:  26.565 m²/g
D-H Desorption cumulative surface area of pores
    between 1.7000 nm and 300.0000 nm width:  28.5359 m²/g

                                Pore Volume
Single point adsorption total pore volume of pores
less than 40.4123 nm width at p/p° = 0.950000000:  0.169696 cm³/g
Single point desorption total pore volume of pores
less than 40.4123 nm width at p/p° = 0.950000000:  0.176708 cm³/g
                     t-Plot micropore volume:  0.130714 cm³/g
BJH Adsorption cumulative volume of pores
    between 1.7000 nm and 300.0000 nm width:  0.047473 cm³/g
BJH Desorption cumulative volume of pores
    between 1.7000 nm and 300.0000 nm width:  0.037437 cm³/g

                                Pore Size
Adsorption average pore diameter (4V/A by BET):  2.22033 nm
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( S 3 )                         Summary Report

                                Surface Area
Single point surface area at p/p° = 0.300000000:  480.3124 m²/g
                          BET Surface Area:  481.5743 m²/g
                    Langmuir Surface Area:  782.1117 m²/g
                   t-Plot Micropore Area:  374.4741 m²/g
             t-Plot External Surface Area:  107.1002 m²/g
BJH Adsorption cumulative surface area of pores
    between 1.7000 nm and 300.0000 nm width:  92.415 m²/g
BJH Desorption cumulative surface area of pores
    between 1.7000 nm and 300.0000 nm width:  82.3138 m²/g
D-H Adsorption cumulative surface area of pores
    between 1.7000 nm and 300.0000 nm width:  52.960 m²/g
D-H Desorption cumulative surface area of pores
    between 1.7000 nm and 300.0000 nm width:  65.1692 m²/g

                                Pore Volume
Single point adsorption total pore volume of pores
less than 40.4123 nm width at p/p° = 0.950000000:  0.270167 cm³/g
Single point desorption total pore volume of pores
less than 40.4123 nm width at p/p° = 0.950000000:  0.278835 cm³/g
                     t-Plot micropore volume:  0.192800 cm³/g
BJH Adsorption cumulative volume of pores
    between 1.7000 nm and 300.0000 nm width:  0.086687 cm³/g
BJH Desorption cumulative volume of pores
    between 1.7000 nm and 300.0000 nm width:  0.075363 cm³/g

                                Pore Size
Adsorption average pore diameter (4V/A by BET):  2.24403 nm
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(S 4)                           Summary Report

                                Surface Area
Single point surface area at p/p° = 0.300000000:  332.1201 m²/g
                          BET Surface Area:  327.1606 m²/g
                    Langmuir Surface Area:  671.3581 m²/g
                   t-Plot Micropore Area:  239.2460 m²/g
             t-Plot External Surface Area:  87.9146 m²/g
BJH Adsorption cumulative surface area of pores
    between 1.7000 nm and 300.0000 nm width:  103.910 m²/g
BJH Desorption cumulative surface area of pores
    between 1.7000 nm and 300.0000 nm width:  83.9260 m²/g
D-H Adsorption cumulative surface area of pores
    between 1.7000 nm and 300.0000 nm width:  62.325 m²/g
D-H Desorption cumulative surface area of pores
    between 1.7000 nm and 300.0000 nm width:  66.6799 m²/g

                                Pore Volume
Single point adsorption total pore volume of pores
less than 40.4123 nm width at p/p° = 0.950000000:  0.226812 cm³/g
Single point desorption total pore volume of pores
less than 40.4123 nm width at p/p° = 0.950000000:  0.234494 cm³/g
                     t-Plot micropore volume:  0.125377 cm³/g
BJH Adsorption cumulative volume of pores
    between 1.7000 nm and 300.0000 nm width:  0.125884 cm³/g
BJH Desorption cumulative volume of pores
    between 1.7000 nm and 300.0000 nm width:  0.104739 cm³/g

                                Pore Size
Adsorption average pore diameter (4V/A by BET):  2.77310 nm
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Figure (2):- Value BET Surface area, Pore Volume and Pore Size (width) For activated carbon samples.

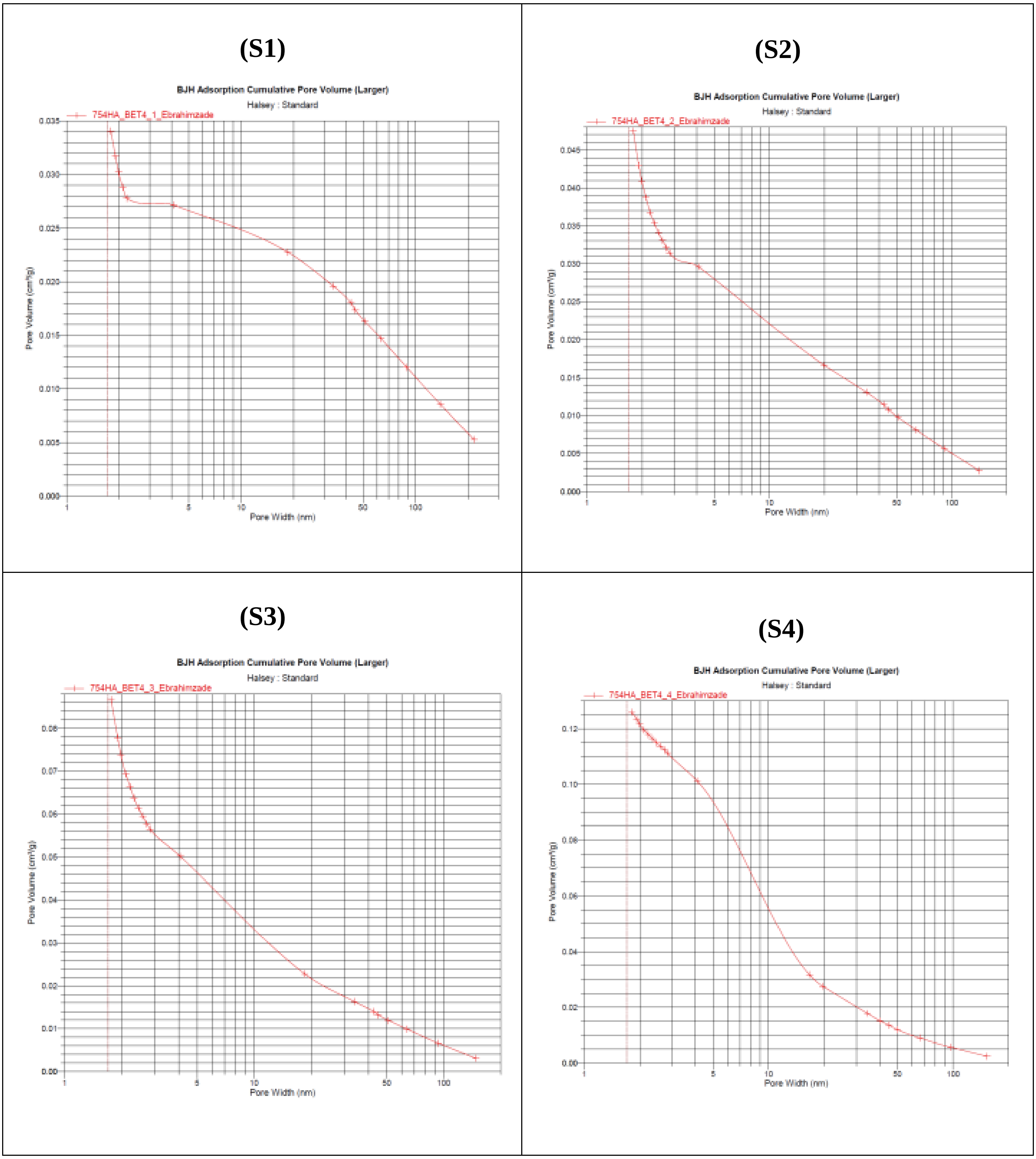

Figure (3):- BET Isotherm Adsorption For activated carbon samples (Pore width Vs. Pore Volume).

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