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Preparation Of Activated Carbon From The Stems Of The Caparis Spinosa Plant By Chemical Treatment With Sodium Hydroxide And Identification Of Some Of Its Physical Properties And Study Of Its Adsorption Qualities By Examining BET.

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Abstract
In this study, caper stems were used to prepare activated carbon samples. The stems of the plant were cut into small pieces of approximately equal size, and they were left at laboratory temperature to dry for seven days. After that, it was placed in the primary carbonization device (a metal cylinder containing small holes for the exit of water vapor and smoke). After the initial carbonization, the carbon was left to cool, then it was finely ground with an iron mortar, and the final carbonization and chemical activation process was carried out with sodium hydroxide (NaOH). It was washed with a dilute hydrochloric acid solution (10% HCl) and distilled water. Finally, it was placed in glass containers in an oven at a temperature of 150 degrees for 3 hours to get rid of moisture, after which the required measurements were taken on it. It was noted that model 4 gave the best absorbance of the blue dye. While model 3 gave the highest value of iodine uptake (iodine number). The physical results (density, humidity, ash) showed similar values and within acceptable limits academically and practically.
Keywords: *activated carbon, Caparis spinosa plant, chemical treatment with sodium hydroxide, examining BET.*

INTRODUCTION

Activated carbon is one of the most versatile adsorbent materials. It is used in the effective removal of impurities from its aqueous solutions. The use of activated carbon prepared from vegetable sources (Charcoal) dates back to 2000 BC, when the ancient Egyptians used it to purify water used for medical purposes, and activated carbon has a strong physical adsorption. The pore size is larger than any known commercial adsorbent(1, 2).Activated carbon is defined as a group or classes of materials that have a large internal surface area and an advanced porous structure. For this reason, activated carbon has a high adsorption capacity for chemicals. Therefore, it is used especially in industry for the purpose of purifying gases and liquids, or as catalysts or as a support material for catalysts. Activated carbon occupies an advanced position In the commercial market, this is due to the unique properties that it enjoys, as well as the low cost of its production when compared with other inorganic adsorbents such as clays, activated carbon and a wide distribution in the size and types of pores, and that the shape of these pores has a clear change if compared to the size of the pores It is almost stable in zeolite and this characteristic makes activated carbon a multi-use adsorbent(3, 5).Activated carbon is adsorbed in an amount equal to 25) - (100% of its weight. Its industrial uses include purification of sugar solutions in the manufacture of white sugar and removal of unpleasant odors and colors from drinking water. It is also used in masks from toxic gases and large air conditioning systems, and in the recovery of fumes and solvents in manufacturing processes As the adsorbents on the surface of the activated carbon can leave the surface of the carbon by raising the temperature, and thus the adsorbent materials can be recovered and the activated carbon re-used. It is estimated that the United States of America produces of activated carbon at (610 x 75) tons annually (6, 7).Activated carbon is a chemically and thermally stable substance, and the change in its effectiveness is attributed to its possession of surface oxygen groups, which enables it to absorb metal ions from aqueous solutions. Thus, activated carbon can be used in the treatment of water contaminated with mineral ions (8, 9).

MATERIALS AND METHODS

1- Preparing Samples of wood for Carbonization Purpose:- (10)

The stems of the Caparis spinosa plant were allowed to dry in the air before being cut into uniform pieces. The pieces were then placed in a furnace at 110 to 130 degrees Celsius until they reached equilibrium weight. Finally, they were reduced in size using the base NaOH to achieve a carbonize-able size.

2- Primary Carbonization by Soaking Method:- (11)

All of the samples were filtered normally without the use of filtering sheets after the stems of the Caparis spinosa plant were soaked for 48 hours in various ratios (0.5, 1, 1.5, 2) of the base (NaOH), the carbonising agent, and the activating material. All of the dried samples were kept for the last carbonising stage after the models were dried at 110 to 130 degrees Celsius and their weights were determined.

3- The final stage of thermal carbonization and the final activation of the various samples: (13)

The material resulting from the primary carbonization process has been attained, and warmed to a temperature approximately (550±50Co) by a direct flame for 3 hours over a period of time of 3 hours. After that, the samples were decreased to a temperature that was comfortable to you.

The procedure of activating and purifying carbon is called carbon farming.

1. The created carbon samples were rinsed with distilled water in order to eliminate any remaining sodium hydroxide that was not interacted with. Sunflower paper was employed as a witness to confirm that the amount of water remaining was limited. To obtain a consistent mass sample, all of the samples were then dried out in a furnace that was set to 110-130 degrees Celsius for three hours.

2- The carbon sample was inserted into a round container following the conclusion of the washing and drying processes. A 10% solution of hydrochloric acid was then incorporated. After 30 minutes of boiling the solution, the carbon was dissociated and removed with distilled water until the washing water's composition was identical. Following this protocol, the sample's weight was held constant at 110-130 degrees Celsius and the sample was dried. It was then combined and stored in a container for further measurement.

5.Finding the Internal and External Pores of Activated Carbon:

-5.1- The internal area of activated carbon is measured by iodine adsorption from aqueous solution: (16–20)

The measurement of the adsorbed iodine amount by the activated carbon samples is considered to be simple and quick, this method is used to obtain information about the interior volume of a vehicle, and the determination of the amount of the adsorbed iodine by milligrams by one gram of the activated carbon, the procedure involves the following: -

1. Add one gram of activated carbon and 10 ml of 10% HCl to a 250 ml conical vessel.
2. Combine the ingredients and bring them to a boil for 30 minutes. After that, let the mixture cool to a temperature of room.
3. 100 millilitres of the 0.1N solution of iodine were transferred to a flask that was sealed with a lightweight stopper, this was then placed in an electrical agitating device for half an hour. The contents of the flask were then separated, and the remainder was gathered in a separate dry flask.
4. 50 ml of the collected material was transferred to a 250 ml conical bottle. It was then mixed with a standard sodium thiosulphate dehydrate (0.1N) solution to dilute it, until it acquired a pale yellow color.

When we finally added one millilitre of starch pilot, the solution's colour changed from blue to yellow, depending on the size of the sodium thiosulphate dehydrate (0.1N). Then, we added another millilitre of starch pilot, which caused the solution's colour to change to blue. The admixing process was then continued until the blue colour vanished based on the sodium thiosulphate dehydrate that was consumed and the size differences.

5. The weight of iodine adsorbed by the activated carbon has been calculated using the following formula:

"X = A – [2.2B × ml volume of sodium thiosulfate dehydrate]

A = N1 × 12693....

B = N2 × 126.93....

Whereas: -

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

N1 = Iodine solution concentration

N2 = sodium thiosulfate dehydrate 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-Using the Adsorption of Methylene Blue from Aquous Solution to Estimates the External Area of Actived Carbon: (21)

The adsorption study of methylene blue from an aqueous solution offers insights into the interaction of large molecular weight compounds with activated carbon, specifically reflecting the external surface area of the adsorbent. In this process, 0.1 grams of activated carbon were used and exposed to a 20 ppm methylene blue solution. The quantity adsorbed is interpreted as the number of milligrams of dye removed from the solution per gram of activated carbon, representing surface binding efficiency. The mixture was then placed on an electric shaker for 24 hours at room temperature to allow for maximum adsorption. After the visible disappearance of color, additional solution was added, and the shaking process continued until equilibrium was achieved. To measure the concentration of the unadsorbed dye, the solution was centrifuged for 3–5 minutes to separate the carbon particles. The clarified solution was then transferred to an absorption cell, and absorbance was recorded at 665 nm—the characteristic wavelength of methylene blue—to assess the amount of dye retained in the solution.The measurement of the pigment's concentration that was removed from the aqueous solution is accomplished by the standard curve, which is created for this purpose by taking different concentrations of pigment from the solution (5, 10, 15, 20, 25 pmm), and by measuring the absorption of these solutions by creating a line between the concentrations and the absorption lines at the wavelength (665 nm

6-Measuring Certain Physical Properties of Activated Carbon: 6.1-Humidity Content Determination: (22)

After calculating the weight difference of 1 gram of the wet activated carbon samples and placing it in a 150 degree Celsius oven for 3 hours, the percentage of humidity was determined.

$$\%\text{Humidity} = \frac{\textit{weight of the wet activated carbon} - \textit{weight of the dry activated carbon}}{\textit{weight of the dry activated carbon}} \times 100$$

6.2- Ash percentage: - (23)

To determine the percentage of ash in each sample, one gramme of activated carbon was placed in a crucible, and placed in an electric oven that was set to 1,000 degrees Celsius for three hours. The crucible was then allowed to cool down for the weight of the various materials, including the ash, for every sample of activated carbon that is greenish.

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

6.3- The determination of apparent density: - (24)

The concentration is determined by placing a specific amount of activated carbon (after being crashed and then sieved in a specific size of 80 mmash) in a volumetric bottle and weighing the bottle with a sensitive scale. The concentration is then determined as follows: -

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

RESULTS AND DISCUSSION

Resveratrol purification from black grape skin

In this research, different ratios of the base were taken with the Non-activated carbon in order to activate. The base (NaOH) ratios taken are (base: non-activated carbon, 0.5:1, 1:1, 1.5:1 and 2:1).

Table 1: Change in Weight Activated Carbon Sample after Final Activation with the Base

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 percentages 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) shows the percentages of the base (NaOH) used for activation, weight non-activated carbon before finally carbonization, the weights of the final activated carbon after activation with the base, Reducing the weight of activated carbon and the percentages of activated carbon produced.Density, Humidity and ash measurements were within the acceptable values. As for Methylene blue and Iodine number examination, samples (3) and (4) had the highest absorbance values. As we can see in Table (2) below:

Table 2: The value of the physical properties and the results of the adsorption test of methylene blue and the iodine absorption of the prepared activated carbon.

Samples measurements	**S 1**	**S 2**	**S 3**	**S 4**	**BDH***
Density gm./cm^3	0.227	0.152	0.133	0.130	0.35
Humidity %	0.028	0.032	0.036	0.035	0.8
Ash %	0.567	0.542	0.530	0.521	3.2
M.B mg/g	190	397	812	978	90
I.N mg/g	96	406	728	712	908

BDH* = Commercial Model. (25)

The BET Surface area as these models (4), (3) which gave the best measurement of MB and IN. The measurements of (BET) have proven that the tests are identical to M.B and I.N in terms of preference.
Model (4) gave the largest BET surface area (374.621 m2/g), and Model (3) had the largest surface area (362.944 m2/g). Also, the Pore Volume
and Pore Size (width) values were given preference to model (4), as it gave the largest values for these tests. As shown in Table 3:

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

Samples / **Measurements**	**S (3)**	**S (4)**
BET Surface area m^2/g	362.944	374.621
Pore Volume cm^3/g	0.0471	0.1385
Pore Size (width) Nm	6.8737	9.0923

Figures (Fig 1, Fig 2) show the measured BET Surface area values:

CONCLUSION

The ratio of 2 base:1 non-activated carbon was determined as the best activation ratio. The percentage of the prepared activated carbon was very high 89%, when using the ratio of sodium hydroxide 2:1 non-activated carbon, Table 1. As for the internal area, we note that model 3 (1.5) base: 1 non-activated carbon) gave the largest internal area as shown by iodine adsorption, Table (2). The reason for this is that increasing the base leads to the destruction of the internal pores of the activated carbon. While the external surface area is preferred for model 4 which gave the largest adsorption of methylene blue dye, Table (2). The results of the BET test (Table 3) were completely consistent with the measurements of iodine adsorption and methylene blue adsorption in terms of
preference (table 4).

Fig. 1. BET Surface Area measurement results for the model (3)

Full Report Set
MicroActive MicroActive Page 1
Serial # 283 Unit 1 Port 2

Sample: 2168DH_BET4_1_Ebrahimzade
Operator: Kamal_Shirzad
Submitter:
File: C:\Users\BET_DrBadiei...\2168DH

Started: 5/6/2023 1: Analysis Ad N2
Completed 5/6/2023 5: Analysis Ba -196.882 °C
Report Tim 5/6/2023 5: Thermal Co No
Sample Ma 0.0500 g Warm Free 11.3602 cm
Cold Free S 32.9716 cm Equilibratio 10 s
Low Pressu None Sample De 1.000 g/cm
Automatic I No

Summary Report

Surface Area	
Single poin	7.5008 m²/g
BET Surfac	362.944 m²/g
Langmuir S	-59.0404 m²/g
t-Plot Micro	15.5284 m²/g
t-Plot Exter	20.7660 m²/g
BJH Adsorp between 1.	41.247 m²/g
BJH Desor between 1.	30.0264 m²/g
D-H Adsorp between 1.	21.198 m²/g
D-H Desorp between 1.	24.1039 m²/g

Full Report Set
MicroActive MicroActive Page 1
Serial # 283 Unit 1 Port 2

Sample: 2168DH_BET4_1_Ebrahimzade
Operator: Kamal_Shirzad
Submitter:
File: C:\Users\BET_DrBadiei...\2168DH

Started: 5/6/2023 1 Analysis Ad N2
Completed 5/6/2023 5 Analysis Ba -196.882 °C
Report Tim 5/6/2023 5 Thermal Co No
Sample Ma 0.0500 g Warm Free 11.3602 cm
Cold Free S 32.9716 cm Equilibratic 10 s
Low Pressu None Sample De 1.000 g/cm
Automatic No

Isotherm Tabular Report

Relative Pr	Absolute P	Quantity Ad	Elapsed Ti
			01:34
0.001941	1.296795	0.168389	01:39
0.003316	2.214204	0.187969	01:41
0.005791	3.865072	0.211036	01:43
0.011274	7.524419	0.229829	01:46
0.024168	16.12649	0.261527	01:48
0.047655	31.8	0.337565	01:51
0.073066	48.75427	0.435412	01:54
0.107675	71.84972	0.611636	01:56
0.107412	71.66778	0.615589	01:59
0.130528	87.08451	0.763169	02:01
0.146823	97.96933	0.878239	02:03
0.170721	113.9173	1.069294	02:05
0.197318	131.6622	1.297487	02:07
0.222821	148.6817	1.548641	02:09
0.248945	166.1113	1.839965	02:11
0.273549	182.5176	2.126527	02:13
0.299267	199.6829	2.459289	02:15
0.319576	213.2339	2.736771	02:17
0.49685	331.4986	5.661143	02:20
0.889911	593.7322	16.89167	02:23
0.944501	630.2468	19.28667	02:25
0.950693	634.3222	19.75298	02:27
0.955003	637.2075	20.1566	02:29

Fig. 2. BET Surface Area measurement results for the model (4)

Full Report Set
MicroActive MicroActive Page 1
Serial # 283 Unit 1 Port 3

Sample: 2168DH_BET4_2_Ebrahimzade
Operator: Kamal_Shirzad
Submitter:
File: C:\Users\BET_DrBadiei...\2168DH

Started: 5/6/2023 1: Analysis Ad N2
Completed 5/6/2023 5: Analysis Bath -196.883 °C
Report Time 5/6/2023 5: Thermal Co No
Sample Mass 0.0362 g Warm Free 11.5830 cm³
Cold Free S 33.2585 cm³ Equilibratio 10 s
Low Pressu None Sample De 1.000 g/cm³
Automatic I No

Summary Report

Surface Area	
Single poin	36.5249 m²/g
BET Surfac	374.621 m²/g
Langmuir S	831.6396 m²/g
t-Plot Micro	2.1749 m²/g
t-Plot Exter	35.2872 m²/g
BJH Adsorp between 1.	78.475 m²/g
BJH Desor between 1.	72.8497 m²/g
D-H Adsorp between 1.	43.105 m²/g
D-H Desorp between 1.	58.6637 m²/g

Full Report Set
MicroActive MicroActive Page 1
Serial # 283 Unit 1 Port 3

Sample: 2168DH_BET4_2_Ebrahimzade
Operator: Kamal_Shirzad
Submitter:
File: C:\Users\BET_DrBadiei...\2168

Started: 5/6/2023 1: Analysis Ad N2
Completed 5/6/2023 5: Analysis Bath -196.883
Report Time 5/6/2023 5: Thermal Co No
Sample Mass 0.0362 g Warm Free 11.5830
Cold Free S 33.2585 cm³ Equilibratio 10 s
Low Pressu None Sample De 1.000 g/
Automatic I No

Isotherm Tabular Report

Relative Pr	Absolute P	Quantity Ad	Elapsed
			01:34
0.001784	1.190301	4.749803	01:46
0.005488	3.661773	5.674723	01:53
0.010848	7.238622	6.299851	02:02
0.021094	14.07512	6.936642	02:08
0.034698	23.15355	7.46002	02:12
0.053411	35.63986	7.952472	02:15
0.076577	51.09409	8.483674	02:17
0.092351	61.62563	8.81275	02:19
0.11239	74.99363	9.170521	02:21
0.137113	91.48479	9.42213	02:23
0.151323	100.9587	9.664198	02:26
0.176245	117.5981	10.10666	02:28
0.200965	134.0798	10.53978	02:30
0.225606	150.5358	10.86313	02:33
0.251025	167.5138	11.21765	02:35
0.275889	184.0731	11.58628	02:37
0.300693	200.6224	11.9998	02:39
0.320643	213.9459	12.3073	02:41
0.495517	330.6588	18.0924	02:43
0.818834	546.4243	39.91817	02:47
0.894555	596.8658	49.8953	02:50
0.940175	627.3417	59.09543	02:53
0.951549	634.8464	62.18425	02:56

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