Medical Journal of Ahl al-Bayt University, Vol.4, No.1, Aug.2025

Production of Granular Activated Carbon from Sawdust of Fig Stems After Mixing it With Different Proportions of Spent Lubricating Oils and Chemically Activating the Resulting Carbon with Sodium Hydroxide

Hyder Ibrahim Hamed Al-Taha
haideribrahim259@gmail.com
Al-Hamdaniya Department of Education, Al-Tawajna Mixed Secondary School, Mousl, 41001, IRAQ

ABSTRACT: In this study, different ratios of spent lubricating oil were used with fig wood (wood: spent oil, 1:1, 1:075, 1:0.5, 1:025), and it was carbonized at a temperature of 500 degrees Celsius (initial carbonization). The resulting carbon was chemically activated with sodium hydroxide in different proportions (non-activated carbon: sodium hydroxide 1:1, 1:0.5). Model 1 (1:1 wood: spent oil, 1:1 non-activated carbon: sodium hydroxide) was the best in terms of dye adsorption. Blue methylene, as well as in terms of iodine adsorption, in addition to the preference in physical properties (humidity, density, ash content).

Keywords: Fig wood, Activated carbon, Spend lubricating oil.

1. INTRODUCTION

Activated carbon occupies an advanced position in the commercial markets because of its unique properties and its low production cost when compared with inorganic adsorbents such as filler slurries. It is known as a material with a high carbon content and a variety of porous structure, which makes it an effective material for the adsorption of chemicals from its media [1]. Activated carbon was also known as a class of carbon materials with a well-developed and varied pore structure and a large internal and external surface area. Activated carbon has different types of pores that vary in size and shape when compared to the size of the almost fixed pores in zeolite, and this characteristic makes activated carbon a versatile adsorbent. Usage [2], it is used especially in industry as a catalyst or catalyst support material, and in removing brown color from white grape juice concentrate [3]. And with the continuation of dealing with carbon and the expansion of its areas of use, the adsorption properties of it were discovered, which greatly increased its importance. The first industrial applications of it were in Britain at the end of the eighteenth century, specifically in the year (1794 AD), when it was used to shorten sugar and remove colours from it. At the outbreak of the First World War and the advent of chemical weapons and the Germans' use of toxic gases in this war, the need for materials with high adsorption qualities appeared, and here the importance of carbon emerged in this field, as manufacturers began to pay attention to this material and improve its adsorption properties and try to produce it in a granular form of specific sizes for the purpose of use In the manufacture of gas masks. Activated carbon was prepared at the beginning of the twentieth century from coconut shells, but the increased demand for carbon and the limited quantities available of these crusts prompted manufacturers to try to manufacture it from other organic materials, as coal, coal tar, different types of wood, bitumen, and polymers were used. , bones, asphalt materials, and various other materials [4].

Lubricating oils are known as petroleum products that reduce friction of the metal parts of the engine, and it was previously relied upon to obtain them from animal and plant sources, and because of the increased demand for these products and their increased use in the world, it turned to crude oil, which is now the main source for it after conducting a series of operations Which includes vacuum distillation at (300-400)°C, then a second distillation takes place at a temperature of (450-480)°C, and the resulting distillation process is repeated at a temperature higher than (480)°C, after which all The extracts produced are in the three stages of distillation, and the processes of removing asphalt, extraction, filtering and hydrogenation are carried out. Accordingly, it can be said that the lubricating oils are produced from the oil extracts that boil between (300-650) ° C [5]. These oils consist mainly of a complex mixture of hydrocarbons, including both short-branched paraffins as well as monocyclic polyalkyl substituent naphthenic and aromatic compounds, and their chemical composition may contain one naphthenic ring linked to one or more chains of paraffin side chains of different lengths. The number of these side chains increased, the molecule structure approached that of the real paraffinic compound, and the degree of solidification increased. All these pictures do not give but a simplified idea about oils, as it is believed that the composition of these materials, especially those with high boiling points or non-volatile parts, is close to the composition of resins, in which the paraffin chains are linked with the naphthenic rings in various and multiple ways[6,7,8,9]. The molecular weight of the oil fractions ranges from (250) g/mol for molecules that have low viscosity to a molecular weight close to (1000) g/mol for molecules with high viscosity, and thus the carbon atoms in lubricating oils range between (20-30) atoms [10]. The value of the molecular weight is not affected by the nature of the solvent used or the temperature at which the measurement is carried out, which indicates that the recorded values are real values and are not the result of molecular agglomerations or assemblies [11].

2. Experimental

2.1 Preparing samples of wood for carbonization purpose

A stems of the fig plant were left in the air till dryness then, it was cut into homogeneous pieces, then the pieces put in a 110-130Co furnace until getting equilibrium weight, thereafter, Fig wood pieces were mixed with different proportions of spent lubricating oil (Different ratios were mixed between fig wood and spent oil: wood:oil 1:1, 1:0.75, 1:0.5, 1:0.25) and then , The mixture of wood and spent oil was then ground to prepare it for carbonation with the base NaOH.

2.2 Primary Carbonazation By Soaking Method

The stems of the Fig plant has been taken 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.

2.3 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 about (550±50Co) by a direct flame for 3 hours period of time. Then the samples were cooled off to room temperature [12,13].

2.4 The Carbon Activation And Purification

1. The samples of prepared carbon were washed by distilled water for the purpose of removing the un interacted sodium hydroxide, and till the confining of the equivalence of the resultant water of the washing process by using sunflower paper as an 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 put in a round flask. Then a quantity of 10% hydrochloric acid was added. The solution was boiled for 30 minutes, 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 dried at (110-130Co) until the weight is constant, then the samples kept in a container, sealed until the next usage in measurements.

2.5 Determination Of The Internal And External Pores In Activated Carbon

2.5.1. Measurement of Internal area for Activated Carbon using Iodine adsorption from aqueous solution: - [14,15].

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 by one gram of the activated carbon, and 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 colour.

Finally we added one milliliter of starch pilot the colour of the solution will change the blue colour and according to the size of the sodium thiosolfate dihydrate (0.1N), till it acquires the yellow pale colour, then one ml of starch pilot is added (the solution colour will shift to blue), and the process of admixing is continued until the blue colour 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:-

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

$A = N1 \times 12693\ldots..$

$B = N2 \times 126.93\ldots.$

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:-

$I.N = \ldots..$

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

D = correction factor.

2.5.2. Measurement of External Surface Area For Activated Carbon Using Adsorption of Methylene Blue From Aqueous Solutions:-[16,17]

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 colour is disappeared, at this case, an additional amount of the pigment solution is added, and the shaker continues until the colour 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 pmm), 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..

2.6 Measurement Of Some Activated Carbon Physical Properties

2.6.1. Determination of Humidity Content:-[18,19]

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=

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

2.6.2. Percentage of Ash: - [20,21]

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.

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

2.6.3. Determination of apparent Density: - [22,23]

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:-

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

3. Results And Discussion

Table 1. -Properties of activated carbon prepared from fig wood sawdust and activated with the base without adding spent lubricating oil.

Iodine adsorption g/mg	Absorption of methylene blue dye g/mg	Humidity %	Ash %	Density g/cm3	Lactin %	The scientific name	Common name
789	290	.06	4.130	0.226	32.8	Ficus caricá	Fig
908	90	0.8	3.2	0.345	---	B.D.H**	

** BDH [24]

Table (1) shows the adsorption properties and physical properties of activated carbon samples of fig wood not mixed with spent lubricating oil, where the values were close to the standard model BDH.

Table 2. - Physical properties and adsorption properties of activated carbon prepared from fig wood sawdust mixed with different proportions of spent lubricating oil

Iodine adsorption g/mg	Absorption of methylene blue dye g/mg	Humidity %	Ash %	Density g/cm3	Lactin %	Weight ratio Fig wood: spent oil g : g	١ models
972	239	1.658	6.130	0.182	32.8	1 : 1	1
1152	258	1.605	5.128	0.168	32.8	1:0.75	2
1245	512	1.592	3.297	0.152	32.8	1:0.50	3
1269	529	1.182	2.455	0.147	32.8	1:0.25	4

Treating fig wood with spent lubricating oil increases the adsorption of methylene blue dye and also increases the adsorption of iodine, as is clear in Table (2) above.

We note that the adsorption of methylene blue dye to model 3 increased from (290 without oil) to (512 with oil), and also the adsorption of iodine for the same model increased from (789 without oil) to (1245 with oil).

As for Model 4, it gave an increase for the adsorption of methylene blue dye from (290 without oil) to (529 with oil), and for iodine adsorption, the adsorption increased from (789 without oil) to (1269 with oil). As shown in Table (1 and 2) above.

Table 3. - Value Pore Volume, Pore Size (width) and BET Surface area for activated carbon models 3 and 4.

Samples / measurements	3	4
BET Surface area m^2/g	486.764	525.354
Pore Volume cm^3/g	0.1045	0.1148
Pore Size (width) nm	8.6125	10.1572

The BET test that was performed to determine the internal surface area and pore size showed the values shown in Table 3, and they were completely identical to the increase in surface area and pore

size on the one hand, and the increase in the adsorption values of methylene blue and iodine on the other hand. As shown in Table (3) and Tables (1 and 2).

Funding

None

Acknowledgement

None

Conflicts Of Interest

The author declares no conflict of interest.

References

[1] H. Marsh, "Activated carbon'', 1st Ed., Francisco Rodriguez-Reinoso, pp. 16, 182-183, 2006.
[2] D.F. Sheriver, P.W. Atkins, and C.H. Angford, "Inorganic Chemistry" , Oxford P.342, 1990.
[3] I. Mochida, Y. Kawabuchi, S. Kawano, Y. Matsumura, and M. Yoshikawa, "High catalytic activity of pitch-based activated carbon fibres of moderate surface area for oxidation of NO to NO2 at room temperature". Fuel, 76(6), 543-548, 1997.
[4] S. Parakash, "The Chemistry of Activated Carbon", J. Chemistry and Industry, Vol.1-13, p.445, 1974.
[5] O. S. Ogbeide, "An Investigation to the Recycling of Spent Engine Oil", J. Engineering Science and Technology Review Vol. 3, No. 1, PP. 32-35, 2010.
[6] J. G. Speight, "The Chemistry and Technology of Petroleum'', 2nd ed., Marcel Dekker, Inc., pp. 701- 704, 1991.
[7] J. G. Speight, "Handbook of Petroleum Product Analysis'', John Wiley and Sons, Inc., pp. 269-273, 2002.
[8] T. Mang, and W. Dresel, "Lubricants and Lubrication'' 2nd ed., WILEY-VCH verlag GmbH& Co. KGa A, British, pp. 36-40, 2007.
[9] L. R. Rudnick, "Lubricant Additives Chemistry and Applications', 1st ed., Marcel Dekker, Inc., New York, USA, pp. 1, 2, 27, 171-174, 2003.
[10] J. D. Udonne, "A comparative Study of Recycling of Used Lubrication Oils Using Distillation, Acid and Gass Engineering" Vol. 2, No. 2, PP.12-19, 2011.
[11] A. Abdul-Halim, K. Mohammed, and R. Safaa, "Dewaxing of Distillate Oil Fraction (400-500oC) Using Urea", J. of Engineering, Vol.13, No. 1, pp. 1268-1281, 2007.
[12] ASTM D2854-70, Standard test method for apparent density of activated carbon.
[13] R.C. Bansal, J.B. Donnet, F. Stoeckli, "Active carbon" , Marcel Dekker, 2nd ed . New York, 1988.
[14] A. R. Shepherd, and R. Austin, "Granular Activated Carbon for Water and Wastewater Treatment", Carbtrol Corporation September Rev, 10, 92, 1992.
[15] W.A. Abbasi, M. Streat, "Adsorption of Uranium from Aqueous Solution Using Activated Carbon", Journal of Separation Science and Technology 29(9), 1217-1230, 1994.
[16] J. Machnikowski, K. Kierzek, K. Lis, H. Machnikowska, and L. Czepirski, "Tailoring porosity development in monolithic adsorbents made of KOH-activated pitch coke and furfuryl alcohol binder for methane storage", Energy Fuels , 24, 3410–3414, 2010.
[17] I. Martin-Gullon, J.P. Marco-Lozar, D. Cazorla-Amoros, and A. Linares-Solano, "Analysis of the microporosity shrinkage upon thermal post-treatment of H3PO4 activated carbons." Carbon 42, no. 7: pp. 1339-1343, 2004.
[18] O. M. Hamdoon, "Production of activated carbon from spent lubricating oils by chemical treatment", National J. of Chemistry, Vol. 18, pp. 216-225, 2005.

[19] R. Ljubisa Radovic, “Chemistry and Physics of Carbon”, Taylor and Francis Group., Boca Raton London New York, V. 30, pp. 24-25, 2008.
[20] M. Naito, T. Yokoyama, K. Hosokawa, and K. Nogi, “Nanoparticle Technology Handbook”, 3rd Ed., Elsevier, p.p. 5,23-25, 2018.
[21] S. M. Manocha, "Porous carbons" Sadhana, 28, pp. 335-348, 2003.
[22] J. Toth, “Adsorption; Theory, Modeling and Analysis”, Marcel Dekker, Inc., p.p. 68-73, 80-81, 2001.
[23] D. O. Cooney, “Adsorption Design for Wastewater Treatment”, Lewis Publishers, Boca Raton, FL, 1999.
[24] O. M. Hamdoon, “Production of activated carbon from spent lubricating oils by chemical treatment”, National J. of Chemistry, Vol. 18, pp. 216-225, 2005.