



Preparation and Characterization of Activated Carbon from Household Waste Foods

Iman Hussein Zainulabdeen*

Fuel And Energy Department, Technical College, Kirkuk, Iraq.

E-Mail: imanhuseyin@yahoo.com

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Abstract

Waste food residues are considered as suitable raw materials for the production of low cost adsorbents. In this work, activated carbons were prepared from household waste food (orange peels, banana peels, walnut shells, olive stones and their mixture). Chemical carbonization at 500°C for 1.5hr was used to prepare carbons and their activation by KOH and CaCl₂ solutions for 24h. Then added 0.1g of activated carbons to the solution of blue dye prepared laboratory to demonstrate the activation of the types of activated carbons prepared to remove the blue dye. The results indicated that characteristics (yield, burn off, density, moisture content, ash content, pore volume, porosity percent, iodine number, methyl blue number and removal percent of methyl blue) for all activated carbons were compared with commercial activated carbon. It has been found that activated carbon from orange peels and mixtures activated with CaCl₂ had the best adsorption properties reach to the (80, 77.5) % removal blue dye respectively and iodine numbers (741, 735) mg/g. This low cost activated carbons can be used for wastewater treatment.

Keywords: Activated carbon, waste food, chemical activation.

تحضير ودراسة خواص الكربون النشط من فضلات الاطعمة المنزلية الخلاصة

الخلاصة

تعتبر الفضلات المنزلية الناتجة عن الاستخدام اليومي للأطعمة مادة أولية مناسبة لتحضير كربون منشط ذات كلفة واطنة. في هذه الدراسة تم تحضير الكربون المنشط من (قشور البرتقال وقشور الموز وغلاف الجوز ونواة الزيتون إضافة إلى مزيج مكون من جميع المواد المذكورة)، استخدمت عملية الكربنة الكيميائية في درجة حرارة 500 °م لمدة ساعة ونصف، بعدها تم التنشيط الكيميائي باستخدام محلولي KOH وCaCl₂ لمدة 24 ساعة بطريقة الغمر، تم إضافة 0.1 غرام من الكربون المنشط لمحلول الصبغة الزرقاء المحضر مختبرياً لبيان فاعلية أنواع الكربون المنشط المحضرة في إزالة الصبغة الزرقاء. نتائج الخواص المدروسة (الإنتاجية ونسبة الاحتراق وكثافة والمحتوى الرطوبة ومحتوى الرماد وحجم المسامات ونسبة المسامية والعدد الأيوني ونسبة إزالة الصبغة الزرقاء) لجميع أنواع الكربون المنشط المحضر مقارنة بالفحم النشط التجاري أظهرت بأن الكربون المنشط المحضر من قشور البرتقال ومزيج المواد بمحلول CaCl₂ أظهرت أحسن قيمة لخواص الإمتصاصية إذ بلغت نسبة إزالة الصبغة الزرقاء حوالي (80 و 77.5) % على التوالي والعدد الأيوني (741 و 735) ملغم/غم كربون نشط. الكربون النشط الواطي الكلفة يمكن استخدامها في عملية معالجة المياه الملوثة بالأصبغة.

Introduction

Pollution is one of the most serious problems that effect on the environment and all life aspects [1]. Pollution takes many forms, such as pollution of air, soil, water, noise and others. Water is one of the basic

needs of life on earth. It may be contaminated by natural sources or by industrial effluents [2]. Protecting water sources is essential to ensure human health. Every year at least five million people die from water-related diseases

*Corresponding author: E-mail : imanhuseyin@yahoo.com

worldwide. Hence, it is important to know whether common water treatment methods can effectively remove contaminants [3].

Many types of treatment are used to treat waters; physical, chemical and energy intensive methods. The most commonly used methods for the removal of contaminant from industrial effluents include: chemical precipitation, solvent extraction, oxidation, reduction, electrolytic extraction, reverse osmosis, ion-exchange, evaporation, cementation, dilution, adsorption, filtration, flotation, flocculation and sedimentation. However, it still seems necessary to develop a new method that attains a higher removal level at low cost [4,5].

For the past ten years, attention has been shifted towards adsorption technique, which emerged as one of the widely accepted methods for the removal of contaminants from wastewater [6].

Adsorption is a process during which a soluble material accumulates on the surface of another substance [7]. In fact, adsorption is the function of transferring from liquid phase to solid phase. This principle can be used for the removal of pollutants from wastewaters [8].

There are many adsorbents in use. Activated carbons are known as very effective adsorbents due to their highly developed porosity, large surface area, variable characteristics of surface chemistry, and high degree of surface reactivity. Activated carbon is a trade name for carbonaceous material, which is predominantly amorphous in nature [9-11].

Activated carbon is one of the most important types of adsorbents widely used as a tool in environmental protection in various industrial applications [12]. In recent time, special attention has been focused on the rise of natural adsorbents as an alternative to replace the commercial adsorbent, based on both the environmental and economical points of view [13]. Waste food materials offer the most available and cheapest of all the known raw materials. Activated carbon is inexpensive and hence very widely used adsorbent [11]. Researchers have studied the production of activated carbon from cheap and renewable resources, such as olive husk, coffee, cotton stalk, plum kernels, fruit peels, olive stone, seeds and walnut shell [14].

The production process of activated carbon is mainly divided into three steps: dehydration, carbonization, and activation. There are two process for preparation of activated carbon: chemical & physical activation [8,15]. Physical activation involves oxidizing the raw material at high temperatures (800-1000) °C in the presence of an oxidizing agent, usually water steam and CO₂ [16].

Chemical activation is known as a single step method of preparation of activated carbon in the presence of chemical agents such as ZnCl₂, H₃PO₄, H₂SO₄, KOH, and CaCl₂ that have dehydration and oxidation characteristics [15]. The chemical activation usually takes place at a temperature lower than that used in physical activation, therefore it can improve the pore development in the carbon structure because of the effect of chemicals. The yields of chemical activation are higher than physical ones [8].

In Iraq there are many people living far from cities in the villages & agriculture area depending on the waters from wells, rivers and lakes for drinking and daily uses, most of them haven't any water technique treatment due to its cost & limited powers for operation, accordingly this research aimed to:

1. Possibility of using food household waste to prepare activated carbon.
2. Study the characteristics of prepared activated carbon from waste material.
3. Compare the low cost activated carbon made by household waste with commercial activated carbon.
4. The possibility of using activated carbons in the treatment of water contaminated with dyes.

Materials and Methods

Collection and Dehydration of Raw Materials

Waste food used in this study are (orange peels, banana peels, walnut shell and olive stone) from houses, Figure (1).

The peels of orange and banana were washed with tap water three times and dried by leaving under sun rays for one week, then it crashed. This process increases the surface area of material and the efficiency. After that it dried by oven to remove any moisture in the crashed banana and orange at 95°C for 24h, then sieved through (1mm) size [17].

Walnut peels and olive stone washed and crashed by excruciating, then dried by an oven to remove any moisture content of the milled materials at a temperature (110°C) for 24h, then sieved through (1mm)[18], they need to higher temperature for drying more than orange and banana peels for being more hardness material.



Fig. 1. Waste foods (a: walnut shells, b: orange peels, c: banana peels, d: olive stone)

Carbonization

The samples (orange peels, banana peels, walnut shells, olive stone and their mixtures) are putted in a steel bottle containers, Figure (2), weighted the samples before burning, then putted in the furnace at the temperature 500°C for time 1.5hr[18,19]. At which all of the materials were completely carbonized, the carbonized materials were cooled and kept in containers, to find the solid yield by Equation (1) and the burn off by Equation (2):-

$$\text{Solid yield \%} = W_f / (W_i) * 100 \dots\dots\dots (1)$$

$$\text{Burn off \%} = (W_i - W_f) / W_i * 100 \dots\dots\dots (2)$$

W_i = Initial weight of raw material (g)

W_f = weight of final products of carbon (g)



Fig. 2. Furnace and steel container

Chemical Activation of Carbon

KOH and CaCl_2 are used by impregnation process to activated all waste food carbons:

A. Preparation of the CaCl_2 25%:- weighted 125g of the CaCl_2 , putted in the beaker 1000ml, added 500ml of the distilled water and mixed it, then 25g from each samples are impregnated with this solution for 24hr[20].

B. Preparation of the KOH 25%:- weighted 25g of KOH, mixed with 25g of each samples, then added 100ml of distilled waters and mixed it, left for 24hr [21].

After 24 hours, the samples were washed by distilled water (8–10) times and use pH meters for test washed water until the pH meter reads (6.5- 7.5). After that dried the samples in 70C° for 24hr in electric normal furnace to be ready to study the characteristics of activated carbons.

Characterization of Activated Carbon Density

The bulk density is defined as the mass of a unit volume of the sample in air. The sample Put in the cylinder of 10 ml and then weighted. The bulk density of activated carbon depends on the shape, size and density of the individual particles, it can calculate by Equation (3)[22].

$$\rho = \frac{M}{V} \dots\dots\dots (3)$$

ρ = Density (g/ml), M = mass (g)

V = volume (ml)

Moisture Content

Moisture content of activated carbon is measured by heating a known quantity (W_i) of sample in a preheated oven at 150°C for 3hr[22]. After heating, the sample is taken out from the oven and placed in a desiccator, it is allowed to cool to the room temperature and weight (W_f) is measured. The loss in the weight of the sample is the moisture content determined as follows:

$$\text{Moisture \%} = ((W_i - W_f) / W_i) * 100 \dots\dots\dots (4)$$

Where:

W_i = weight of the sample before heating.

W_f = weight of the sample after removal from the oven [23].

Ash Content

For the purpose of estimating the total content of the ashes placed 1g of sample to a crucible and reweighted with its content, heated in a muffle furnace at 650°C for 3hr, after that the sample was cooled to room temperature and reweighted. Ash content was calculated by Equation (5)[22].

$$A_c = (F - G) / (B - G) * 100 \dots\dots\dots(5)$$

Where :

G = weight of empty crucibles in g.

B = weight of the crucible plus dry samples in g.

F = weight of crucible plus Ash sample in g.

Iodine Number

Iodine number is very important characteristic in activated carbon, put 1g of activated carbon in the conical flask 250ml and add 10ml of HCL 5%. Then heated the contents for half hour, at after cooled add 100ml of iodine solution 0.1N, and shaking the mixture for 30min. The solution must be filtration cancel 20ml initial and take 50ml titration by 0.1N of sodium thiosulfate until the color of solution change from the orange to yellow color then added 2ml of starch solution were added and the titration was carried on until a colorless solution was produced, the iodine number is calculated from the Equation (6):

$$I_n = \frac{X}{M} A \dots\dots\dots(6)$$

Where :

X = mg of iodine adsorbed by the activated carbon

$$X = (12.692 N_1) - (279.246 N_2 * V)$$

N_1 = normality of iodine solution.

N_2 = normality of sodium thiosulphate solution.

V = volume of sodium thiosulphate solution in ml.

M = mass of activated carbon (g).

A = correction factor.

Methylene Blue Number

Prepare 1000ml (1500ppm) of methylene blue stay for 24hr, at the same time prepare 5 sample of methylene blue (5ppm, 10ppm, 15ppm, 20ppm, 25ppm) stay for 24hr also. Then take 0.1g from each sample and putted in 100ml (20ppm) methylene blue and put them in the shaking

device for mixing for 30 minutes until absorbed.

The calibration of the five samples of methylene blue of different concentration putted in the U.V. spectrometer to read absorbance value and draw the relation between the concentration and absorbency shown in Figure (3) after 30 minutes we read the absorbency of samples in U.V. and it must be cleaned the test container by alcohol and distilled water between each sample then we can read the residual concentration for each sample from Figure (3) and find the removal percentage (R%) of dye by the adsorbents and amount of dye adsorbed at equilibrium (q_e) (mg/g) were calculated by the following equation respectively[24,25].

$$\text{Removal \%} = ((C_o - C_e) / C_o) * 100 \dots\dots\dots(7)$$

$$q_e = (C_o - C_e) V / W \dots\dots\dots(8)$$

where:

C_o = is the initial concentration of M.B (mg/l)
 C_e = is the residual concentration of M.B concentration in solution (mg/l).

V = volume of the solution in liters.

W = amount of adsorbent used (g).

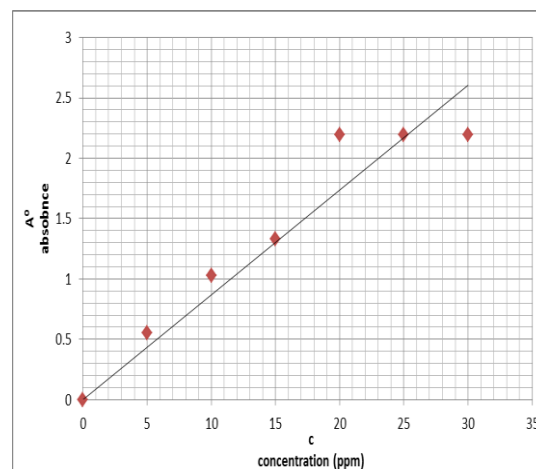


Fig. 3. Relation between concentration & absorbance

Pore Volume and Porosity

One gram of samples was transferred into a measuring cylinder of 10ml and volume of the particles was recorded. This sample was placed into a beaker containing 20ml of distillate water and boiled for 5 minutes (to displace air in the sample), the contain was superficially dried and weighted. The increase in weight of the sample divided by density of water gave the

pore volume for sample. Porosity was calculated by dividing the pore volume of the particle of the total volume of the particle [26].

Results and Discussion

Orange and banana, this samples has been chosen according there availability and they are most widely used in the houses all seasons and they are at good price (1000-1750)IQ, it is available in all families. Walnut is nuts that available in the north of Iraq and, it widely uses as a foods & in sweets making. Olive is used in all families and all days specially in the breakfast meal and others, it is a good price that make all the people able to use it. For that reasons it chose this four samples to prepare activate carbon. In addition to that, mixed this samples in purpose to get rid of waste food for one house.

Solid Yield & Burn Off

The yield is the quantity of the final product formed from the starting raw material. A high yield is required for a feasible economic production of activated carbon.

Figure (4) show the high yields is for olive stone 59.3% more than walnut shell 58.1%, this result corresponds to what was said both[27,28]. Others were mixture 50.43%, banana 46.4 and orange has the lowest yield 41.31% because it contains a large amount of aromatic volatile material[19], also it was show the higher sample has burn off 58.68%.

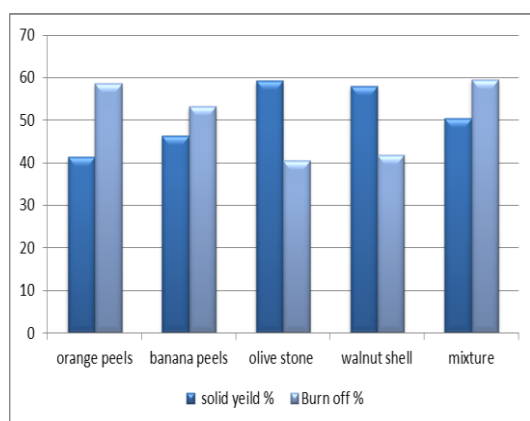


Fig. 4. Solid yield and burn off for activated carbons

Activated Carbon Characteristics

Result of the activated carbon characteristics are given in Table (1). Commercial activated carbon characters

are Incorporated for the purpose of comparison [29,25].

Table 1 : Characteristics of activated carbons

Activated Carbon type	Density g/ml	Moisture content %	Pore volume ml.	Porosity %	Ash %
Orange KOH	0.23	0.93	0.79	26.33	2.45
Orange CaCl ₂	0.22	0.90	0.94	31.44	2.38
Banana KOH	0.23	1.24	0.75	25.14	2.57
Banana CaCl ₂	0.21	0.92	0.82	27.36	2.53
Olive KOH	0.41	0.21	0.63	21.20	3.96
Olive CaCl ₂	0.38	0.20	0.68	22.66	3.67
Walnut KOH	0.35	0.39	0.70	23.36	2.85
Walnut CaCl ₂	0.32	0.31	0.73	24.40	2.84
Mixture KOH	0.25	0.80	0.80	26.85	2.38
Mixture CaCl ₂	0.24	0.30	0.91	28.66	2.31
C.A.	0.30 - 0.70	0.80 *	0.6 - 1.8	-	2.0 - 10.0

C.A. = Commercial Activated carbon

* From [25]

Density is an important characteristic of activated carbon as it is a measure of the amount of adsorbent, the carbon can hold per unit volume. Higher density also imparts more mechanical strength [11]. The density measured for the samples of activated carbon by using Equation (3), the higher density was for olive stone activated by both of CaCl₂ and KOH (0.41 & 0.38) g/ml respectively, the lower density obtained from banana peel activated by CaCl₂ (0.21 g/ml). In general, we find in Table (1) that the density of activated carbon by calcium chloride gave less density for all samples compared with carbon's activated by potassium hydroxide in the same activated conditions as a time of immersion and concentration of solutions, it has been largely due to the nature of the interactions resulting from the use of different stimulant solutions. Also all density data are less than the higher value of commercial activated carbon's density.

By the results shown in the Table (1), it find that the higher moisture content percentage obtained from activated carbon produced from banana peels which activated by KOH and orange peels activated by KOH (1.24,0.93) % respectively, followed by activated carbon of banana and orange peels activated with CaCl_2 (0.92,0.90) % respectively. The lower value of moisture content from both olive stone activated by KOH and CaCl_2 (0.21,0.20) % respectively. It is noticeable also when compared with commercial activated carbon that all the moisture data for different types of activated carbon except banana peels and orange peels which have exceeded the commercial moisture content due to the nature of raw materials.

Result of pore volume and porosity showed that orange peels activated carbon by CaCl_2 recorded higher pore volume and porosity (0.94ml, 31.44%) followed by Mixture activated with CaCl_2 too (0.91ml., 28.66%), the lower pore volume and porosity were taken from olive stone activated by KOH & CaCl_2 were (0.63ml., 21.20%) and (0.68ml., 22.66%), the density of activated carbon may be effect on pore volume and porosity of activated carbon, the higher density value showed less pore volume and porosity. Also all data located in the commercial range for pore volume.

High ash content showed in olive stone activated with KOH and CaCl_2 (3.96, 3.67)% and less content are in mixture KOH and CaCl_2 (2.38, 2.31)% . In general for all activated carbons, it appear that ash content are higher in carbons activated with KOH than CaCl_2 , it may be caused due to the nature of the reaction through the activation. These data showed less ranges if it compared with commercial activated carbon. High ash content in activated carbon is undesirable because it reduces the activated carbon produced. The ash content can be due to the inorganic constituent of the raw material [15].

Iodine Number

Iodine removal by activated carbon as indication of it is ability to adsorb low – molecular weight compounds. Carbon that removes a high percentage of iodine normally has a high surface area, those a greater degree of iodine adsorption reflects a higher surface area and a largely microspores structure[30]. The general

range of iodine number of a good quality of activated carbon is from (500–1200) mg/g [31].

Figure (5) shows that the orange peel and mixture activated with CaCl_2 had the higher value of iodine absorption (741, 735) mg/g higher than the same sample activated with KOH. Olive stone, walnut shell activated with both of KOH and CaCl_2 also banana peels activated with KOH are under the acceptable range of iodine number.

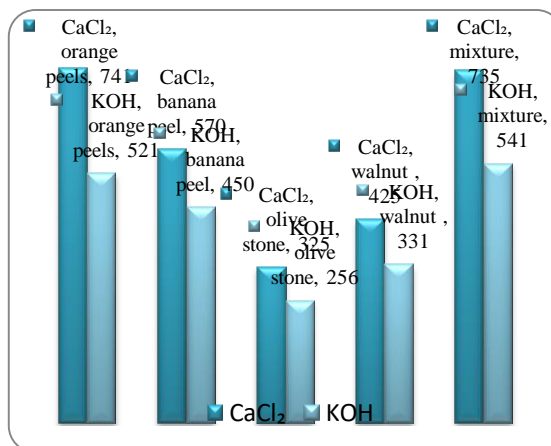


Fig. 5. Values of Iodine number for activated carbons

Removal %

This is very important test to know the degree of activation to removal dye by different activated carbons, Figure (6) refers to the superiority of orange peels activated carbons by CaCl_2 following with mixture activated with CaCl_2 also with removal % values (80.0 , 77.5)% respectively.

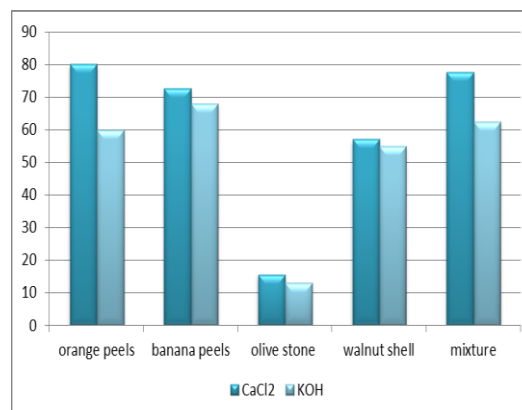


Fig. 6. Removal percent of methyl blue for activated carbons

method condition were (contact time:30min , agitation speed : 20ppm ,

dosage of active carbon : 0.1g ,
temperature : 27°C and initial pH : 6-7).

Table (2) shows that the low concentration of blue dye decrease from 20ppm to 4ppm in orange peels activated with CaCl_2 given a higher activity in removal percentage 80%. The methyl blue number of a good quality activated carbon is within the range of q_e (11.0 – 28.0)mg/g [31].

All activated carbons in the same range except olive stones activated by KOH and CaCl_2 . Adsorptive properties are directly linked with the porosity of activated carbon as the highly porous carbons can adsorb relatively large quantities of adsorbate[12]. So orange peels activated by CaCl_2 recorded porosity percent 31.44% with high removal percent of methyl blue.

Table 2. methyl blue number properties at constant initial concentration

Activated carbon	C_o mg/l	C_e mg/l	q_e mg/g	R %
Orange peels KOH	20	8.0	12.0	60.0
Orange peels CaCl_2	20	4.0	16.0	80.0
Banana peels KOH	20	6.5	13.5	68.0
Banana peels CaCl_2	20	5.5	14.5	72.5
Olive stone KOH	20	17.4	2.6	13.0
Olive stone CaCl_2	20	16.9	3.1	15.5
Walnut shell KOH	20	9.0	11.0	55.0
Walnut shell CaCl_2	20	8.6	11.4	57.0
Mixture KOH	20	7.5	12.5	62.5
Mixture CaCl_2	20	4.5	15.5	77.5

Conclusions

This study proved that waste food material (orange peels, banana peels, walnut shells and olive stones) can be used to produce activated carbons, which can be useful in recycling process. The carbons activated by CaCl_2 showed higher characterizes than carbons activated by KOH at same conditions. Activated carbon that prepared from orange peels and a mixture of waste food materials have higher iodine numbers (good surface area) and higher porosity (good removal percent) that can adsorb low – molecular weight compounds from contaminated water.

Environmental and economic side, the activated carbons from mixture are better to the possibility of raising its raw materials in a quantities sufficient for the production of a

short duration and a long of the year, noting that yields was the best.

Activated carbon from waste food matters have characteristics within acceptable range except olive stone activated carbons. Even though it's cheaply available and environmental friendly adsorbent can be used to economical waste water treatment.

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