

Research Article

Preparation and Characterization of Activated Carbon from Iraqi Khestawy Date Palm

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Received 16 December 2014; Revised 25 June 2015; Accepted 10 August 2015

Academic Editor: Mohammad A. Al-Ghouti

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This work includes a synthesis of three types of the activated carbon (AC) from three different positions from the same Iraqi Khestawy date palm. These three positions are the palm fronds (AC1), the date palm seeds (AC2), and the palm fiber (AC3). These three types of AC were synthesized by a physiochemical activation method using the same activator which was H_3PO_4 . These materials were investigated using different techniques such as Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). The adsorption activity of the synthesized AC samples was investigated by following the removal of both Bismarck brown G (BBG) and reactive yellow dye 145 (RY145). Both the kinetics of adsorption and the removal percentage of these dyes were investigated from the batch tests in this study. Different reaction parameters and conditions for adsorption processes were investigated. Also an investigation of both Langmuir and Freundlich adsorption isotherms was considered. The different physical properties of these materials were undertaken such as the point zero charges of the synthesized samples (PZCs), the percentage of humidity, and the adsorption capacity also being investigated. The activity of these materials in the removal of BBG from the aqueous solution was as follows: AC1 > AC2 > AC3.

1. Introduction

Activated carbon (AC) is a type of carbonaceous materials. It normally differs from elemental carbon by the oxidation of carbon atoms that are present on the inner and outer surfaces [1]. Recently, these materials have become an important research area due to their excellent properties. This involves their large specific areas, nontoxicity, high porosity, and the ability of their tunable surface to contain different functional groups. According to these properties, AC types have been widely used as adsorbent materials for a wide range of applications [2–4]. They can be used to adsorb materials with a high capacity from gas and liquid phases. AC is used in wastewater treatment, the purification of drinking water, gas phase adsorption in air pollution, and liquid phase adsorption. AC has a high applicability in adsorption which arises from its high porosity, rapid adsorption, and thermal stability. Until now, synthesized AC has been relatively expensive, so that the synthesis of a relatively cheap and commercially available AC for mass applications

seems to be an interesting challenge [5–8]. This aim can be approached via the use of inexpensive and widely available raw materials [9]. These AC raw precursors include using agricultural wastes as inexpensive raw materials with high carbon contents and low inorganic contents. Currently, an important application of AC is the removal of polluted dyes from industrial wastewater. It is well known that industrial effluents produced from different industrial and human activities can cause high levels of environmental pollution as these wastes are highly colored and contain large amounts of toxic organic species [10, 11]. Currently, more than 7×10^5 tons of wastewater containing dyes and pigments stuff is produced annually worldwide [12, 13]. The disposal of this colored water into the surrounding environment results in the contamination by these polluted materials of the soil and/or water. In terms of industrial wastewaters, an important class of polluted materials comprises dyes and pigments. This material can affect the environment for both toxicological and environmental reasons [14, 15]. However, these dyes are not biodegradable and are costly. On the other hand, the physical

and chemical processes involved in the dyes removal are more adequate in comparison with biodegradable method but they are very costly and cannot be used for mass applications. Consequently, adsorption processes seem to be the best method for the dyes removal. Out of the different adsorbents, activated carbons appear promising [16, 17]. However, AC is widely used as a standard adsorbent for the removal of a wide range of polluted dyes from colored wastewaters. Recently, activated carbon has been considered as an important type of adsorbents due to its high microporosity with a high surface area and nontoxicity [18]. Thus, it can be used as a good adsorbent for a wide range of adsorbates. This process can be performed by using AC in both powder and granular phases. For this reason, many researchers have been focused on the development of low cost adsorbent materials. These adsorbents involve sugarcane, bagasse, soy meal hull, orange peel, saw dust, palm ash, rice husk, rice straw, and flay ash [19, 20].

In this study, activated carbon has been synthesized by using a physiochemical activation method from different three positions from the same Khestawy Iraqi date palm using phosphoric acid as activators for two of the cases. It is well known that Iraq is famous globally for its agriculture of many classes of date palms and due to the fact that these precursors' raw materials are available widely. Therefore, this project can be developed for use in mass applications. The activity of AC was investigated by the adsorption of Bismarck brown G and reactive yellow dye 145 as a model of a polluted dye from their aqueous solutions.

2. Experimental Part

2.1. Adsorbate. The adsorbates that were used in this study were the Bismarck brown G ($C_{18}H_{20}N_8Cl_2$) and reactive yellow dye 145 ($C_{28}H_{20}ClN_9Na_4O_{16}S_5$). These dyes were used as adsorbed dye models in this study and they were used as provided without any further purification. Distilled water was used to prepare all the solutions and reagents in this work.

2.2. Synthesis of the Activated Carbon. Each of AC1, AC2, and AC3 was synthesized from raw materials that were collected from different parts of the same Iraqi Khestawy date palm (IKDP). For each sample, the raw materials were washed with hot distilled water several times to remove dust and other wastes. After that the samples were dried at $110^\circ C$ for two hours. Then the resultant dry materials were mixed with the desired activator at the appropriate ratio in a chemical activation process. This process involved heating materials at $700^\circ C$ in a graphite furnace for one hour in an inert atmosphere by flushing nitrogen prior to and during heating time. After that, the resultant samples were cooled to room temperature and the pH of the samples was adjusted around the value of 7 by washing with distilled water. Then the final products were dried at $110^\circ C$ for two hours to yield the final activated carbon. Photographic image of Iraqi Khestawy date palm is shown in Figure 1.

2.3. Adsorption Studies. The influence of different parameters on adsorption processes was investigated in this work.

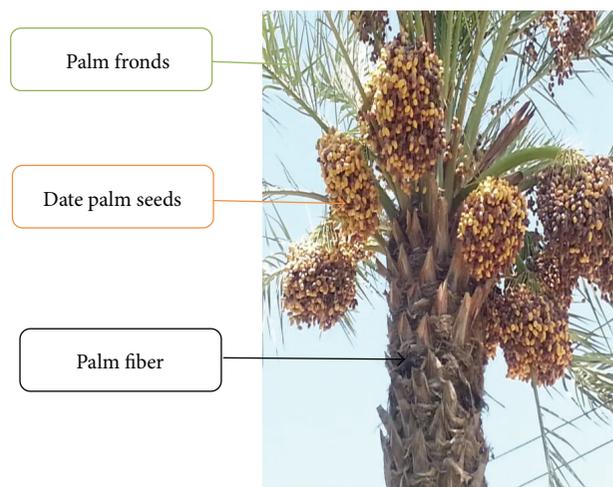


FIGURE 1: Diagrammatic representation of Iraqi Khestawy date palm structure.

Adsorption processes were performed in magnetic stirrer under air atmosphere. The processes of adsorption were studied using an initial concentration of 40 ppm of the used dyes in this study. The adsorbent was loaded in different concentrations in a solution of 100 mL of aqueous solutions of the dye. These masses were 0.01, 0.05, 0.10, 0.15, and 0.20 g. During run of adsorption processes and periodically 2 mL of the reaction mixture was withdrawn from the reaction mixture. Then, this volume of reaction mixture was centrifuged and the absorbance recorded at a wavelength of 448 and 416 nm for BBG and RY145, respectively. For both cases, the optical density of the supernatant liquid was recorded using Spectrometer photometer Shimadzu 1650 PC-UV-visible. The efficiency for dye removal from the aqueous suspension ($R\%$) was estimated using the following relationship [21–23]:

$$R\% = \frac{C_i - C_f}{C_i} \times 100. \quad (1)$$

From the above equation, C_i is the initial concentration of both BBG and RY145 dyes and C_f is the final concentration after one hour of the adsorption process. The term q refers to the amount of the dye adsorbed on the adsorbent in mg/g. Adsorption capacity at a given time of the adsorption is q_t and this can be calculated according to the following relationship:

$$q_t = \frac{(C_i - C_t) \times v}{m}. \quad (2)$$

The term C_t refers to the concentration of the used dyes as a function of time, v refers to the volume of solution, and m is the mass of the used adsorbent in this work.

2.4. Adsorption Capacity of the Synthesized Activated Carbon. Adsorption uptake capacity of each of AC1, AC2, and AC3 was determined by suspension of 0.1 g of each of AC1, AC2, and AC3 in an aqueous solution of methylene blue (MB) 100 mL, 20 ppm. The resultant mixture was suspended and

shacked in air atmosphere at room temperature for 24 hrs. This mixture then was centrifuged three times to insure complete removal of all fine particles of the AC. The absorbance of the resultant clear solution was recorded at 665 nm using UV-visible spectrophotometer. The adsorption capacity was calculated using a suitable calibration curve of standard solutions of MB. Then the adsorption uptake capacity of each of AC1, AC2, and AC3 was evaluated by comparing these concentrations with the initial concentration of MB (20 ppm) [24]. These results are shown in Table 2.

2.5. Humidity of the Synthesized Activated Carbon. Humidity content for the AC samples was estimated as a humidity percentage by subjecting 0.10 g of dried AC to the ambient lab atmospheric conditions for 24 hours. Then these samples were weighed accurately and dried in oven at 110°C for one hour. After drying, the samples were reweighed accurately and the humidity percentage was calculated from differences in the weights for the desired sample [25]. The results of humidity for each of AC1, AC2, and AC3 are summarized in Table 3.

2.6. The Point Zero Charge of the Activated Carbon Samples (PZCs). The isoelectric point of these materials was investigated as point zero charges. The PZCs values for all the AC samples were calculated according to the potentiometric titration [26]. In this method, 100 mL of 0.03 M KNO_3 was used as a blank solution and to this amount of the blank NaOH (1 mL of 1 M) was added. Then the formed mixture was titrated against HNO_3 (0.10 M). A mixture that is formed of 100 mL of KNO_3 with 0.10 g of the AC was stirred in air atmosphere for 24 hours. Then, 1.0 mL of NaOH was added and it was titrated with HNO_3 . The obtained results were plotted as a volume of the added acid against pH of the mixture. The obtained intersection point for each case was taken to be equal to the PZC of the investigated AC. These results are shown in Table 4.

2.7. Scanning Electron Microscopy (SEM). Surface morphology of the AC samples was investigated using Scanning Electron Microscope Inspect 550, Netherland. This equipment was operated at 25 kV and prior to run in SEM machine, the AC samples were dried and they were adhesive on carbon tape attached to aluminum-stubbed sputter coated with platinum. SEM images of AC1, AC2, and AC3 are shown in Figure 7.

2.8. Frontier Transform Infrared Spectroscopy (FTIR). Surface chemistry of the synthesized AC samples in this study was investigated using FTIR spectroscopy. FTIR spectra for all samples were recorded using Perkin Elmer Spectrophotometer. All measurements were undertaken in the range from 450 to 4000 cm^{-1} with a resolution of 1 cm^{-1} for each scan. To prepare samples for the run, all samples of the AC were grounded with KBr salt at a ratio roughly 1/50 of all samples. After that, these samples were made as pellets. The results of FTIR for AC1, AC2, and AC3 are shown in Figure 8.

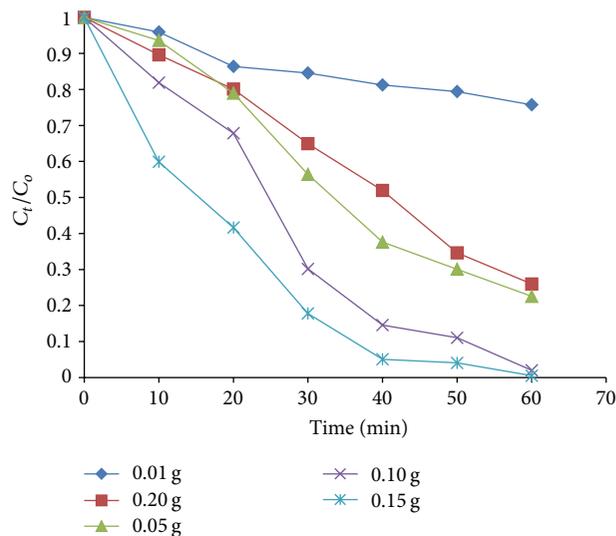


FIGURE 2: The effect of dosage and contact time of AC1 on the removal of BBG dye.

3. Results and Discussion

3.1. Effect of Dosage and Contact Time on the Removal of BBG. The effects of dosage and contact time for AC1 that was synthesized from palm fronds of IKDP on the removal of BBG from their aqueous solutions are shown in Figure 2. From this figure it can be seen that there was a remarkable increase in the removal of dye with increasing dosage of the used adsorbent (AC1). This probably arises from the increase in the adsorption capacity with increase of the concentration of AC1 in the mixture. The shaking time was one hour at 25°C for all experiments that were performed in this work in order to achieve a full equilibration for all doses of the AC types that were synthesized [27]. In terms of the effect of contact time on dye removal also there was a progressive increase in the percentage of dye removal with duration time. This can be attributed to increase uptake capacity of the adsorbent with increase of time and does not reach the maximum adsorption capacity in the range of one hour of adsorption process. These results are shown in Figure 2.

3.2. Study Effect of Type of the AC Samples on Dye Removal. In order to investigate the effect of type of the used AC in the removal of BBG from the aqueous solutions under the same conditions, a series of experiments were performed using 0.150 g of each of AC1, AC2, and AC3 at 25°C and under shaking for one hour. The obtained results showed that AC1 was more efficient than the other two types of the AC though all of them were synthesized from the same date palm and the same activator was used for all of them. The higher effectiveness of AC1 in relation to the other types probably arises from its relatively higher porosity that is confirmed with relatively higher humidity content for AC1 in comparison with the other two types. It is well known that the activity of catalyst in adsorption is increased with increase of

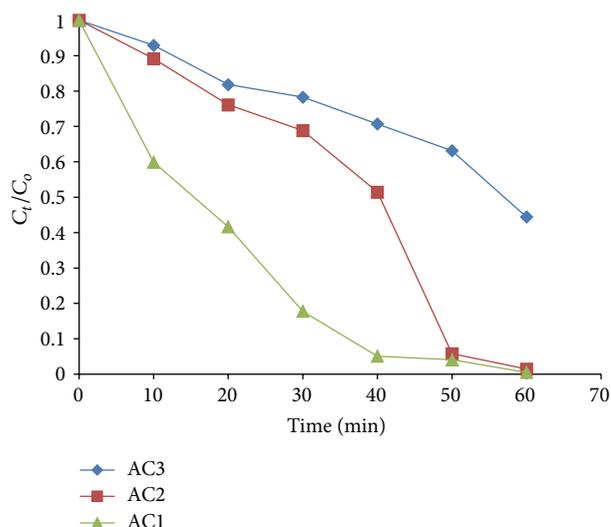


FIGURE 3: Comparison between the efficiency of AC1, AC2, and AC3 on the removal of BBG.

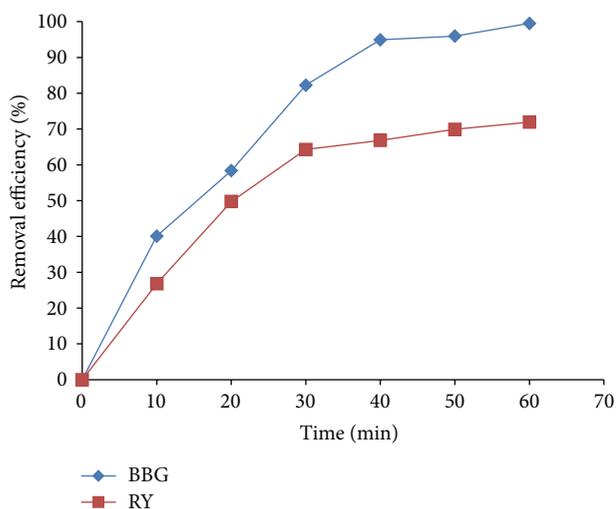


FIGURE 4: Comparison between removal efficiency of BBG and RY145 dyes on AC1.

its porosity. The results of BBG removal on these three types of AC are shown in Figure 3.

3.3. Effect of Dye Type on the Dye Removal. In order to investigate the effect of the used dye to be adsorbed on AC1, a series of experiments were performed using 40 ppm of each of BBG and RY145 under the same conditions, 40 ppm of each dye in 100 mL solution, and 0.15 grams of AC1 at 25 degrees. The results of these dyes removal over AC1 are shown in Figure 4. From these results it is clear that the removal of BBG was more efficient than that for RY145 applying the same adsorption conditions. Removal percentage of BBG approaches hundred percent after only one hour of adsorption duration which is much higher than that for RY145 (around 65%). This result can be attributed to the effect of diffusion limitation; this factor can have an effect on the adsorption/desorption processes on the surface which

normally occur in liquid phase reactions. Reactive yellow dye 145 has bulky structure in comparison with BBG that has a small structure. For this reason BBG is not affected significantly by diffusion limitation as in RY145. However, diffusion limitation factor can have an effect on the rate of the processes that occur in heterogeneous processes in liquid phase and does not significantly affect gas phase processes.

3.4. Adsorption Isotherms. Study of adsorption isotherms was investigated using both Langmuir and Freundlich isotherms for adsorption of BBG dye on the AC1 that was synthesized from IKDP. Langmuir adsorption isotherm is based on the formation of homogeneous monolayer on surface of the adsorbent [28]. For this type of homogeneous adsorption, all adsorption sites are considered to be energetically equivalent. For the physical adsorption, Freundlich equation is applied to follow adsorption isotherm. Both Langmuir and Freundlich isotherms can be explained mathematically as summarized in the following relations [29, 30]:

$$\frac{1}{q_e} = \frac{1}{q_m} + \frac{1}{K_L q_m C_e} \quad (\text{Langmuir}) \quad (3)$$

$$\log q_e = \log K_F + \frac{1}{n \log C_e} \quad (\text{Freundlich}).$$

The term q_e refers to the amount of adsorbate on the adsorbent in mg/g, or it represents concentration of the BBG on the AC1, and q_m refers to concentration of the BBG in the solution in mg/g; it represents capacity of the monolayer adsorption of the adsorbed dye. The term K_L refers to Langmuir adsorption constant in L/mg. The term C_e refers to concentration of dye in the equilibrium in mg/L and both K_f and $1/n$ are the constants of Freundlich adsorption isotherm.

As mentioned above, maximum adsorption capacity for the AC1 can be obtained using Langmuir equation. In this context, reaction conditions that were applied in this part were as follows: temperature was 25°C, pH 5.5, and the initial concentration of BBG was 40 ppm. The amount of the used AC1 ranged from 0.05 to 0.20 g with increasing rate of 0.05 g for each dose. The obtained results for the isotherms constants and for the R^2 are summarized in Table 1. These results are represented graphically in Figures 5 and 6 as $1/q_e$ against $1/C_e$. These results were more fitted with Freundlich isotherm. This arises from a high value of correlation coefficient for these results.

3.5. Uptake Capacity of the Synthesized Activated Carbon. The uptake adsorption capacity of AC1, AC2, and AC3 was investigated by following the adsorption of MB from the aqueous solution. The results of uptake MB by AC samples are presented in Table 2 as mg/g. From the obtained results it is clear that these types of AC showed high adsorption capacity and this indicated that these materials have a high porosity in their structures [31]. High adsorption capacity of these types of AC enables them to adsorb materials with high efficiency. The ability of adsorption is mainly dependent on the pores structure and the pores volume.

TABLE 1: Langmuir and Freundlich isotherm constants.

Isotherms	Constants/correlation coefficients	Values
Langmuir	R^2	0.9508
	q_m	65.7894
	K_L	0.49673
Freundlich	R^2	0.6407
	K_F	21.9280
	N	2.3798

TABLE 2: Adsorption capacity for the synthesized AC by MB adsorption.

Type of AC	Uptake capacity of AC (mg/g)
AC1	199.8
AC2	199.4
AC3	198.8

TABLE 3: The percentage of moisture content for AC samples.

Type of AC	Humidity%
AC1	48
AC2	46
AC3	45

TABLE 4: PZC for AC1, AC2, and AC3 samples synthesized from IKDP.

Type of AC	pH of AC
AC1	8.25 ± 0.10
AC2	8.62 ± 0.12
AC3	8.45 ± 0.15

3.6. Humidity of the Synthesized Activated Carbon. The humidity content of the synthesized AC arises from the ability of AC to absorb moisture into the porous structure when it is subjected to humid ambient conditions. However, from the obtained results in this study it was found that there is a relatively high content of humidity for all AC types in this work. So these materials can be considered as good adsorbents for many types of adsorbate and can be used in different applications as in analytical and chromatographic methods and industrial and environmental applications [32, 33]. The percentages of humidity content for AC1, AC2, and AC3 samples are presented in Table 3.

3.7. The Point Zero Charges of the Activated Carbon (PZC). The PZC of the AC samples synthesized from three different positions of IKDP was estimated according to potentiometric method [26]. The results of the PZCs for these materials are presented in Table 4. From these results it is clear that all the samples showed weak alkaline pH values ranging from 8.25 to 8.62. However, the lowest pH value was assigned to the AC1 and the highest value was assigned to the AC2 sample. Generally, these values of PZCs are dependent on the presence of proton and hydroxyl groups on the surface of AC samples. Thus, the presence of acidic and/or basic impurities

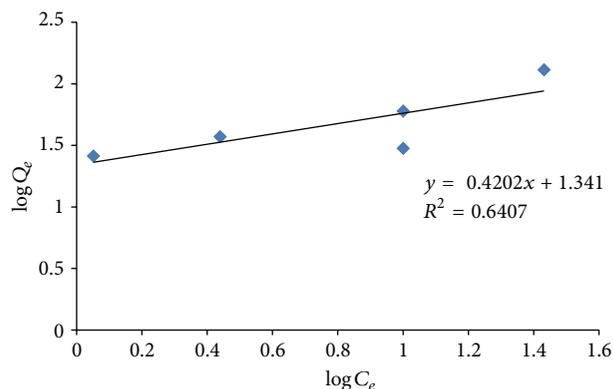


FIGURE 5: The linear Freundlich adsorption isotherms for BBG dye adsorption by the AC1.

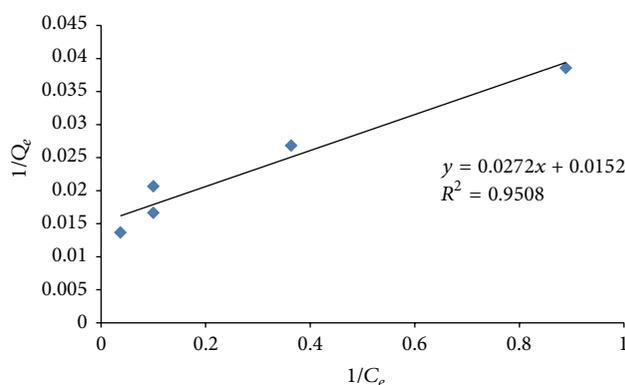


FIGURE 6: The linear Langmuir adsorption isotherms for BBG dye adsorption by the AC1.

on the surface of AC samples can have an effect on the value of the PZCs of these materials.

3.8. Scanning Electron Microscopy (SEM). Morphology of the synthesized AC was studied using SEM technique. SEM images of these materials showed irregular distribution with heterogeneous morphology of the surface. These images showed the presence of pores and cavities for all activated carbon samples. High porosity and irregularity can be seen with samples of AC1 and AC2 more than AC3 samples. The increase in porosity and cavities for these materials results in the enhancement of adsorption capacity that enables these materials to adsorb high concentrations of the adsorbates and accordingly they can have high efficiency as adsorbent materials and they can be applied in many of the environmental and industrial applications. Morphological images of these types of AC samples are shown in Figure 7.

3.9. Fourier Transform Infrared Spectroscopies of AC Samples (FTIR). In general the FTIR spectra for all the samples of AC that we synthesized from IKDP were almost the same. FTIR spectra of the AC showed three main absorption bands in the region from 1400 to 1700 cm^{-1} . The band that appears around 1700 cm^{-1} is related to the stretching modes of C=O bonds.

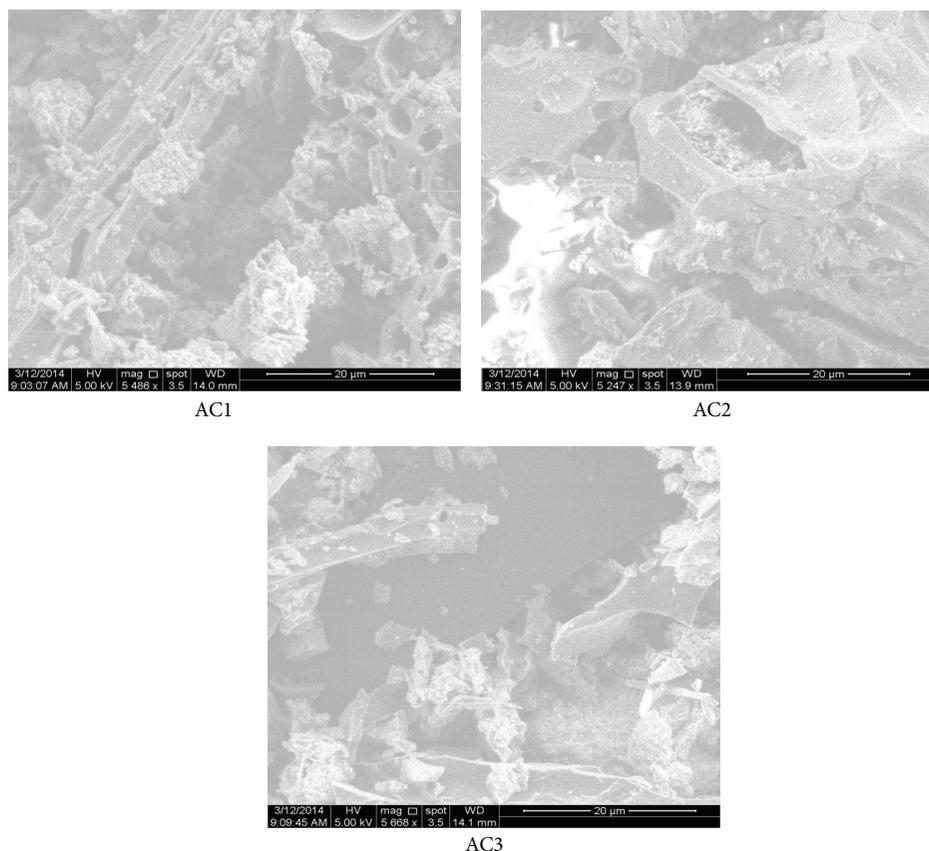


FIGURE 7: SEM images for the AC1, AC2, and AC3 samples synthesized from IKDP.

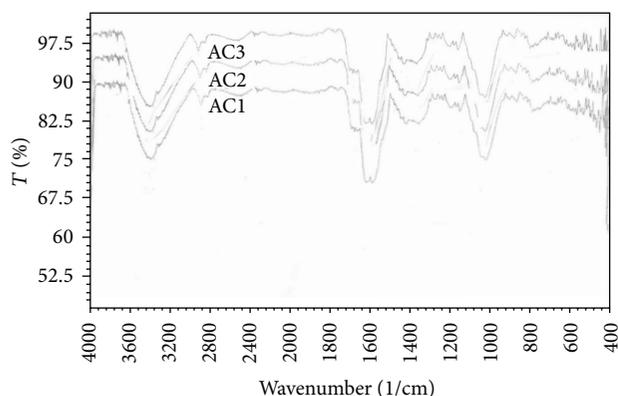


FIGURE 8: FTIR spectra for AC1, AC2, and AC3 samples synthesized from IKDP.

The band that appears around 1425 cm^{-1} can be assigned to the stretching vibrations of C-C bonds [34]. Strong band around 1600 cm^{-1} is related to the stretching vibrations of bonds of aromatic rings, those coupled with conjugated carbonyl groups on the surface [35]. The stretching modes of C=O bonds can be seen around $1100\text{--}1200\text{ cm}^{-1}$. In all samples of AC, there were weak peaks around 3000 cm^{-1} indicating the presence of the unsaturated alkynes C=C stretching modes; the peaks around $600\text{ to }850\text{ cm}^{-1}$ confirmed vibration of C-H bending mode [36]. Bands around

$3500\text{--}3600\text{ cm}^{-1}$ are assigned to the stretching modes which are related to OH groups. From these peaks, it can be seen that all these three samples of AC have almost the same oxygenated surface functional groups that are present on the surface of the synthesized AC. FTIR spectra for AC1, AC2, and AC3 are shown in Figure 8.

4. Conclusions

This study showed that an activated carbon can be prepared from raw widely available agricultural materials in Iraq such as date palms. This prepared AC can be used as an efficient adsorbent for wide range of materials. Also this study proved that AC1 that was derived from palm fronds of IKDP showed higher efficiency for removal of the BBG in comparison with AC2 and AC3. The synthesized AC samples showed high adsorption capacities, so that these AC samples can be used as good adsorbent materials. Also the synthesized materials showed relatively high humidity content which means that these materials have high porosity structure. These properties are considered as encouraging physical properties for these materials to be used as adsorbent material.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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