

## VALORIZATION OF DATE SEEDS FOR PREPARATION OF BIO ACTIVATED CARBON

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### ABSTRACT

The date industry currently produces thousands of millions of metric tons of waste per year. Hence, there is an urgent need to find suitable applications for this waste which are still largely unused. The aim of this research is to value date waste and its potential application for water treatment. Three types of activated charcoal were prepared from the seeds of dates. Different essays were done for each material prepared (moisture rate and ash, pH, concentration, mass, granulometry). The Materials obtained were characterized by different methods like FTIR and BET. The results obtained indicate that the materials prepared from date seeds are low-cost activated carbon with an effective surface similar to commercial activated carbon which has an important economic advantage through the valuation of these wastes.

**Keywords:** Palm date; Date seeds; Waste valorization; Activated carbon, Adsorption.

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## 1. INTRODUCTION

The date palm (*Phoenix dactylifera* L.) is considered the most important source of food for both humans in arid and semi-arid regions in the world [1]. The pericarp is an edible part and a seed is considered a by-product and waste [2]. On average, the mass of date seeds varies from 10 to 15% of the total date fruit mass [3]. The date palm is the fundamental element of the oasis ecosystem. It plays a vital role in economic production with the date and by-products (pasta, flour, syrup, vinegar, yeast, alcohol, confectionery, ...). Algeria is the world's second-largest with an annual production of 720 000 tones [4]. Palm dates where most of this production comes from the Arab World (>80%). This fruit has great importance from the nutritional and economic points of view [5]. Dates are one of the main crops in Algeria and represent a major source of income for the majority of the population in the south of Algeria. The fruits of the date palm are commonly consumed in many parts of the world especially in most Arabic countries. Date seeds, by-products of date fruit processing industries, have shown a potential application in the food, fermentation, chemical, and feed industries [6].

Date seeds showed many industrial applications such as a source of edible oil. And could be used in cosmetics, pharmaceuticals and a source of dietary fibers, these are also used for extracting oil for cosmetic and pharmaceutical purposes [7]. Date seeds are generally used as complementary feed materials for animals and poultry [8]. Date seeds are most effective in inhibiting the growth of bacteria as compared to antibiotics. Date seeds have the potential to be used as a supplement for antioxidants in nutraceutical, pharmaceutical, and medicinal products [1]. Date seeds for use as a fertilizer consisting of 70% of date palm wastes and date seeds 30% shrimp and crab shell wastes were reported as good fertilizer containing different elements for the plant growth like potassium, phosphorus and organic matter [9]. Replacement of the gas boiler by biomass that uses the date seed [10], as antidiabetic by date seeds drinking [11]. Date seeds as a water filter medium activated carbon are perhaps one of the most widely used adsorbents. It contained distinguished properties to the extensive surface area and internal porosity, as well as their developed surface structure. Date seeds have been recycled into adsorbent for removing of selected heavy metals from artificially contaminated aqueous solutions [12]. Modified date seeds, low cost, minimal pretreatment steps were effectively

employed as an adsorbent for remediating  $\text{Hg}^{2+}$  from aqueous media [13]. The waste utilization could provide economic gain to the farmers, industry, food security, environmental safety, and sustainability.

The objective of the present work was to conduct a preliminary analysis of the date seeds from *Phoenix dactylifera* L, to develop a low cost, efficient and environment-friendly process by the use of an agro-industrial waste of date palm for the development of value-added products.

## 2. EXPERIMENTAL

### 2.1. Materials

Date seeds were used as the raw material, charcoal Merck (Aktivkohle gekörnt etwa 2,5 mm reinst) ultra pur analytical grade, the chemicals used in this study are ortho-phosphoric acid  $\text{H}_3\text{PO}_4$  (85%), nitric acid  $\text{HNO}_3$  (65%), acetic acid  $\text{CH}_3\text{COOH}$  (99%), distilled water.

#### 2.1.1. Preparation of the raw material

The seeds of palm date were collected from the fruits of the Deglet-Nour dates (*Phoenix dactylifera* L.) from east south of Algeria, shown in (Figure.1), the core represents 7-30% of the weight of the date. After separation of the seeds (Figure.2) from the pulp, they were washed extensively with distilled water and put in an oven at 110 °C for 24 hours, from 10 kg of dates we obtained 1.367 kg of date seeds.



**Fig.1.** Palm Dates Fruits



**Fig.2.** Seeds from palm Dates

#### 2.1.2. Carbonization of the date seeds

The carbonization was done in a preheated electric oven (HERAEUS ELECTRIC furnace) at 600°C with for 3hours. The product obtained was crushed by a (RETSCH electric grinder) then sieved, with 1mm sieve. The obtained ground material was kept safe from air in tightly

closed bottles.

### **2.1.3. Chemical activation of date seeds**

Some crushed date seeds samples were chemically activated with 10N HNO<sub>3</sub> (Nitric acid) and other samples was activated with phosphoric acid H<sub>3</sub>PO<sub>4</sub> (40%) [14,15]. The collected date seeds were washed with distilled water and dried at 110° C for 24h, the seeds were ground, sieved, activated with HNO<sub>3</sub> (10 N) and calcined in an oven at 450° C for 1hour, then washed with distilled water to remove hydrosoluble impurities, the sample obtained dried at 110° C for 24h to obtain seeds activated charcoal.

### **2.1.4. Treatment of commercial activated charcoal**

About 100 g of commercial charcoal was added to 300ml of distilled water and the suspension was stirred for 30 minutes. After filtration, the charcoal was dried at 110°C for 24hours. Activated charcoal was crushed and sieved to 1mm in diameter.

### **2.1.5. Characterization of seeds activated carbon and commercial activated charcoal**

A comparative study between commercial activated charcoal (Merck) and activated carbon of date seeds was carried out to evaluate the efficiency of activated carbon prepared from date seeds as potential adsorbant, differents characterization method were used (acid-base character, moisture and ash content, IR spectroscopy and BET).

#### **1. Determination acid-base character of activated carbon**

About 10 g of each type of activated carbon from date seeds and commercial activated charcoal was added to 100 ml of distilled water, with continuous stirring for 30min after filtration the filtrate was tested by a pH meter. The results of the pH of commercial activated charcoal and date seeds activated carbon was shown in (Table.1).

**Table 1.** pH of commercial and date seeds Activated carbon

<b>Activate carbon</b>	<b>Commercial carbon</b>	<b>Date seeds carbon</b>
pH	6.43	6.75

#### **2. Determination of moisture content**

About 5g of each sample was placed in a crucible at 105°C for 1h in an oven and weighed

The Moisture Content of activated commercial charcoal and date seeds carbon was presented in (Table 2).

**Table 2.** Moisture Content of commercial and date seeds activated carbon

<b>Adsorbents</b>	<b>Moisture content %</b>
Commercial charcoal	1%
Date seeds	1%

### 3. Determination of ash content

The determination of the ash content was based on the destruction of all organic matter under the effect of high temperature. About 2g of commercial activated charcoal and seeds Activated carbon was put in a porcelain crucible and incinerated in an oven at  $550 \pm 5^\circ\text{C}$  for 8 hours, then put in a desiccator for 30minutes. The ash content is expressed as a percentage of the dry matter. The results of ash rates of commercial activated charcoal and date seeds activated carbon are shown in (Table.3).

**Table 3.** Ash rates of commercial and date seeds activated carbon

<b>Adsorbents</b>	<b>Ash %</b>
Commercial charcoal	3.5%
Date seeds	1.5%

The adsorption procedure was done by acetic acid ( $\text{CH}_3\text{COOH}$ ) on commercial activated charcoal and seeds activated carbon, the procedure comprises the following steps:

- 1-About 1g of activated carbon was put in contact with 50 ml of solution  $\text{CH}_3\text{COOH}$  (0.1M) under stirring for 30min.
- 2- Filtration.
- 3- Dosage of 10 ml of the filtrate in the presence of 2drop of phenolphthalein with NaOH (0.1M) until neutralization.

### 2.6. Optimization of the adsorption conditions

#### 2.6.1. Influence of the contact time

About 50 ml of acetic acid (0.1M) is mixed with 1g of each commercial activated charcoal

and date seeds activated carbon each one separately and stirred for different contact times ranging from 10 minutes to 60 minutes at 25°C.

### **2.6.2. Effect of activated carbon mass**

Different masses (1-10g) of activated carbon were put in 50 ml of acetic acid solution (0.1M) with stirring for 30 minutes at 25°C.

### **2.6.3. IR spectroscopy analysis**

The infrared analysis was performed on a spectrometer (SHIMADZU) FTIR-8201PC, the frequency range between 400 and 4000  $\text{cm}^{-1}$ . The powder is finely ground, sieved and then mixed with KBr (1/300 by weight).

### **2.6.4. BET isotherm and surface area**

Multiple point nitrogen adsorptions analysis (BET) was used to determine the surface area of Activated carbon from palm seeds. The adsorption and desorption isotherms of  $\text{N}_2$  at 77°K consist of determining the volume of liquid nitrogen necessary, to form a monolayer of this gas, on the surface of the carbon, in order to measure the specific surface. The adsorption isotherms were obtained using the ASAP2020 type analyzer according to the classic method of Brunauer Emmet and Teller or BET.

## **3. RESULTS AND DISCUSSION**

### **3.1. Characteristics acido-basic of commercial charcoal and seeds activated carbon**

From (Table.1), the activated commercial charcoal and seeds carbon samples were slightly acidic during the activation. The majority of acid groups formed on the surface of the activated carbon, are mainly carboxylic acid, lactone, phenol, and carbonyl functions, during their manufacture predominate on the surface of the pores.

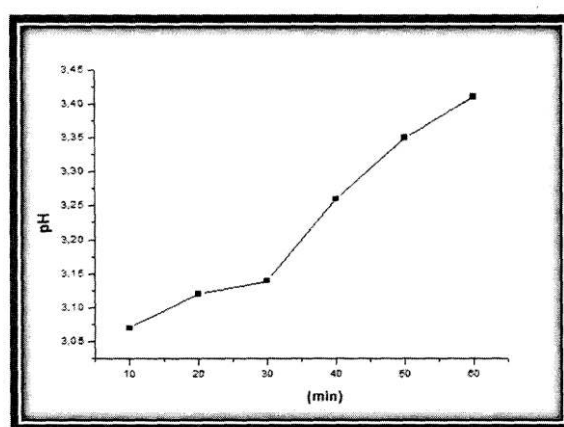
### **3.2. Moisture and Ash Rates**

The moisture content of humidity % and ash % of both date seeds and commercial activated carbon was given in (Table.2 & Table.3). The moisture values were less than 5 %, corresponds to the free water of hydration, the determination of ash% content was based on the destruction of any organic material under the effect of high temperature which was ( $500 \pm 25 \text{ C}^\circ$ ).The ash content was less than 10 %, these values were in the norms and can be indicative of the

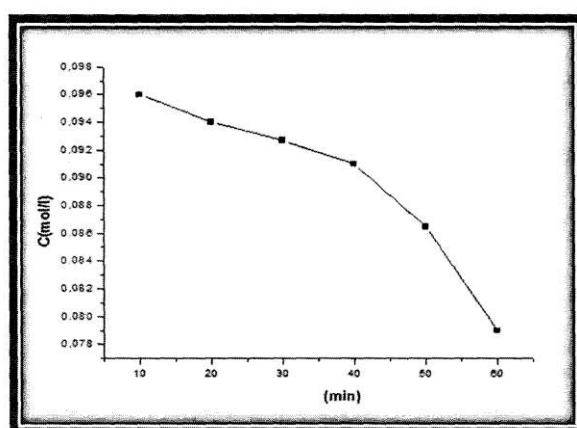
quality of the new adsorbent phase from the date seeds.

### 3.3. Effect of contact time on adsorption

The evolution of the adsorption process as a function of time shows that the new phase adsorbent has a capacity adsorption comparable to commercial activated charcoal. According to the results of (Figures 3,4,5 and 6), based on the variation of the pH. It was noted that the pH of the medium increases, as a function of contact time and the pollutant concentration decreases. Indicated the effectiveness of date seeds for acid removal from the water.

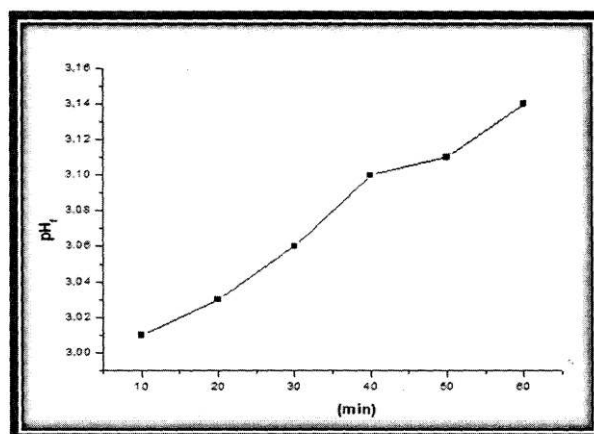


**Fig.3.** pH versus time variation for date seeds activated carbon

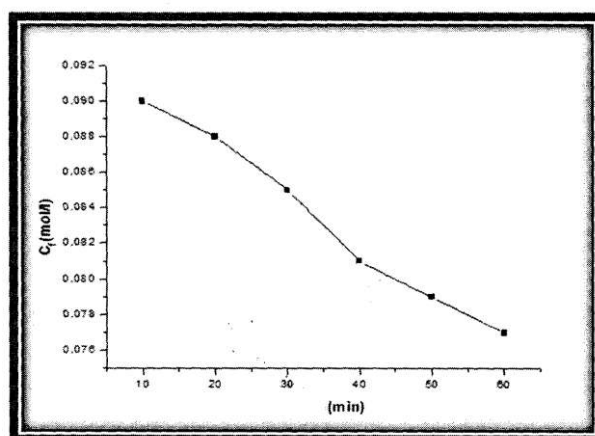


**Fig.4.** Variation of concentration as a function of time for date seeds Activated carbon





**Fig.5.** Variation of pH versus time for commercial activated charcoal



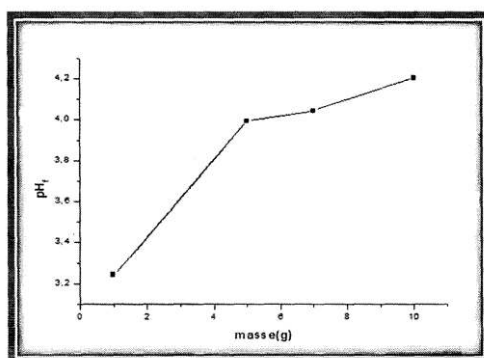
**Fig.6.** Concentration change as a function of time for commercial activated charcoal

### 3.4. Effect of activated carbon mass

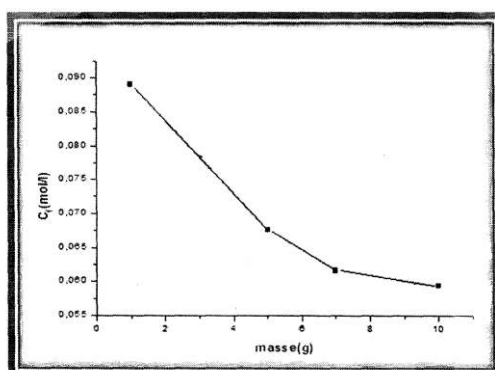
Based on the Figures (7,8,9, and 10), it was noticed that the pH of the date seeds activated carbon was slightly increased due to the formation of a new functional group contain N, O on the surface of seeds. These functional groups increased the adsorption capacity of the date seeds activated carbon by augmentation of adsorbent mass. The activation of date seeds by phosphoric acid produces activated carbon with larger micro-pores which promote higher molecular mass pollutant adsorption [16]. Many researchers [17-19] used phosphoric acid to increase the activated carbon micro-pores and raise the surface area of adsorbents prepared from different materials. The activated carbon has usually acid functions, which allow a duple



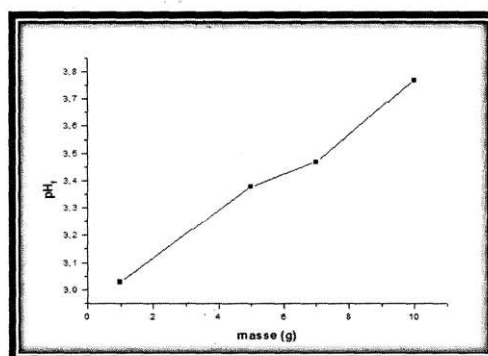
reactivity for both anionic and cationic adsorbates [20]. In our study, the samples of date seeds activated by  $\text{HNO}_3$  have improved the adsorption rate of acetic acid compared to samples activated by  $\text{H}_3\text{PO}_4$ . Due to the small molecular mass of  $\text{CH}_3\text{COOH}$  adsorbed at date seeds activated with  $\text{HNO}_3$ , the adsorption was more significant in this phase. We suggest that the activated date seeds with  $\text{HNO}_3$  have better adsorption for the low molecular mass pollutants as a result of the increases of the functional groups at the surface of activated date seeds.



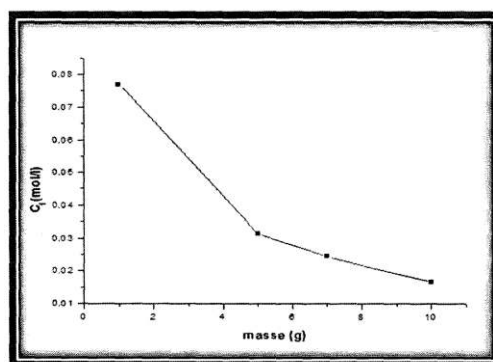
**Fig.7.** Variation of pH versus masses for date seeds activated carbon with  $\text{HNO}_3$



**Fig.8.** Variation of concentration versus masses of date seeds activated carbon with  $\text{HNO}_3$



**Fig.9.** pH variation versus mass of commercial activated charcoal



**Fig.10.** concentration variation versus mass of commercial activated charcoal

### 3.5. IR spectroscopic analysis

The IR spectroscopy technique is an essential method for the identification of raw and activated carbon constituents. The infrared analysis spectra of the raw material and the prepared activated carbon are shown in the figures below. The analysis was performed over a wavelength range of  $400\text{-}4000\text{cm}^{-1}$ . The presence of the hydroxyl groups of the phenolic function and the carboxylic function offers an acidic character on the surface adsorbents. While the presence of the carbonyl functions gives basic character to the surface of the adsorbents. We observed that the activated date seeds studied are similar. The only difference lies in the intensity of the peaks, which shows the influence of the treatment on the characteristics of the activated carbon obtained. The main recorded bands are located in the region  $3436\text{-}3444\text{cm}^{-1}$ ,  $2922\text{-}2927\text{cm}^{-1}$ ,  $2362\text{cm}^{-1}$ ,  $1620\text{-}1635\text{cm}^{-1}$ ,  $1449\text{cm}^{-1}$ . The adsorption bands observed between  $3436$  and  $3444\text{cm}^{-1}$  are attributed to the presence and vibration of free hydroxyl groups (OH) [21].

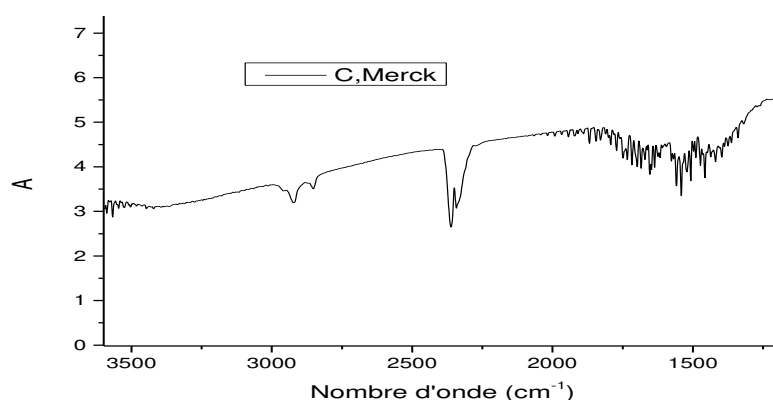
The vibration at  $3444\text{cm}^{-1}$  (Figure.12) becomes more intense after the adsorption of the  $\text{CH}_3\text{COOH}$  (Figures 13, 14). The bands observed between  $2922\text{-}2923\text{cm}^{-1}$  (Figure.11 and Figure.12) correspond to the vibration of the symmetrical and asymmetrical valence of the C-H bonds present in the alkyl groups [22]. There was also the presence of the peak at  $2927\text{cm}^{-1}$ , this indicated the presence of C-H in the raw date seeds (Figure.12) and the activated date seeds (Figures 13,14). The bands observed in the interval  $2362\text{cm}^{-1}$  (Figure.11) can be attributed to the vibration triple bond of the nitriles  $\text{C}\equiv\text{N}$  [23]. The bands observed in the interval  $1620\text{-}1635\text{cm}^{-1}$  can be attributed to the vibration of the  $\text{C}=\text{C}$  bonds of the aromatics [21,22,24]. Other bands also present in the activated date seeds by  $\text{HNO}_3$  ( $1627\text{cm}^{-1}$ ) and by  $\text{H}_3\text{PO}_4$

( $1635\text{cm}^{-1}$ ). The bands at  $1149\text{ cm}^{-1}$  are attributed to the vibration of the C-O bonds.

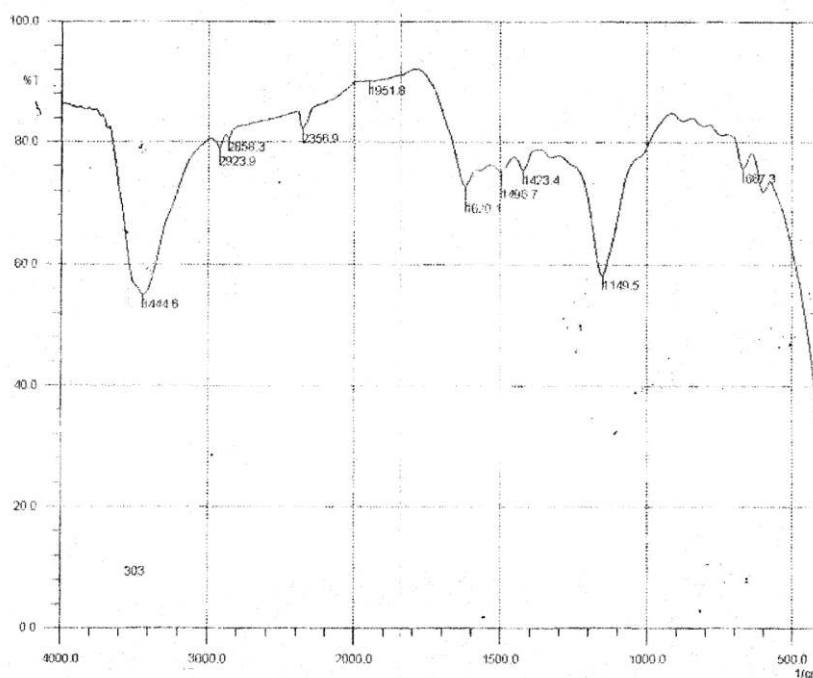
The spectra in Figures (12, 13, and 14) also show broadband at  $1149.5\text{ cm}^{-1}$  attributed to the C-O elongation [24]. This band was also characteristic of phosphorus and phosphocarbon compounds present in the activated carbon of date seeds [25]. The band at  $972\text{cm}^{-1}$  that observed clearly only in the spectrum (Figure.14), that of  $\text{H}_3\text{PO}_4$  activated carbon could correspond to the vibrations of the elongation of P-O-C (aliphatic), of asymmetric elongation of P-O-C (aromatics), elongation of P- O in P = OOH. The P-O functional group was present due to the use of phosphoric acid in the process of preparing the activated. The bands at  $667\text{ cm}^{-1}$  (Figures 12, 14) and  $568\text{ cm}^{-1}$  (Figure. 13) are due to the C-H of the aromatic rings.

IR of date seeds activated carbon and Commercial activated charcoal confirms the existence of the following functions: O-H, C=C, C-H, C=O, and, C-O. The comparative study between native date seeds and those activated by  $\text{HNO}_3$  and  $\text{H}_3\text{PO}_4$  shows a shift in the values of the peaks and a change in the intensity, especially the O-H vibrations.

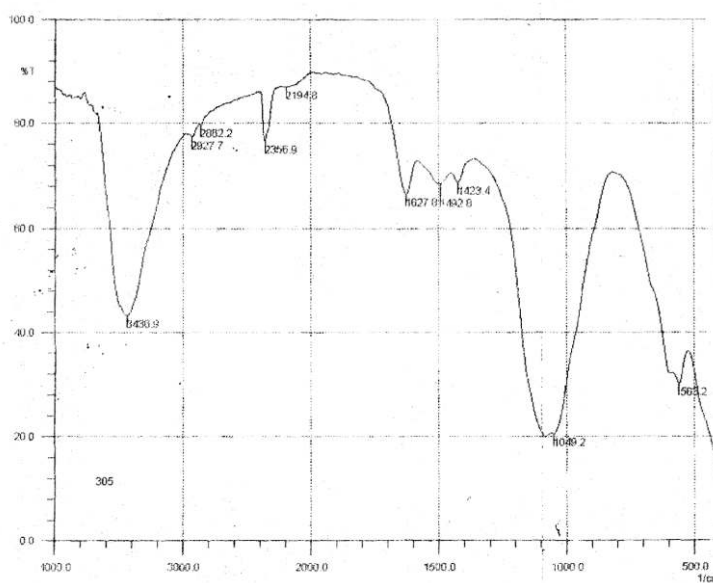
From the FTIR results, it is shown that the appearance of functional groups in the date seeds activated by  $\text{H}_3\text{PO}_4$  occurred due to the impregnation process. The infrared spectra are marked by several absorption bands, indicating the complexity of the surface chemical structure of the activated carbon studied. The results indicated that the studied activated carbon exhibit different functional groups such as hydroxyl, carboxyl, and carbonyl groups, which may be potential sites of adsorption.



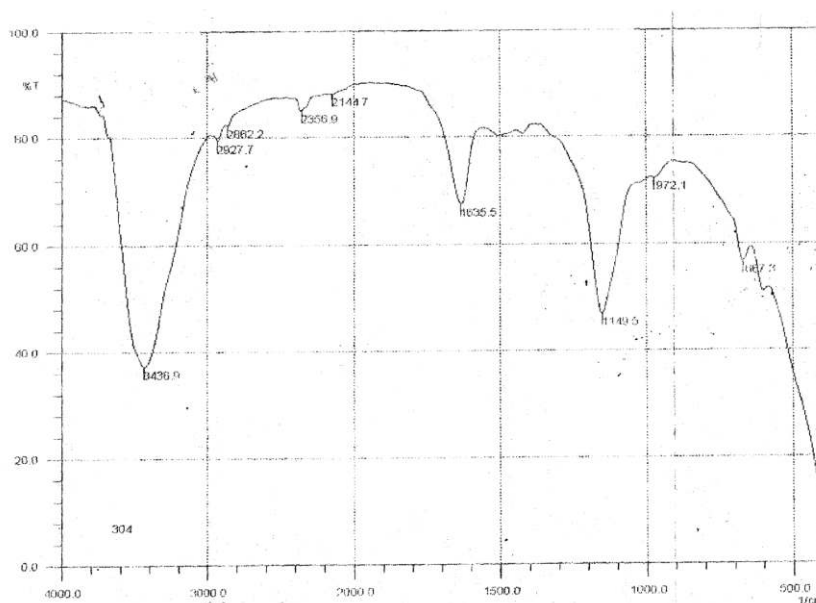
**Fig.11.** IR spectrum of Commercial charcoal activated granular (Merck)



**Fig.12.** IR spectrum of raw date seeds before activation



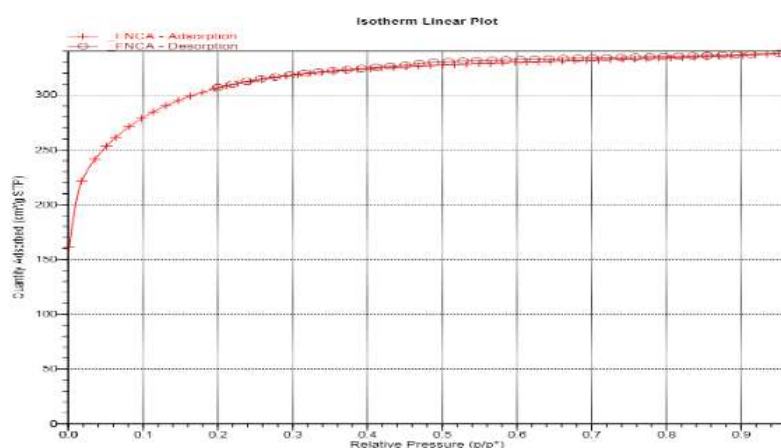
**Fig.13.** I.R spectrum of seeds dates activated with HNO<sub>3</sub> after adsorption



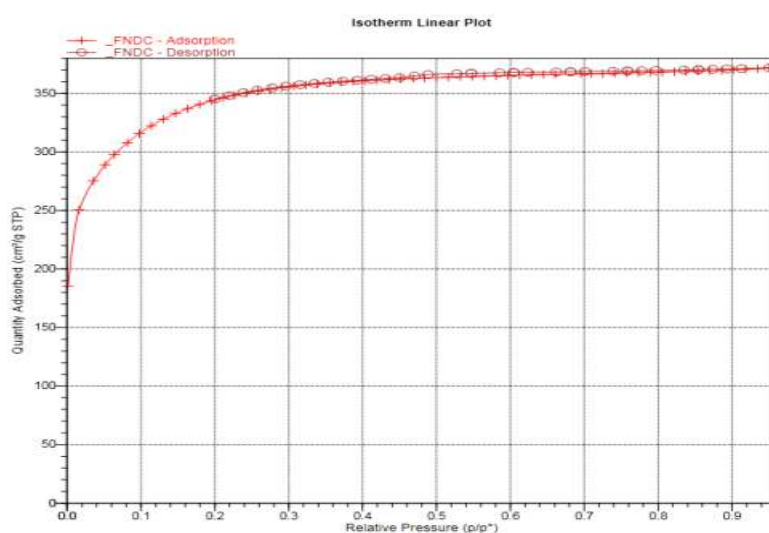
**Fig.14.** IR spectrum of seeds dates activated with  $H_3PO_4$  after adsorption

### 3.6. BET isotherm surface area measurement

According to the results of the adsorption and desorption of nitrogen on chemically activated carbon from seeds (Figure.15) and the commercial activated charcoal (Figure.16), the isotherms were of type I isotherm, also known as langmuir isotherm characterized the activated carbon microporous [26,27]. The medium contains only saturated micropores for low values of  $p/p^0$ . However, no adsorption occurs at higher values that would fill pores [28]. The isotherm showed that the adsorption was reversible and the amount of gas adsorbed was approaching the limiting value as  $P/P_0 \rightarrow 1$ . This indicated that the date activated seeds consisted of micropores with a micropores filling mechanism of activated carbon from seeds [29]. The specific surface area of the date's seeds prepared by chemical activation with phosphoric acid was  $942,1481m^2/g$  it was comparable to the specific surface of commercial charcoal which was  $1105,1301m^2/g$ .



**Fig.15.** Isotherm of the adsorption and the desorption of N<sub>2</sub> on the activated carbon of the Date seeds activated by H<sub>3</sub>PO<sub>4</sub>



**Fig.16.** Isotherm of the adsorption and the desorption of N<sub>2</sub> on charcoal activated by H<sub>3</sub>PO<sub>4</sub>

#### 4. CONCLUSION

Date seeds are waste material from the date palm, show a high potential to be used in the extraction of the bioactive compounds and biomass production. Date palm seeds can be a suitable precursor for the preparation of activated carbon that has been explored. Phosphoric acid and Nitric acid were used as the activating agent, from this study, there are the possible

uses of these waste materials as promising green adsorbents, for removing unwanted materials from water and wastewater. Activated carbon prepared from waste palm materials, available locally, efficient and environment-friendly. In order to find an alternative to commercial activated carbon to add considerable economic value, and provide a potentially inexpensive of activated carbon for water treatment.

### **CONFLICT OF INTEREST STATEMENT**

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

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