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Activated Carbon Prepared from Date Pits Using Microwave Heating

Benouali Djillali[1,2], Benhammadi Samia[3], Kherici Samira[1], Berrebah Alioua Amel[4]

[1]Department of physical-chemistry, USTO-MB, Oran

[2,4]Laboratory of toxicology environment and health (LATES), USTO-MB, Oran

[3]Department of chemical engineering, USTO-MB, Oran

BP 1505 El-Menaouer 31000, Oran -Algeria.

ABSTRACT: Activated carbon (AC) adsorption remains one of the most technically efficient water treatment processes. However, its high preparation cost, mainly due to energy-intensive methods, has led to research on developing efficient and cost-effective preparation methods using less expensive materials such as agricultural waste. The objective of our study was to use date pits for the preparation of AC that can be used for water treatment (Adsorption and elimination of dyes). For the activation process, a microwave radiation under steam method was employed. The AC prepared from date pits /H_3PO_4/carbonization 400°C using the steam-microwave radiation activation technique (radiation time: 15 minutes, power: 700W) demonstrated promising results. The adsorption tests show 80% removal rate of the reactive dye (Drimaren ROT CL-5B GR) from water. This study highlights the potential of date pits as a viable source for producing AC from bio-material. The obtained product was analysed by FTIR and MEB.

KEYWORDS: Water treatment, dyes, date pits, activated carbon, adsorption, microwave heating.

I. INTRODUCTION

AC has been produced from fossil coals and wood for a very long time [1]. However, there are currently a considerable number of studies on the preparation of AC from agricultural wastes. Different agricultural wastes and by-products have been studied depending on their local availability. And it is known that the properties and characteristics of AC, such as porosity and surface area, are essentially determined by the raw material used and the activation method. Therefore, using inexpensive agricultural raw materials poses a challenge in reducing the price of AC and improving its properties [2-7]. Moreover, when transforming waste that can have a negative environmental impact into AC, the primary aim must be its elimination [8,9]. Yet, very little agricultural waste has reached the mass production stage.

The type of process used for the manufacture of AC carbon plays a vital role in the porous structure of the produced material. The first step in AC involves drying the raw material (precursor) and carbonization, often at a temperature above 400°C. As a second step, physical activation, aims at develop of the porous system. It occurs at high temperatures of 800-1000°C in the presence of CO_2 or steam. In some works, traditional heating has been replaced by microwave radiation [10-14]. Heating assisted by microwave radiation directly heats the particle interior, inducing rapid volumetric heating. Also microwave radiation it's an alternative that requires more development, for example microwave radiation coupled with ultrasonic energy activation has been introduced by [15].

At first AC were produced by zinc chloride ($ZnCl_2$) as a chemical activation process. Nowadays, phosphoric acid (H_3PO_4) has largely replaced the zinc chloride process. However, mixed activation (physical and chemical) is often employed, preferably in a single step.

The objective of our study is to use date pits to prepare effective AC for water treatment. Some works in literature have used date pits as precursor using traditional heating [16-18]. The produced activated carbon can be utilized for example in the adsorption process for dyes and phenol removal. The variety of date palm used in this study for the production of AC is called “Ghars”. It is widely distributed throughout the Lower Sahara in Algeria, and statistics show significant varietal diversity, with the “Ghars” variety and the “Deglet Nour” variety being the most predominant. The “Ghars” variety's pits are elongated, brown in colour, smooth in surface, and straight in shape. On average, they have dimensions of 2.5 / cm [19]. The pits primarily consist of cellulose (33-36, 26%), proteins, and lipids (18-20%), as well as sugar (10-12%).Traditionally, these pits are used as fuel in ovens and as food for animals. They can also serve as a coffee substitute due to their pleasant flavour and aroma. Additionally, oils extracted from date pits are used for human consumption and soap making.

II. MATERIALS AND METHODS

In the present study, date pits (Figure 1) are used as aprecursor for the preparing of activated carbon using the chemical activation method and microwave radiation.

Figure 1: 1- Date pits variety "Ghars" (Algerian Low Sahara)
2- Crushed date pits: 0.5 <D <3mm

The date pits are washed to separate the pulp, and then they are soaked in distilled water for 20 minutes. This process is repeated three times. Afterward, the date pits are air-dried and then further dried in an oven at 105°C for 20 minutes. The dried date pits are then crushed, ground, and sieved using a mechanical sieve (Figure 1).The ground date pits undergo carbonization by heating them on a benzene burner until no more black smoke is released. After the pre-treatment, the carbonization samples of the precursor are heated in a muffle furnace at a temperature of 400°C for one hour and allowed to cool. For activation, the carbonized material is impregnated with a phosphoric acid solution (70%) at room temperature in a ratio of 1- 5 for 3 hours. After filtration, the carbon is washed multiple times with distilled water until a neutral pH is achieved. It is then subjected to microwave radiation under steam (Figure 2). A system comprising two stacked multi-mode microwave apparatuses is used. One apparatus serves as a steam reactor at high temperature, while the second is used for activation (radiation time: 15 minutes, power: 700W). Vacuum cooling is used to prevent steam from escaping at the junction between the carbon-containing tube and the refrigerant. The adsorption tests are conducted in batches using 30 ml of Drimaren ROT CL-5B GR dye solution in contact with different quantities of activated carbon ranging equal to 10 - 80 ppm. The residual dye concentration (λ = 540 nm) was measured to monitor the adsorption kinetics on the studied samples. After adsorption, the samples are filtered under vacuum using a 0.45µm porosity membrane.

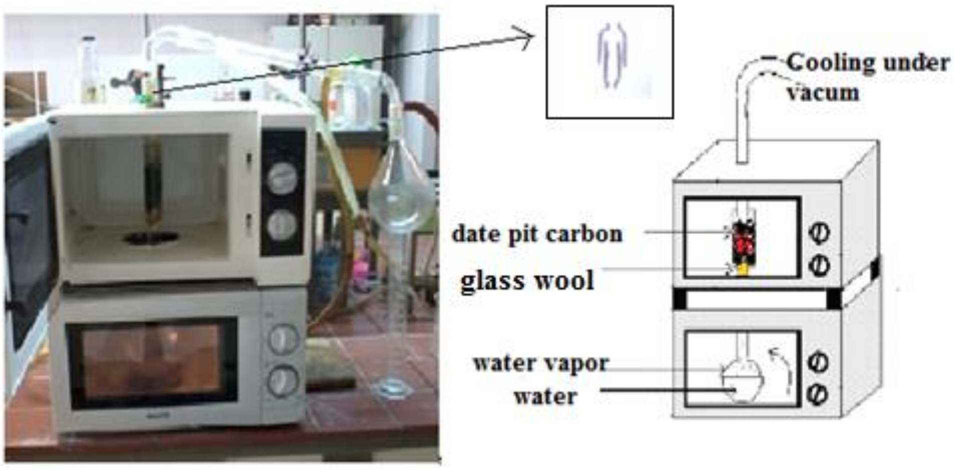

Figure 2: Devise for carbon activation assisted by microwave irradiation

III. RESULTS AND DISCUSSION

Following cleaning, the date pits were dried in oven and then carbonized. The weight loss observed is about 20% and 75% after drying and carbonization respectively. After sieving the obtained carbon, we obtained particles with five different diameters (Figure 3). The first weight loss is due to the release of surface bound water and moisture release. The second weight loss (75 %) is attributed mainly to the decomposition of cellulose and other low molecular weight organic compounds.

After microwave radiation, the decomposition of cellulose can occur. The radiation absorption is effective within an exposure time range of 10 to 15 minutes at a low power range of 500-700 W. Heating the carbon precursors without chemical impregnation may not be effective, so the activation agents act as the primary microwave absorber during the activation stage. At radiation intensities greater than 700W, the rate of new pore formation and the beginning of pore destruction may decrease. In traditional heating, structure transformation processes initiate around 400°C, resulting in an increase in carbon content in the form of condensed aromatic rings. However, the rise of microwave radiation's instantaneous temperature easily triggers the rearrangement of organic matter, leading to a porous structure without prolonged destruction of the material.

Figure 3: Date pits crushed and carbonized.(D= diameters)

In case of raw material (Figure 4a), the peak observed at 3344 cm^{-1} (FTIR analysis) is attributed to the hydroxyl groups (O – H) on the surface. At 2921 cm^{-1} and 2852 cm^{-1} we observe the asymmetric and symmetric stretching of the methylene (C – H) group, further enhancing our understanding of the material's composition. The presence of the two characteristic bands for $–CH_3$ and $–CH_2$– (2921 and 2852 cm^{-1}) suggests the existence of aliphatic groups, with their content decreasing after microwave radiation (Figure 4d). Also, a decrease in intensity for methylene groups at 1412 cm^{-1} (CH_2 stretch) is observed and compared to the raw material (date pits). The bands located around 1560 cm^{-1} corresponds to the vibrations of the aromatic rings.

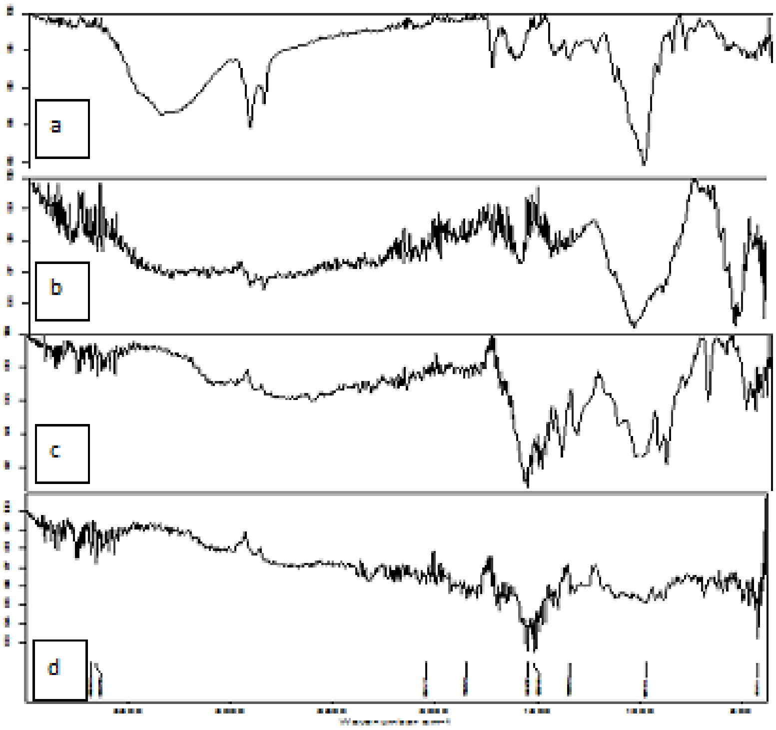

Figure 4: FTIR spectra : a- Raw date pits ;b- Carbonized date pits c- Commercial AC; d- Date pits AC

These bands show reduced intensities with microwave radiation. Peaks at 1507 cm^{-1} are attributed to primary amine groups, the N – H bend, is due to the C – N stretch of phenols. The N – H bend of the secondary amines is observed at 1541 cm^{-1}.

The analysis conducted using the Scanning Electron Microscope (SEM) revealed a heterogeneous surface structure of the analysed sample. Additionally, it clearly depicts the presence of microporous network (Figure 5) as precursor materials with high cellulose yields AC with predominantly microporous structure.

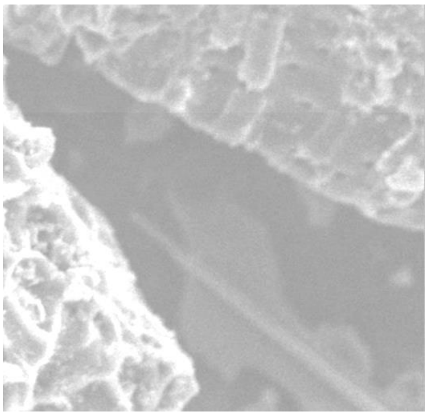

Figure 5: SEM of activated carbon from date pits

A visible scan of a solution (40 ppm) shows that the reactive dye Drimaren ROT CL-5B GR has a maximum absorption at 540 nm (Figure 6).

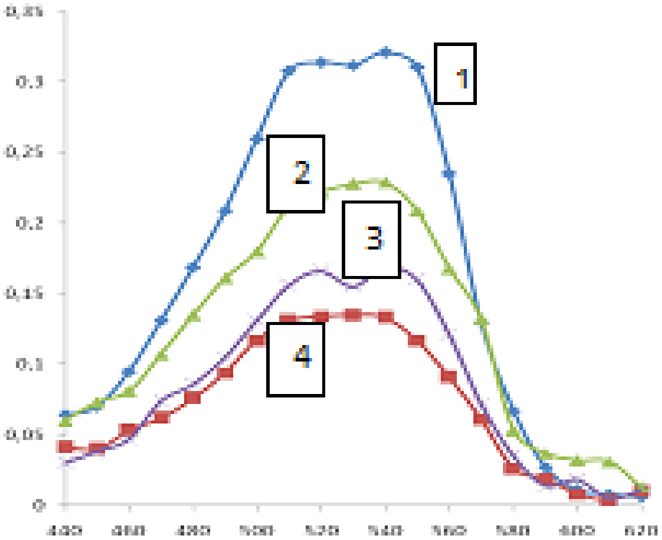

Figure 6 : Absorption Spectra of dye solution (40 ppm): 1 – non treated solution; 2- treated with commercial AC; 3- treated with date pits carbonized at 400 °C and impregnated with H_3PO_4; 4- date pits AC activated with microwave irradiation.

Figure 6 shows a highly positive effect of microwave radiation on the adsorption of reactive dyes in aqueous solution.

IV. CONCLUSION

In conclusion, our study demonstrated the transformation of agricultural waste (date pits) into high-value product: Activated carbon. The carbonized date pits were effectively activated using a phosphoric acid solution (70%) and microwave radiation under water vapour (15 min of radiation 600W intensity). To carry out this activation method, we modified and adapted two multi-mode microwaves, with one apparatus generating high-temperature steam and the other used for carbon activation (radiation). The prepared activated carbon exhibited a good removal rate of 80% for the reactive dye Drimaren ROT CL-5B GR, without the need for pH adjustment. This finding highlights the effectiveness and potential of this activation method. By utilizing agricultural waste such as date pits, we contribute to waste valorization and provide a sustainable and cost-effective solution for water treatment applications.

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