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Optimization of the Porosity of Biochar Produced from Date Stones using Full Factorial Design

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Abstract:- Biochar is a low-cost material that has found wide applications as an adsorbent for the removal of various pollutants. The aim of this study is to evaluate the porosity of biochar derived from agricultural solid wastes such as date stones. For this reason, this research focused on the optimization of the factors influencing the preparation of biochar by pyrolysis using experimental designs. The influence of two parameters, temperature (400-550°C) and pyrolysis time (1-3h) with a heating rate of 10°C/min, on biochar preparation was studied. The two factors were taken into account in the two-level complete factorial plan used for optimization. The experimental responses studied were the yield of biochar, the iodine adsorption (I2), and the methylene blue (MB) adsorption indexes. A pyrolysis temperature of 400°C with a pyrolysis time of 1h would produce the best biochar in terms of yield: 31.67 %. The best result in terms of iodine adsorption is obtained for a pyrolysis temperature of 550°C and time of 1h with 606.57 mg/g. For MB adsorption, the best result is obtained under the same conditions as iodine adsorption with a value of 6.226 mg/g, and a removal percentage of 88.94 %.

***Keywords**:- Biochar; Pyrolysis; Agricultural solid wastes; Characterization; Adsorption.*

I. INTRODUCTION

The valorization of agricultural solid waste has been attracting the interest of many researchers for two main purposes: environmental protection and economic exploitation [1], [2]. These agricultural by-products have gained significant attention for their ability to remove pollutants from aqueous solutions, reuse them as fertilizer, and convert them into biofuel [3]. Adsorbents from agricultural solid waste have great attention from the scientific community because they are from renewable sources, cheap, biodegradable, and after complete usage, do not generate secondary contaminants [4]. Among these agricultural solid wastes, date stones. They are available and abundant in Morocco. These by-products are often considered to be used as domestic fuel by local communities, whereas these biomasses can be recovered as porous materials for wastewater purification applications. Date stone is an agricultural waste existing on the national territory that can be used as an adsorbent. Morocco maintains its position as the 12th largest producer of dates and it consolidates its position with a record forecast production of 143000 T for the 2019-2020 campaign [5]. This work focused on the valorization of this biomass in the development of biochar as an adsorbent. The production of biochar from biomass as an adsorbent has been the subject of numerous studies [6], [7], [8]. It is considered the adsorbent the most promising, efficient, and used for a wide range of pollutants, with a high adsorption capacity.

Biochar is the porous carbonaceous solid produced via carbonization or pyrolysis of biomass. The pyrolysis process is the thermal decomposition of raw materials in a furnace under N2 purge in an inert atmosphere to remove volatility, non-carbon species like nitrogen, oxygen, and hydrogen and to enhance fixed carbon content [9]. Pyrolysis is a thermochemical process that is generally utilized to transform any organic biomass into biochar at temperature ranges from 300°C to 600°C [10]. The pyrolysis parameters significantly affect the process and highly influence the quality of final products, which requires a careful selection of parameters [11]. Carbonization temperature has the greatest importance as it has the most significant effect on the process. The heating rate of the reaction and the carbonization residence or holding time are also the main factors influencing the characteristics of prepared biochar. Controlling the texture and porosity of biochar requires mastering the stages of the biochar synthesis process. This can be realized by optimizing the steps of the biochar manufacturing method. For these reasons, the experimental design method was used to optimize the factors influencing the preparation of biochar. The optimization tools generally used are experimental designs, which guarantee the best quality of the results performing a minimal number of experiments. Among the several types of the above-mentioned method, the two-level complete factorial plan (denoted 2^k) is the most commonly type used, due to its simplicity.

In this work, we optimized the biochar preparation conditions, taking into consideration the pyrolysis temperature and time. Thanks to the important annual production of date stones' by-products in Morocco, we aimed to enhance the biomass by using them as precursors of biochar and to optimize the factors of preparation of biochar.

II. MATERIALS AND METHODS

A. Biochar preparation

The date stones analyzed were selected from Errachidia, Morocco. After cleaning and washing the samples several times with distilled water to remove any impurities, an ambient air drying and a drying at 105 ºC for 24 hours were performed. The dried stones obtained were crushed and sieved to eliminate particles larger than 500 µm. 5g of the resulting powder was placed in a crucible with a lid, the assembly was placed in the muffle furnace. The temperature of the furnace was increased from ambient temperature to 400°C or 550°C with a heating rate of 10°C/min. After a plateau of 1h or 3h at the final pyrolysis temperature, the crucible was removed from the furnace and placed in a desiccator to cool for 30 min.

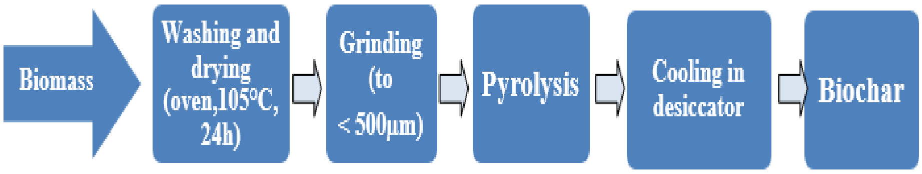


Fig. 1: Process scheme for biomass pyrolysis

B. Biomass characteristics

➢ *Proximate analysis*

- Moisture content

A quantity of biomass was dried using a hot air oven (WiseVen-Wisd, DAIHAN Scientific, Korea) at 105°C until a constant weight. After cooling in the desiccator, the moisture was determined using the following equation (1):

$$\mathbf{H\% = \frac{W_i - W_f}{W_i} \times 100} \qquad (1)$$

Where H = moisture content (%), Wi = initial weight of sample in grams, Wf = final weight of sample in grams after drying.

- Dry matter content

From the water content, the dry matter is easily calculated according to the formula (2): below:

$$\mathbf{DM\ \% = 100 - H\%} \qquad (2)$$

Where DM = dry matter content (%), H = moisture content (%)

- Ash content

A quantity of biomass was placed in a crucible, the assembly was weighed and placed in a muffle furnace at 550°C for 5 hours. Then the crucible was removed from the furnace and cooled in a desiccator and then reweighed. The ash content was determined using the following formula (3):

$$\mathbf{Ash\% = \frac{P_3 - P_1}{P_2 - P_1} \times 100} \qquad (3)$$

Where Ash = ash content (%), P1 = weight of empty crucible (g), P2 = weight of crucible + sample before incineration, P3 = weight of crucible + sample after incineration

➢ *Study of surface morphology*

The morphological characteristics of the raw materials were analyzed using Scanning Electron Microscopy (SEM).

➢ *Chemical characterization*

Elemental constituents of the raw materials were examined by energy dispersive X-ray spectrometer (EDX).

C. Biochar characteristics

➢ *Yield (%)*

The yield reflects the mass loss of the biomass during pyrolysis. After obtaining the biochar mass (in grams), the yield (%) was deduced according to the following equation (4):

$$\mathbf{Yield\ (\%) = \frac{Biochar\ mass}{Biomass\ mass} \times 100} \qquad (4)$$

➢ *Iodine adsorption index (mg/g): I_2*

The purpose of the iodine adsorption index test is to determine the ability of biochar to adsorb small molecules. It characterizes the microspores accessible to small particles. This test was conducted in accordance with the procedure reported by Kouadio et al (2019) [12]. A mixture of 0.200 g of biochar previously dried and 20 ml of 0.1N iodine solution was stirred for 5min before filtration. 10 ml of the filtrate was titrated with 0.1N sodium thiosulphate solution, and using starch as a color indicator. The following formula (5) was used to calculate the iodine adsorption index.

$$\mathbf{I_2\ index\ (mg/g) = \frac{\left(C_0 - \frac{C_{thio} \times V_{thio}}{2V_{I2}}\right) M_{I2} V_{ads}}{m_b}} \qquad (5)$$

C0: Initial concentration of iodine solution (mol/l)
Cthio : Concentration of sodium thiosulphate solution (mol/l)
Vthio: Volume of thiosulphate poured to equivalence (ml)
VI2: Dosed volume of Iodine (ml)
MI2: Iodine molar mass (g/mol)
Vads: Adsorption volume (ml)
mb: Biochar mass (g)

➢ *Methylene blue adsorption index (mg/g): MB*

Methylene blue adsorption index (mg/g) is an indicator of the ability of biochar to adsorb medium and large organic molecules. It characterizes the mesopores of biochar. For the determination of this index, we followed the procedure reported by Kouadio et al (2019) [12]. 0.1g of biochar previously dried was mixed with 100 ml of methylene blue solution (7mg/l), stirred for 20 min, and then filtered. The residual concentration of methylene blue was measured using a UV-Visible spectrophotometer (V-1200, VWR) at a wavelength of 620 nm. Thus, the methylene blue adsorption index is given by the following formula (6):

$$\textbf{MB index (mg/g)} = \frac{V \times (C_i - C_r)}{m_b} \quad (6)$$

V: Volume of methylene blue solution (l)
Ci: Initial concentration of methylene blue (mg/l)
Cr: Residual concentration of methylene blue (mg/l)
mb: Biochar mass (g).

D. Experimental designs

Table 1: Experimental area

Factors	***Input variables***	***Low level (-1)***	***High level (+1)***
Pyrolysis temperature (°C)	X1	400	550
Pyrolysis time (h)	X2	1	3

Table 2: Experimental responses

Response	***Code***	***Unit***
Yield	Y_1	%
I2	Y_2	Mg/g
MB	Y_3	Mg/g

Table 3: Characteristics of the problem

Aim of the study	***Quantification of effects***
Input variables number	2
Experiences number	4
Responses number	3

Table 4: Matrix of experiences

Test	***Average***	***Pyrolysis temperature (°C): X_1***	***Pyrolysis time (h): X_2***	***X_1X_2***	***Response***
1	+1	-1	-1	+1	y_1
2	+1	+1	-1	-1	y_2
3	+1	-1	+1	-1	y_3
4	+1	+1	+1	+1	y_4
Effects	a_0	a_1	a_2	a_{12}	

In order to optimize the synthesis of biochar in terms of pyrolysis yield, iodine adsorption and methylene blue adsorption indexes, an experimental research methodology through an experimental design was carried out.

This approach ensured a complete study of the influence of all the factors on the characteristics of the biochars. It also allowed a limited number of tests to obtain as much information as possible on the effects of these factors.

Experimental designs are essentially a planning strategy for experiments in order to obtain solid and adequate conclusions in an efficient and economical manner. The methodology of the experimental designs is based on the fact that a properly organized experiment will frequently lead to the analysis and a relatively simple static interpretation of the results (ISO Standard 3534-3).

The main goal of using this method is to sort among all the input variables the most likely to influence the output variables. In our case, using the calculation of effects, we will determine the factor the most influencing the pyrolysis yield, iodine adsorption, and methylene blue adsorption indexes. A complete factorial plan with two factors was developed: the pyrolysis temperature (°C) and the pyrolysis time (h) and each factor takes two levels. Table 1 summarizes the characteristics of the problem to be addressed.

Tables 2 and 3 represent the responses chosen, and the model used to study the global effects and interaction effects on the quality and quantity of prepared biochar.

The chosen experimental plan required 2^k tests, with k being the number of factors and 2 being the number of levels. For the present study, a matrix of 4 experiments were needed. The corresponding experimental design is shown in Table 4.

The mathematical model used is presented below (7):

$$\mathbf{Y = a_0 + a_1X_1 + a_2X_2 + a_{12}X_1X_2} \quad (7)$$

Where Y (Response), X1 (Pyrolysis temperature), X2 (Pyrolysis time), ai (Effect of the factor Xi), and aij (Effect of the interaction between the two factors).

In order to determine the coefficient for each factor and for the interaction between the factors studied, the sum of the responses yi divided by the number of experiments (4) where each response is multiplied by the coded unit (1 or -1) according to the column corresponding to each coefficient. We follow the same approach for all the effects of each response.

- *Proximate analysis*

Table 5: Proximate analysis of the biomass studied

Biomass	***Moisture (%)***	***Dry matters (%)***	**Ash (%)**
Date stones	1.62	98.38	1.10

Table 5 presents the results of the proximate analysis carried out on raw date stones. It shows moisture, ash and dry matter contents. The moisture content obtained is very low 1.62%, which can be an advantage for a biochar with high calorific value.

The substrate studied is very rich in dry matter, which gives it a very hard consistency. Low water content levels would contribute to a good conservation of the substrate for a later use. The results obtained show that date stones have a low amount of ash, and this is a sign of good quality of the biochar we have prepared. The high ash content reduced the surface specific. Indeed, ashes did not decompose during pyrolysis, due to their inorganic nature [13].

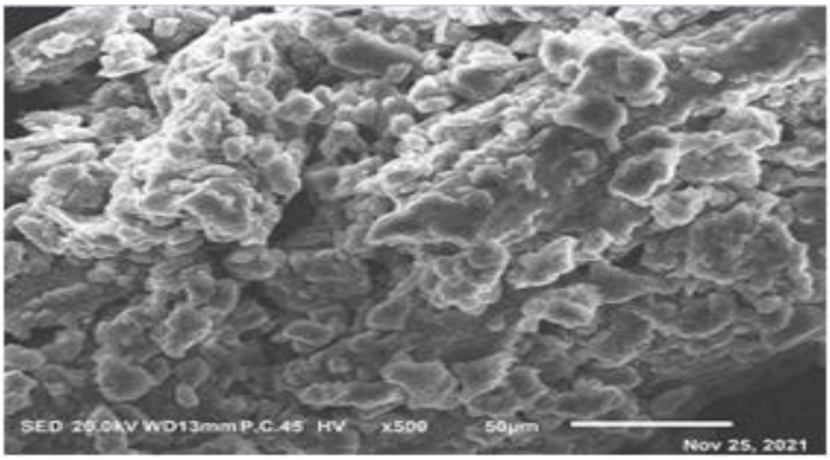
III. RESULTS AND ANALYSES

A. Biomass characteristics

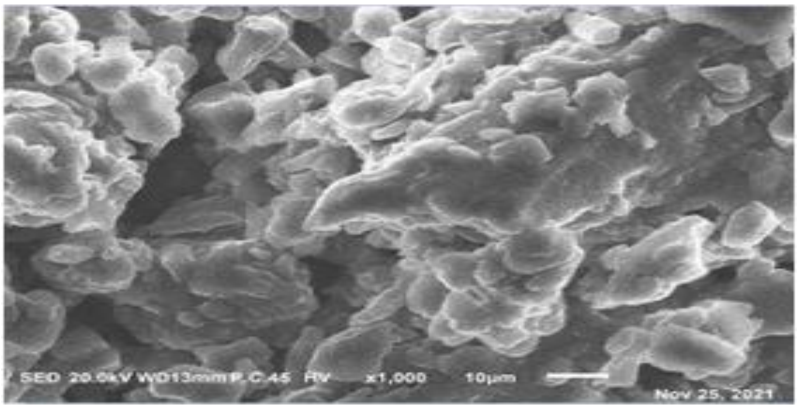
The biomass was first characterized with a view to testing the possibility of synthesizing biochar from date stones. The biochar was then prepared and characterized in order to study the way the synthesis parameters affected the adsorption properties in terms of both iodine adsorption and methylene blue adsorption indexes. Lastly, biochar with the best porosity is selected.

B. Morphology study

The biomass was observed using the scanning electron microscopy (SEM) (Figure 2) with two magnifications (×500) (Figure 2 (a)) and (×1000) (Figure 2 (b)). It is clearly seen that the pores inside the sample were quite homogeneous and well distributed.



(a)



(b)

Fig. 2: SEM images of raw date stones powder with two magnifications (×500) (Figure 2 (a)) and (×1000) (Figure 2 (b)).

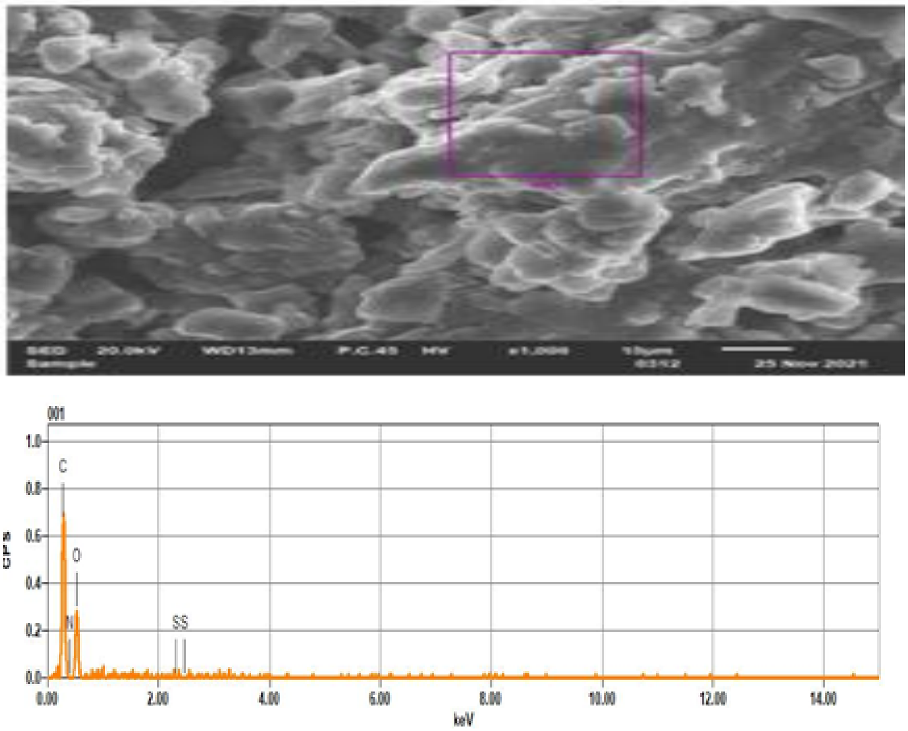


Fig. 3: EDX of raw date stones sample

Table 7: Elementary composition of raw date stones

Element	***Weight (%)***	***Atomic (%)***
C	57.49	64.31
O	42.49	35.68
N	Nd	Nd
S	0.01	0.01

Nd : not determined

The scanning electron microscope (SEM), associated with analysis by energy dispersive X-ray spectrometer (EDX), also allowed the determination of the elementary composition of the samples. EDX spectrum of date stones is presented in Figure 3. The hydrogen content cannot be detected by this technique. The mass and atomic percentages of the constituents of the biomass are grouped in Table 7.

EDX spectrum represented elemental constituents of biomass. The EDX analysis quantifies the mass and atomic percentages of the constituent elements of biomass. The analyses shows that date stones were rich in carbon and oxygen contents (57.49% and 42.49%). On the contrary, the relative content of sulfur was very low (0.01%). The high carbon and oxygen contents attributed the material a high calorific value. This value showed that the biomass could be considered suitable for pyrolysis processes.

C. Test results

Table 8 presented according to the four tests, the results of the three responses; the pyrolysis yield (%), iodine adsorption index (mg/g) and methylene blue adsorption index (mg/g).

Table 8: Experimental design of the biochar

Test	***Temperature (°C)***	***Time (h)***	***Yield %***	***I2 (mg/g)***	***MB Index (mg/g)***	***Methylene Bleu adsorption rate (%)***
1	-1	-1	31,67	581.29	6.132	87.6
2	1	-1	24,28	606.57	6.226	88.942
3	-1	1	29,73	505.47	6.2	88.571
4	1	1	21,64	568.66	6.055	86.5

In order to show the influence of the pyrolysis processes on the iodine adsorption and methylene adsorption indexes of date stones precursors, these indexes were determined before pyrolysis as shown in table 9.

Table 9: Iodine and methylene blue adsorption indexes of the raw date stones

Iodine adsorption index (mg/g)	***Methylene blue adsorption index (mg/g)***	***Methylene Bleu adsorption index rate (%)***
492.83	5.943	84.9

All the biochar obtained had index values of both iodine adsorption and methylene blue (Table 8) higher than those of raw date stones (492.83mg/g and 5.943mg/g, respectively). This suggests that the pyrolysis processes resulted in an adsorbent with high adsorption capacity for small molecules. Also, these processes helped the pores to open allowing the adsorption of large methylene blue molecules.

D. Influence of factors on the experimental responses (yield, iodine adsorption, methylene blue adsorption)

- *Yield*

Table 10: Effect of the two factors and their interaction on yield response

Effects / ***Biomass***	a_1	a_2	a_{12}
Date stones	-3.87	-1.145	-0.175

Based on the effects of the two factors on the yield, the factors have negative effects; an increase in the pyrolysis temperature and time cause a decreasing of the yield of the biochar. We observed that the temperature is the most influential factor (a1).

Since it is often difficult to obtain information by looking at a mathematical equation, global effects graphs are almost always important. The graphical representation of the global effects can be plotted from the table of mean global effects responses (Table 11).

Table 11: Mean effects of the two factors

	Temperature	***Time***
Low level : -1	a_1^{-1}	a_2^{-1}
High level : +1	a_1^{+1}	a_2^{+1}

Where a_1^{-1}, a_1^{+1} are the mean effects of the temperature factor and a_2^{-1}, a_2^{+1} are the mean effects of the time factor, they were calculated using the following equations (7), (8), (9), (10), and (11):

$$a_1^{-1} = \frac{y_1+y_3}{2} \quad (8) \quad ; \quad a_1^{+1} = \frac{y_2+y_4}{2} \quad (9)$$

$$a_2^{-1} = \frac{y_1+y_2}{2} \quad (10) \quad ; \quad a_2^{+1} = \frac{y_3+y_4}{2} \quad (11)$$

Graphical representations based on the results of the experimental design used are shown in Figures 4 and 5.

Based on the results of the experimental design, graphical representations were built to visualize the effects of the pyrolysis temperature (Figure 4 (a)), the pyrolysis time (Figure 4 (b)), and the interaction of the two factors (Figure 5) on the yield of the biochar studied.

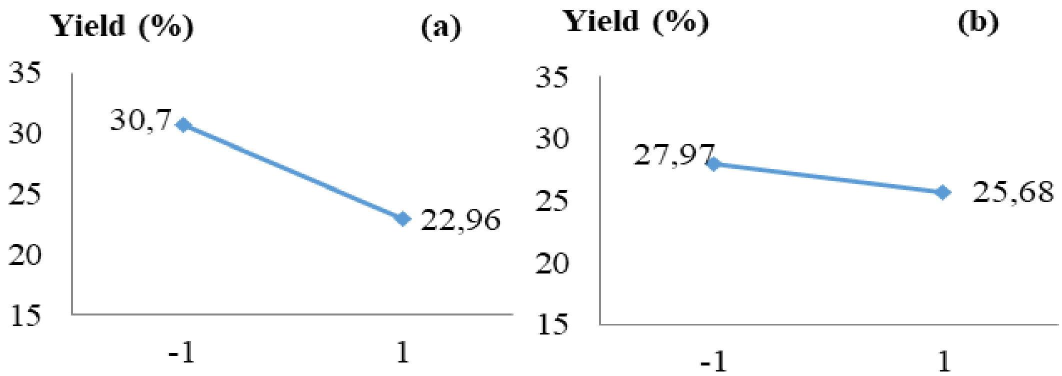


Fig. 4: Graphical representation of the effects of pyrolysis temperature (a) and pyrolysis time (b) on the yield pyrolysis of biochar

The graphs of the effects of temperature and time showed that both factors had significant effects on the yield for the biomass studied. The yield decreased by 7.74 % (2a1) when the pyrolysis temperature was extended from 400°C to 550°C. Thus, it appeared that the increase in temperature led to a decrease in the yield. While, the effect of pyrolysis time was a decrease of 2.29%, with the increase of pyrolysis time from 1h to 3h. We deduced that an increase in pyrolysis time leads to a decrease in the yield. As can be seen, the effect of pyrolysis temperature on the mass yield is greater (significant variation in yield) than that of pyrolysis time. An increase in the pyrolysis temperature or in hold time in the furnace decreases the mass percentage of biochar. This situation is a class phenomenon in thermo chemistry; under the effect of an increase in heat or in the duration of its exposure, there is a greater loss of macromolecules which constitute the biomass.

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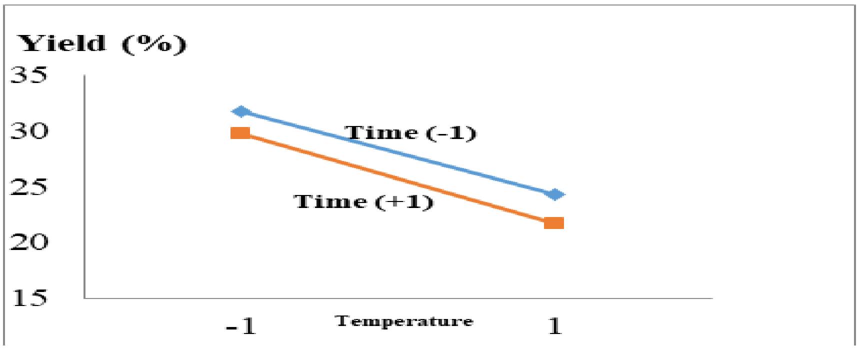

Fig. 5: Graphical representation of the interaction of pyrolysis temperature / time on the yield pyrolysis of biochar

The results of the interaction between temperature and time pyrolysis indicate that there was a lesser or insignificant effect on the yield of the interaction between the two factors as shown in Figure 5 (the two lines of the graph are almost parallel). When the temperature increases from 400°C to 550°C, the yield decreases from 31.67% to 24.28%, it was a reduction of 7.39 % for a pyrolysis time of 1h. Similarly, when pyrolysis time is at its high level (3h), the yield endured a reduction of 8.09%, with the same variation of temperature. The best yield is achieved at the minimum temperature value (400°C) with time pyrolysis of 1h. These results show that the increase in pyrolysis temperature led to a decrease in yield for the two levels of the time pyrolysis. It is clearly shown in Figure 5 that the biochar that had the best pyrolysis yield is the one that has been pyrolyzed at a temperature of 400°C for a time of 1h: 31.67 %.

➢ *Iodine adsorption index*

Table 12: Effect of the two factors and their interaction on Iodine adsorption index response

Effects / ***Biomass***	a_1	a_2	a_{12}
Date stones	22.1175	-28.4325	9.4775

According to the effects on the adsorption capacity of iodine, it was observed that to increase the adsorption capacity of iodine, it is needed to increase the temperature and decrease the time pyrolysis, because the latter had a negative effect.

The figures 6 and 7 visualize respectively the effects of temperature pyrolysis, time pyrolysis, and the interaction between the two factors on the iodine adsorption index of the biochar studied:

The results presented in the figure 6 show that the temperature and the pyrolysis time had significant effects on the iodine adsorption capacity. As the pyrolysis temperature increased from 400°C to 550°C, the adsorption capacity increased by 44.23 mg/g. Indeed, the increase in temperature promoted an increase in the iodine adsorption. Many researchers obtained similar results. For example, Ouedrhiri et al. (2022) [14] found that the iodine adsorption capacity was increased from 101 mg/g to 381 mg/g, when the carbonization temperature increased from 500°C to 900°C for 1h, by carbonization of argan nut shell. In another study realized by Sanda Mamane et al. (2018) [15], by activating the walnut shells of Balanites aegyptiaca (L.) Del. by H_3PO_4, the iodine adsorption capacity was increased from 977mg/g to 990mg/g with the increase of tempearture from 400 to 600°C. In the same study, and with the same variation of temperature, these authors found that the iodine adsorption capacity was increased from 635mg/g to 889mg/g, by activating the walnut shells of Balanites aegyptiaca (L.) Del. by H_2SO_4. The increase in temperature significantly caused the development of micropores leading to an increase in the adsorption capacity of I_2. On the other hand, the response decreased by 56.87 mg/g when the pyrolysis time increases from 1h to 3h. Therefore, an increase in time has a negative effect on the adsorption of iodine.

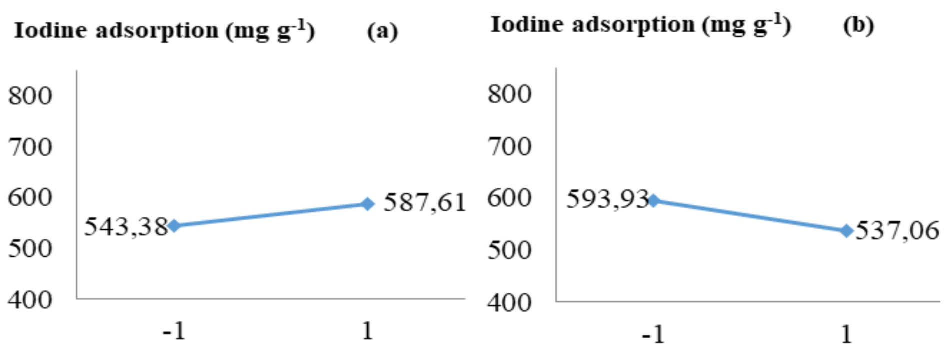

Fig. 6: Graphical representation of the effect of pyrolysis temperature (a) and time pyrolysis (b) on the iodine adsorption capacity of biochar

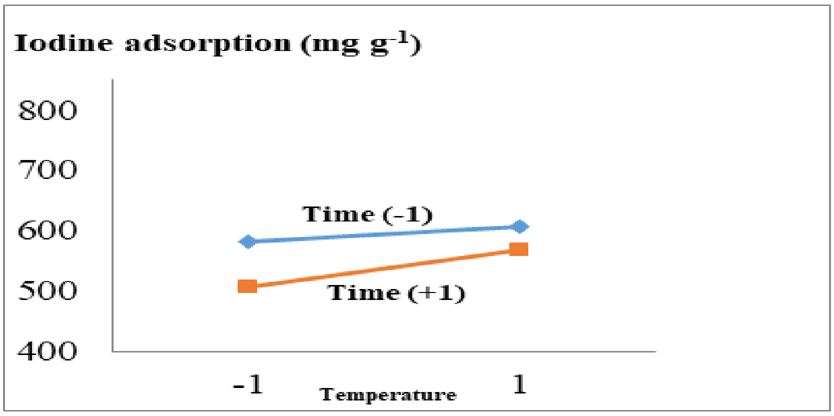

Fig. 7: Graphical representation of the interaction of pyrolysis temperature / time on the iodine adsorption capacity of biochar

We note that there was a significant interaction between the temperature and pyrolysis time for the biomass studied (the two lines of the graph are not parallel). As results, the influence of one of these factors on iodine adsorption capacity depends on the other. Also, an increase of 63.19 mg/g was observed, when the pyrolysis temperature increased from 400°C to 550°C and the pyrolysis time is at its high level (3h). A similar observation when the time pyrolysis is fixed at its low level (1h), the response increased by 25.28 mg/g, with the same variation of temperature pyrolysis. The best adsorption capacity was achieved at the maximum temperature value (550°C) with time pyrolysis of 1h. These results showed that increasing the temperature improved the iodine adsorption for both levels of the pyrolysis time factor.

- *Methylene blue adsorption index*

Table 13: Effect of the two factors and their interaction on Methylene Bleu adsorption index response

Effects / ***Biomass***	a_1	a_2	a_{12}
Date stones	-0.01275	-0.02575	-0.05975

The calculation of the effects of the two factors on the adsorption capacity of methylene blue shows that the increase in the temperature and time pyrolysis has the effect of decreasing the methylene blue adsorption capacity of the biochar.

Figures 8 and 9 present respectively the effects of temperature pyrolysis, time pyrolysis, and the interaction between the two factors on the methylene blue adsorption index of the biochar studied.

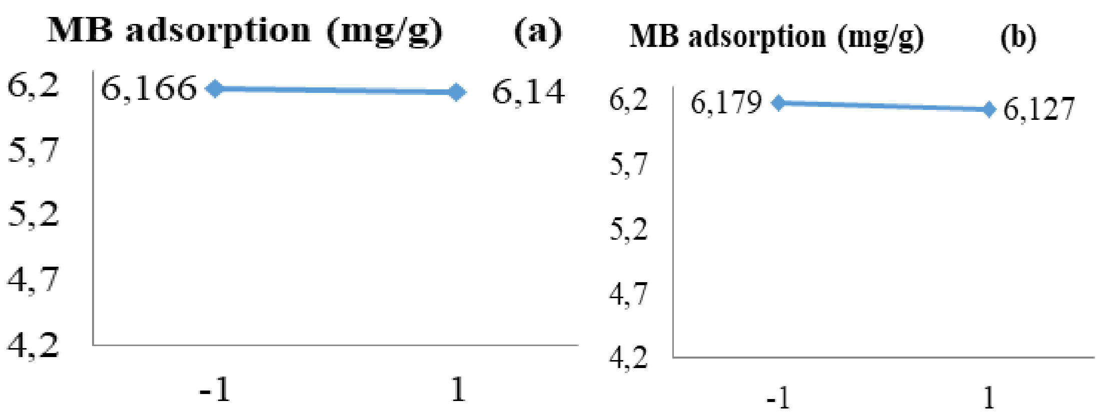

Fig. 8: Graphical representation of the effect of pyrolysis temperature (a) and pyrolysis time (b) on the methylene blue adsorption capacity of biochar

The results presented in figure 8 showed that the two factors did not have an important effect on the adsorption capacity of MB. The methylene blue adsorption decreased only by (0.026 mg/g) with the increase of pyrolysis temperature from 400°C to 550°C. The same results were obtained by Gueye (2015) [18], using activated carbons produced from the wood of jatropha, the hulls of jatropha, the groundnut hulls and the coconut hulls by chemical activation with KOH. These authors found that pyrolysis temperature did not have an important effect on the methylene blue adsorption index. For the effect of the time pyrolysis, there is a decrease of 0.052mg/g with the increase of the pyrolysis time from 1h to 3h. The least pyrolysis time variable impact on the uptake of MB has been observed by a number of researchers. [16] and [17]. reported that activation time or prolonging time of activation did not necessarily lead to the yielding of high surface area and enlargement of pores during production of activated carbon.

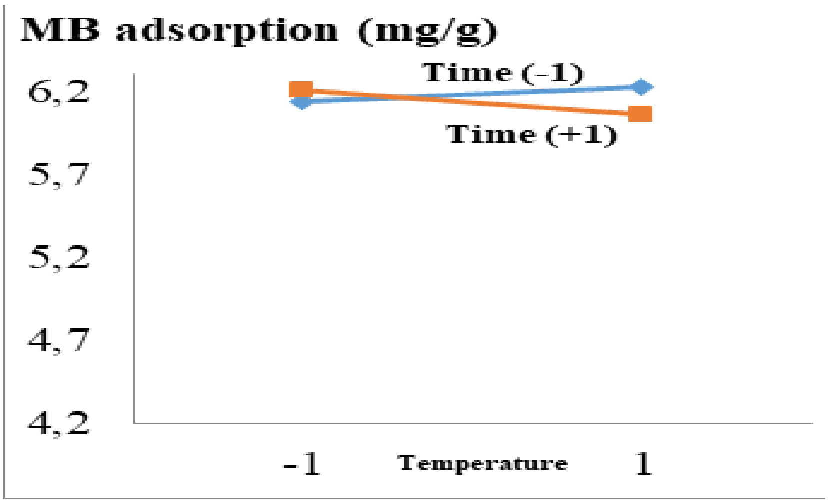

Fig. 9: Graphical representation of the interaction of pyrolysis temperature / time on the methylene blue adsorption capacity of biochar

The results of the interaction between temperature and time pyrolysis on the methylene blue adsorption capacity show that there is a significant interaction between the two factors; the influence of each factor on the adsorption capacity of methylene blue depends on the other factor. An increase in MB adsorption of 0.094 mg/g occurred when the temperature increased from 400°C to 550°C, and the pyrolysis time was at its low level (1h). However, a decrease of MB adsorption (0.145mg/g) happened when the pyrolysis time was at its high level and with the same variation of temperature. The effect of pyrolysis temperature depended on the thermal activation time. The best MB adsorption was achieved at the maximum temperature value (550°C) with a duration of 1h. The best results in terms of MB adsorption were obtained under the same conditions as those of iodine adsorption.

IV. CONCLUSION

The optimization of the production of biochar from date stones has been studied using the experimental design method. The results obtained indicated that the temperature and the pyrolysis time had very remarkable effects on the characteristics of the biochar prepared from the biomass studied. The results of the experimental design showed that:

- The temperature and pyrolysis time had very significant influences on the yield of the biochar, especially the temperature.
- The temperature and pyrolysis time had significant influences on the iodine adsorption.
- The interactions between temperature and time on the iodine adsorption and methylene blue adsorption indexes were very important.
- The best results of yield pyrolysis, iodine adsorption capacity and methylene blue adsorption capacity are 31.67 %, 606.57 mg/g, and 6.226 mg/g, respectively.

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