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Feasibility study of adsorption of a textile dye on activated carbon prepared starting from the coffee grounds

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ABSTRACT/RESUME

Abstract: *The activated carbon used in this study is prepared starting from the coffee ground whose abundance is important in our country and of which the use is often limited to food consummation. The physical characterization (porosity and surface) was determined by the adsorption of gas nitrogen to 77 K (BET), showed the presence of macropores (1.77 m2 /g), as well as the analysis by electronic scan microscopy MEB revealed the presence of a broad specific surface. The activated carbon obtained starting from the coffee ground was the object of an application to the aqueous medium for tests of elimination of a cation dye (methylene blue) used in the industry of textile, prepared synthetically. Several parameters were studied in order to optimize the ideal conditions for a good adsorption of the pollutant to study; in particular, the kinetics of adsorption, the effect of the initial concentration and the effect of the pH of the solution examined. The simulated isotherms of adsorption are very correctly described by the models of Langmuir and Freundlich. The percentage of discolouration reached is of: 99%. Therefore, one can conclude that this study showed that the coffee ground chemically activated can be used like new adsorbent for the water treatment contaminated by the textile dyes.*

I. Introduction

Many industries (textile, paper mill, plastic, agro-foodstuffs…) are water large-scale consumers and use organic dyes (soluble or pigmentary) to color their products. These synthetic dyes are at the same time toxic and persons in charge of the coloring of water [1, 2]. The work presented in this document is based on the treatment of a textile dye (methylene blue) by adsorption on activated carbon resulting from the coffee ground. This study relates to the manufacturing and the capacity of adsorption of an activated carbon, prepared starting from this vegetation waste. The various methods used are: pyrolysis, chemical activation by the phosphoric acid [3, 4,5-8]. The porous texture obtained was characterized by adsorption of gas nitrogen to 77 K (BET) and analyzes by microscopic electronics with sweeping (MEB). The analysis is carried out in statistical mode (discontinuous) by measurement with the spectroscopy of UV/visible.

II. Materials and methods

II.1. Preparation of the Samples

Washing: the coffee ground is washed with the hot water of tap in order to eliminate dust and the various impurities then one carries out the rinsing with distilled water.
Drying: the clean coffee ground is dried with 40-50°C during approximately 1 hour.
Chemical activation: the matter is then mixed with agents activating (we chose the phosphoric acid H3PO4et the chloride ZnCl2 zinc); the coffee ground mixture of activating coffee-agent is introduced into a balloon provided with a cooling agent in which one lets boil until obtaining a pasty state.

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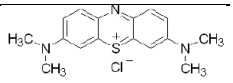
Carbonization: the mixture is then carbonized with high temperature (600°C) in a muffle furnace during 45 minutes. After cooling coal obtained is washed with distilled water then dried with 40-50°C during 24 hours.

II.2. Dye studied

The studied dye is the methylene blue (BM) in cation matter. Its characteristics are presented in table 1.

Table 1.*Caracteristic of the studied dye*

dye	λ_{max}	molar mass g/mol	structure
« Bleu de méthylène »	**664 nm**	**373,9**	

II.3. Characterization physico-chemical of the activated carbon prepared starting from the coffee ground

The main features of the coffee ground used are presented on table 2.

Table2. *Characterization of the activated carbon of the coffee used*

water content (%)	1
Ash contenent (%)	15
Apparent density	0,33
Iodine index (mg/g)	462
Phenol index (mg/g)	38
pH with 20 °C	6,3

Properties acid-basic of the coffee ground

The measures of pH are taken with a pH-meter (JENWAY). A mass of 1 gr of activated carbon is introduced into 150ml distilled water; the mixture is homogenized using a regulated magnetic stirrer with 400 turn/minute with room temperature. The graph obtained thus reveals stable kinetics in the neighborhoods of PH= 6.3 our adsorbent is almost neutral.

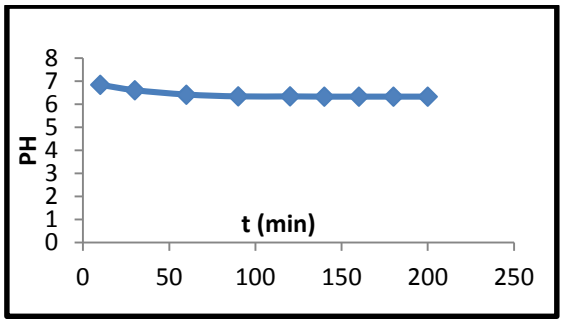


Figure1. *Evolution du PH de marc du café en fonction du temps de contact*

Granulometry

The granulometric analysis carried out on the activated carbon sample of range 400˜1000 microns was carried out on a laser particle-measurement instrument of the type LA 950.The results of this analysis reveal an activated carbon of even granulometry being in margin 400 to 1000 µm as shown in the figure 2.

D (V, 0, 1) = 248,6 µm
D (V, 0, 6) = 456,9 µm
D (V, 0, 9) = 761,94 µm

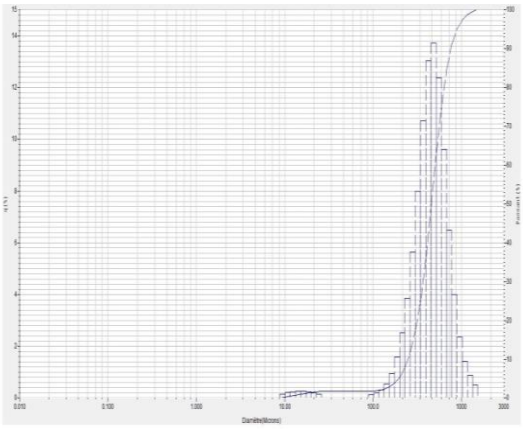


Figure 2. *Curve of granulometry of activated carbon carbon*

10% of the sample to a size lower than 248,6 µm.
60% of the sample to a size lower than 456,9 µm.
90% of the sample to a size lower than 761,94 µm.

$$CU = = \frac{D(60)}{D(10)} = \frac{456,9}{248,6} = 1,83$$

Technique of Brunauer, Emmett and Teller "B E T"

Specific surface, the volume and the average diameter of the pores of our adsorbent (coffee ground) are determined by the technique of Brunauer, Emmett and Teller "BET" by using nitrogen to 7 K, the sample is subjected as a preliminary to a desorption with reduced pressure (< 10^{-4}Torr), at atemperature of degasification of 150°C during 12 hours. The device used is of type "Quantachrome Instruments". The classification of the pores currently adopted by the International union of chemistry pure and applied (U.I.C.P.A) is

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founded on their sizes, three categories of pores were defined [10]:

Micropores: Average diameter of pores (< 2 Nm);porous Volume (0,2-0,6 cm3.g-1);specific Surface (400-900 m2 .g-1).

Mesopores: Average diameter of pores (2-50 Nm);porous Volume (0,02-0,1 cm3.g-1);specific Surface (20-70 m2 .g-1).

Macropores: Average diameter of the pores (>50 Nm);porous Volume (0, 2-0,8 cm3.g1);specific Surface (0,5-2 m2 .g-1).

The results of measurement of the specific surface of coffee ground are gathered in table 3.

Table 3.*Determination of the specific surface of coffee marc by the method of (B.E.T)*

Specific surface ($m^2.g^{-1}$)	Average diameter (nm)	Average volume of the pores ($ml.g^{-1}$)
1,53 **($0,5\text{-}2\ m^2.g^{-1}$)**	60,53 **(>50 nm)**	0,602 **($0, 2\text{-}0,8\ cm^3.g^{-1}$)**

These results enable us to conclude that our adsorbent (coffee ground) comprises primarily macropores.

Porosity

The determination of the porosity of our activated carbon was carried out via the electron microscope with sweeping (MEB) of brand (QUANTA 650) to see the form of the pores and their respective diameters. The figure below watches various states of porosity as well as the presence of cavities. It of form and diameter is varied primarily made up of macroporous, which is confirmed further by the type of isotherm obtained (standard C).

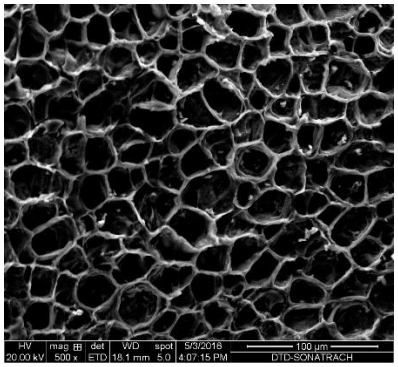

Figure 3. *Photograph with the M.E.B of the activated carbon*

Analysis by x-ray fluorescence

The analyses of x-ray fluorescence were carried out on a spectrometer of x-ray fluorescence of brand PAMALYTICAL-AXIOS. The sample reduced out of powder is subjected to a source of secondary X-radiation of fluorescence characteristic of its chemical composition.

The percentage of the elements obtained by x-ray fluorescence is consigned in table 4.

Table 4. *Results of the analysis by x-ray fluorescence of the activated carbon*

Elements	Percentage (%)	Elements	Percentage (%)
SiO_2	<0,05	MnO	<0,05
Al_2O_3	0,34	P_2O_5	4,45
Fe_2O_3	0,21	K_2O	<0,05
CaO	4,15	Na_2O	0,00
TiO_2	0,01	MgO	1,25
Zn	5,52	Cu	0,053
		Fire loss(CO_2)	95,3
		Total	99,993

Tests of adsorption

The tests of adsorption were carried out of engine batch by agitating the coloured synthetic solution of the methylene blue in the presence of prepared activated carbon. We studied the effect of the principal parameters having an influence on the capacity of adsorption such as the mass of the adsorbent, the pH, the time of contact… etc

The percentage (%) of discolouration of the dye is calculated by using the relation:

$$\textbf{\% of discolouration} = [(C_0\text{-}C_r)/C_0].100$$

With:

C0: initial concentration of the dye (mg/l).

Cr: residual concentration at time T (mg/l).

III. Results and discussion

The kinetics of adsorption (effect of the time of contact)

0.25 gr of activated carbon prepared starting from the coffee ground are put in contact with 100 ml of the coloured solution, the mixture is agitated during 4 hours with room temperature and with a stirring velocity equal to 400 turns per minute. The taking away is collected with predetermined time intervals (10 min).

The kinetic study of the elimination of dye by adsorbent material (activated carbon) shows an increase in percentage of discolouration with the increase in the time of contact. Indeed, after 100 minutes of contact the output of discolouration of the solution of the methylene blue reaches almost the 100% for the concentration of 5mg/l of dye. The results are presented in figure 4.

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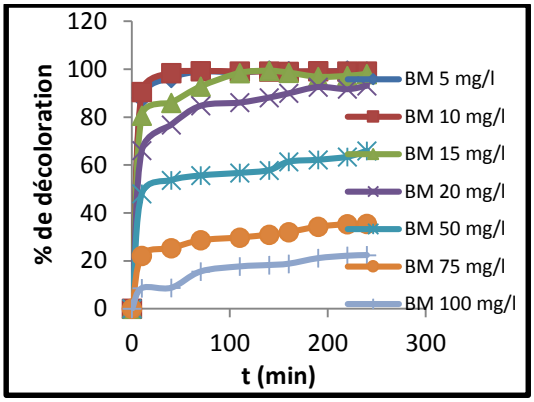

Figure 4. *Effect of the time of contact on the percentage of discolouration of the Methylene blue by the activated carbon (time of contact= 4 hours; m =0, 25 G; pH=6,4; Agitation = 400 tr/min; V =100 ml, T = 19 ±2°C)*

Influence of the pH on discolouration

One adjusts the initial pH of the solutions coloured by using solutions of NaOH (0,1N) and HCl (0,1N) for the various studied values of pH (2, 4,6,8 and 10). Figure 5, watch that with neutral pH (equalizes to 6) made increase the rate of discolouration (84%). beyond this value the percentage of discolouration decreases. We consider that it there is an optimal value of the pH (pH = 6).

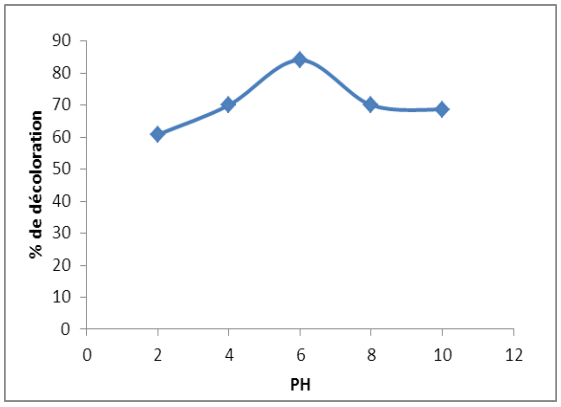

Figure 5. *Effect of the pH on % of discolouration of the Methylene blue by the activated carbon (time of contact= 100 min; m =0,25 G; C= 20mg/l; Agitation = 400 tr/min; V=100 ml, T =19 ±2°C).*

Influence of the mass of adsorbent on discolouration

The activated carbon masses used are: 0.05 – 0.1 – 0.2 – 0.5 – 1 gr. As we can note it according to figure 6, the percentage of discolouration of the methylene blue increases with the increase in the mass of the adsorbent used to stabilize itself with great values of this last. Indeed, the increase in the amount of the adsorbent makes grow the number of the sites available for the fixing of the dyes.

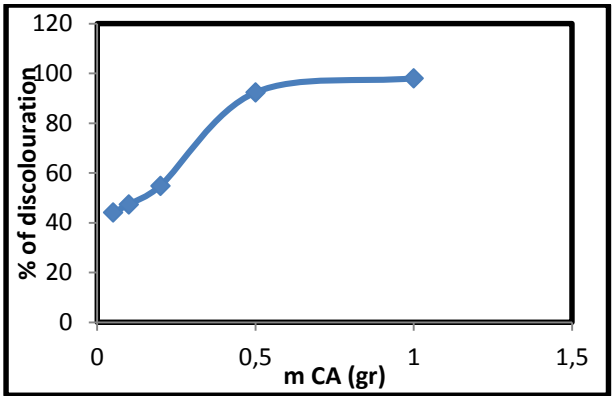

Figure 6. *Effect of the mass of adsorbent (activated carbon) on % of discolouration of the Methylene blue (time of contact= 100 min; PH = 6.4; C= 20mg/l; Agitation = 400 tr/min; V =100 ml, T 19= ±2°C).*

Influence stirring velocity

The experiment is carried out in system batch, we mix 0.25 gr. of adsorbent with solutions of dyes (100ml), while varying the stirring velocity from 300 to 800 tr/min (300, 500,600,700, and 800). The results represented on figures 7, watch that the increase stirring velocity to 800 turns/times, makes increase the rate of discolouration (90%). We consider that it there is a stirring velocity optimal (800 turns/times), sufficient to support the contact between the particles of the developed activated carbon and molecules of dyes.

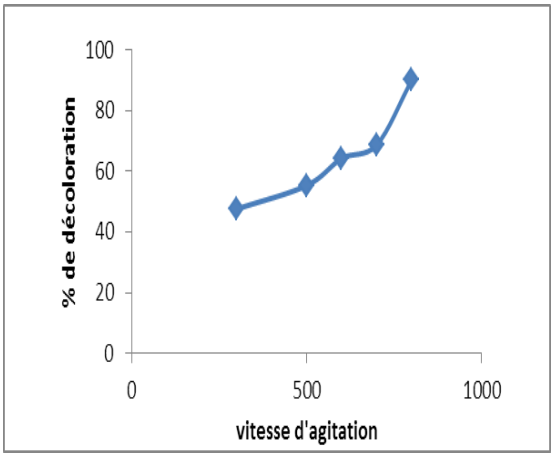

Figure 7*. Effect of the temperature on % of discolouration of the Methylene blue (time of contact= 100min; PH = 6.4; C= 20mg/l; V =100 ml, mass of the activated carbon=0,25gr; T=20 ±2°C*

Isotherm of adsorption

The isotherms of adsorption were studied by agitating a mass of the adsorbent 0.25 G in coloured solutions of various concentrations going from 5 to 100 mg/L. The adsorbent and adsorbed it was put in contact during 100 minutes under an agitation of 400 tr/min. The results of the isotherms of adsorption of the dye on the developed activated carbon are

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presented on figure 8. According to the classification of the isotherms of adsorption of GILLES [10], the isotherms are of type H sub-group max; it is a typical case of the isotherm of the type L, or the initial slope is very high. This case is distinguished from different because the aqueous solution shows sometimes a so high affinity for the solid which the initial slope cannot be distinguished from the infinite one.

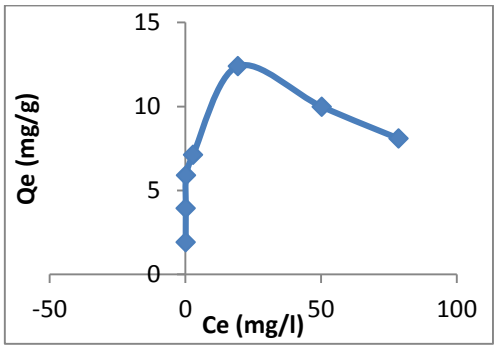

Figure 8. *Isotherms of adsorption of methylene blue by the activated carbon (. (PH=6.4; m = 0.25 G; V = 100 ml; Time of contact = 100min; Agitation = 400 tr/min; T = 20 ±2°C).*

Models of the isotherms of adsorption:

Figures 9 and 10 represent the linear transforms of Langmuir and Freundlich respectively; according to these lines we deduced the values from the maximum capacities and the values of the constants of adsorption determined under the above mentioned experimental conditions.

Model of Langmuir:

$$\frac{x}{m} = \mathrm{Qe} = \frac{\mathrm{a.b.Ce}}{1+\mathrm{b.Ce}}$$

$$\frac{m}{x} = \frac{1}{Qe} = \frac{1+\mathrm{b.Ce}}{\mathrm{a.b.Ce}} = \frac{1}{\mathrm{a.b.Ce}} + \frac{1}{\mathrm{a}}$$

$a = qm$ *ultimate capacity*

$\frac{1}{b} = \mathrm{Kd}$ constant of dissociation of the adsorbent

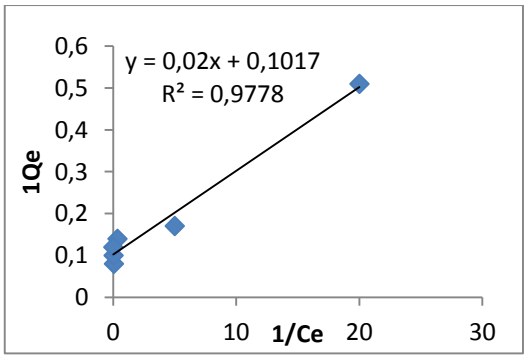

Figure 9*. Linear transforms of the isotherms of adsorption of Langmuir.*

Model of Freundlich

$$\frac{x}{m} = Qe = k.\mathrm{Ce}^{1/n}$$

The linearization gives:

Log x/m=Log Qe=log k+1/n log Ce

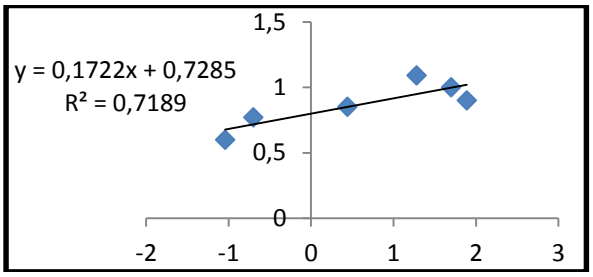

Figure 10. *Linear transforms of the isotherms of adsorption of Freundlich*

The linear representations of the experimental values this process of adsorption enabled us to determine the parameters of balance and the values of the constants of Langmuir and Freundlich calculated by linear regression (Table 5).

Table 5. *Constant of the isotherms of adsorption of Langmuir and Freundlich of the methylene blue on the activated carbon*

Freundlich	n	5,8
	1/n	**0,17**
	K_F	**2,07**
	R^2	**0,718**
Langmuir	b	5
	q_m	**9,83**
	R^2	0,97
	K_d	0,2

The values of the coefficients of regression indicate that the process of adsorption, of Methylene blue by the activated carbon prepared from coffee ground, is described in a favorable way by the isotherm of Langmuir (with excellent linear coefficients of regression R^2 which is very close to one).

IV. Conclusion:

Kinetic studies and the isotherms of adsorption were carried out to clear up the mode of fixing of the methylene blue on material tested. The experiments highlighted that the activated carbon prepared starting from the coffee ground is very effective for the discolouration of water. The percentage of discolouration is influenced by the variation of the pH, it can reach 98% for neutral values of pH, as well as the variation of the mass of adsorb and the stirring velocity affect the output of adsorption. The studies

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continue to seek other less expensive supports which will be combined with the coffee grund in order to in the case of improve the outputs of discolouration for a possible use the rejections of textiles and another effluents charged in organic pollutants.

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