Influence of Some Preparation Parameters on The Efficiency of Activated Carbons Prepared from Teak Wood Shavings (*Tectona Grandis*) and Coconut Shells (*Cocos Nucifera*) for The Treatment of Industrial Wastewater

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ABSTRACT

In this work, we valorized two wastes of plant origin in activated carbon (AC) by chemical activation, with a view to their use for the elimination of industrial dyes and artisanal dyeing simulated by methylene blue and iodine in the water. These are coconut shells (*Coco nucifera*) and teak wood chips (*Tectona grandis*). For the preparation of activated carbon based on coconut residues, the chemical activation was carried out by citric acid extracts of lemon juice (*Citrus aurantiifolia*) and then compared to that carried out with phosphoric acid and hydroxide of potassium. For the preparation of activated carbon from the teak residue, the activating agent used is potassium hydroxide. The results obtained show that activated carbon based on the coconut are microporous and mesoporous with an iodine number between 338.9 and 487.9 mg/g, a methylene blue number between 369.2 and 447.2 mg/g and a more pronounced post-treatment residual acidity for phosphoric acid than with lemon juice. The test conditions (the impregnation ratio, the duration, and the carbonization temperature) made it possible to prepare six activated carbons from the teak residue. The carbonization time does not have a significant effect on the adsorption efficiency although it does have a considerable effect on the carbonization efficiency. The adsorption kinetics of methylene blue on these materials is second order and the adsorption isotherm is more of the Langmuir type than that of the Freundlich model; this isotherm is characteristic of a wide distribution of microporosity and a well-developed mesoporosity. These activated carbons can therefore be used in the treatment of wastewater, in particular for the removal of industrial dyes and artisanal dyeing.

Keywords: Activated Carbon, *Citrus Aurantiifolia*, Coconut, Teak.

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I. INTRODUCTION

The sixth Sustainable Development Goal emphasizes the importance of facilitating, by 2030, equitable and universal access to drinking water for the populations of the world. However, the figures show that efforts are still to be made in the world, in sub-Saharan Africa and mainly in Benin. For example, 80% of the population lives in localities where access to drinking water is difficult or even impossible. These citizens are therefore forced to use water from alternative sources (lakes, rivers, wells, etc.) without any treatment [1]-[4]. At the same time, industries generate large quantities of wastewater without necessarily attaching importance to its treatment [5]. However, if this water were treated, it could be recycled in the processes, which would make it possible to face the challenges of the scarcity of water resources which would be more pronounced by 2050 [6].

There are a large number of wastewater treatment processes including adsorption on activated carbon which is particularly appreciated [7]. One of the reasons for the success of this process is attributed to its effectiveness in removing a wide range of pollutants. The large specific surface of activated carbons explains the use of relatively small quantities to treat large volumes of water.

In the literature, activated carbons have been used to remove different types of pollutants, including heavy metals [8], [9]. The objective of this work is to study the influence of different parameters for the preparation of activated carbons on their effectiveness in removing micropollutants from water. Activated carbons in this study are prepared from two locally available and accessible lignocellulosic wastes: coconut husks (*Cocos nucifera*) and teak wood chips (*Tectona grandis*).

II. MATERIAL AND METHODS

A. Collection And Processing of Activated Carbon Precursors

The teak wood chips used are harvested at a local sawmill in Agassa-godomey (Akassato/Benin). For the coconut shells, they were collected from a tradeswoman in a local market. After collection, the chips were first sorted (so as to rid them of all foreign bodies) and then dried before being crushed. The powder obtained was washed several times with tap water and then with distilled water before being placed in an oven. The dry product obtained was sieved and only the particles with sizes between 0.5 mm and 1 mm were retained for the rest of the chemical activation operations.

The pre-treatment of the coconut shells was done following four successive stages:

- drying in the sun to reduce the moisture content in the shells.
- scraping and cleaning the hulls to remove fibers.
- crushing and grinding to reduce the size of the shells until small grains are obtained.
- sieving with AFNOR sieves to retain only grains with a particle size between 0.5 mm and 2 mm.

B. Activation of Teak Residues

Several factors influence the final characteristics of chemically activated carbons. These are essentially the activating agent, the impregnation ratio, the temperature and the duration of carbonization [10]. Here, potassium hydroxide (KOH) is the activator used for the preparation of all activated carbons made from teak wood chips. Two impregnation ratios are retained after the tests: 1 and 1.5. Indeed, the idea was to verify the hypothesis that an increase in the impregnation ratio would lead to an increase in the carbonization yield [11]. According to the work of [12], the specific surface area of activated carbons chemically activated with KOH increases with the carbonization temperature, reaching its maximum at 800 °C. Based on the work of [13] and [14], the carbonization conditions used are (500 °C, 1h); (500 °C, 2h), (600 °C , 1h). In the end, six activated carbons were prepared as presented in Table I.

For each type of activated carbon, three carbonization cups are inserted into the oven for preparation. Once prepared, the carbons are weighed in order to conclude on the influence of the carbonization time, the carbonization temperature and the impregnation ratio on the carbonization yield. The coals are then ready for washing, a very important step to develop their porosity by eliminating the KOH (chemical activator used) which would still be in the pores.

Thus, each sample of activated carbon is placed in a certain volume of water whose initial pH is known. After shaking the beaker containing the mixture (distilled water + activated carbon) for a few minutes, the pH is measured. The supernatant is poured out and replaced with a new quantity of water, so as to repeat the process until a stable pH between 6 and 7 [16].

After washing, the activated carbon samples are dried in an oven at 105° C. for 24 hours. They are then ground to obtain a homogeneous particle size then stored in small plastic boxes pending the adsorption tests.

TABLE I: CONDITIONS FOR PREPARING ACTIVATED CARBON BASED ON TEAK WOOD CHIPS

Name of activated carbons	Charring time (Hours)	Impregnation ratio	Temperature of carbonization (°C)
CACB2	2	1	500
CACB3	1	1	600
CACB5	2	1	600
CACB7	2	1.5	500
CACB9	1	1.5	600
CACB11	2	1.5	600

C. Activation of coconut residues

The preparation parameter targeted here is the activating agent. We wanted to study the effectiveness of citric acid in lemon juice as an activating agent. Several studies [12], [18] on coconut shells have already focused on the study of the influence of the mode of activation, the carbonization temperature, the impregnation ratio and carbonization time.

Five (5) activated carbon samples designated A_0 to A_4 were prepared using the same process. Samples A_0 and A_4 are first pre-carbonized at a temperature of 550 °C for 2 hours. The sample A_0 will serve as a control and does not undergo any impregnation. The impregnation conditions of the other four samples are presented in Table II.

The impregnation of these four (04) other samples was done on a VWR brand shaker at 250 rpm for 24 hours. After this impregnation, the solid part of each sample was collected and placed in an oven at 105° C. to dry for 48 hours.

All samples are (re)fired and charred up to 700 °C as follows:

- A rise of 10°C/min from ambient temperature to 700°C;
- A two-hour plateau at 700°C;
- Finally, a free descent of the temperature to room temperature to obtain a dry residue free of any humidity.

After leaving the oven, all the samples were washed thoroughly. A first wash was done with distilled water heated to a temperature of around 100°C. Washing then continued with ordinary distilled water repeatedly and ended when the pH of the wash solution is close to neutral (6 to 7).

After rinsing, the clean charcoal was put in an oven at 105° C. for 24 hours to dry . Then it is poured into a mortar and crushed with a pestle until a fine powder is obtained. This fine powder was sieved through a 250 µm sieve and retained by a 71 µm sieve.

TABLE II: CONDITIONS FOR PREPARING ACTIVATED CARBONS BASED ON COCONUT SHELLS

Samples	Hull mass coconut (g)	Amount of activating agent	Volume of distilled water (mL)
A_1	50	35 mL (H_3PO_4)	65
A_2	50	50 g (KOH)	100
A_3	50	50 mL (lemon juice)	50
A_4	50	50 mL (lemon juice)	50

D. Determination of Iodine Number and Methylene Blue Number (MB)

The iodine number is defined as the milligrams of iodine adsorbed by 1 g of charcoal when the iodine concentration of the residual filtrate is 0.02 N. The determination of the iodine number is a simple and rapid test, giving an indication of the microporosity of the activated carbons. The method used is based on the experimental protocol proposed by [15]. The higher the iodine number, the greater the affinity the carbon has for small molecules.

The BM index (QBM index) is used to evaluate mesopores and macropores, which is synonymous with a strong ability to adsorb larger molecules. It is defined as the volume, in milliliters (ml), of the standard solution of methylene blue decolorized by a fixed quantity of anhydrous activated carbon [17].

1) Calibration of methylene blue

To calibrate Methylene Blue, a 10 ppm stock solution was prepared in a one liter flask. Various dilutions were then carried out. A total of 21 points have been fixed. Once the dilutions have been made, the absorbance's are read on-site using a spectrophotometer. The results are then processed using Microsoft Excel. The relationship between the absorbance and the concentration of methylene blue is therefore linear in accordance with Beer Lambert's law.

2) Study of the adsorption kinetics of methylene blue on activated carbons based on teak residues

We prepared 100 mL of a 50 ppm methylene blue solution which we brought into contact with 10 mg of activated carbon. The kinetic models studied are:

a) Pseudo-first-order model:
Its linear form is:

$$ln(q_e - q_t) = lnq_e - Kt \qquad (1)$$

with q_e the amount adsorbed at equilibrium; q_t the quantity adsorbed at time t, t the contact time and K the rate constant for the pseudo first order model.

We have therefore plotted the curve ln $(q_e - q_t)$ as a function of time. The y-intercept and the slope coefficient of the line give the parameters of this model.

b)   *Pseudo-second-order model:*

Its linear form is presented in (2).

$$\frac{1}{q_t} = \frac{1}{q_e}t + \frac{1}{K'q_e^2} \qquad (2)$$

with K' the rate constant for the pseudo second order.

Here we have plotted the curve $\frac{1}{q_t}$ as a function of time. The y-intercept and the slope coefficient of the line give the parameters of this model.

c)   *Intra-particle diffusion model, shown in (3).*

$$q_t = K''t^{1/2} + C \qquad (3)$$

with C the value of the boundary layer thickness and K" the intraparticle diffusion constant.

3) *Study of adsorption isotherms*

At this level, we are interested in two models: the Langmuir model and the Frundlich one.

a)   *Frundlich model:*

The linear form of this model is shown in (4):

$$lnq_e = lnK_f + \left(\frac{1}{n}\right)lnC_e \qquad (4)$$

4) *Langmuir model*

Two linearized expressions are used from the basic formula of the Langmuir model, which are shown in (5) and (6).

$$\frac{1}{q_e} = \frac{1}{q_m} + \frac{1}{q_mK_L} X \frac{1}{c_0} \qquad (5)$$

$$\frac{1}{q_e} = \frac{-1}{q_mK_L} X \frac{q_e}{c_0} + q_m \qquad (6)$$

In this formula, q_e is the quantity of BM adsorbed at equilibrium in mg/g, , q_m is the maximum quantity of BM necessary to cover the first layer of the activated carbon in mg/g, K_L is a characteristic constant of the activated carbon, influenced essentially by temperature and experimental conditions. (L/mg). This is the residual concentration at equilibrium and K_f the Frundlich coefficient. Depending on the value of n, we can conclude on the shape of the isotherm [19] and the ease or otherwise of adsorption [20].

III.   RESULTS AND DISCUSSION

A. Activated Carbons Prepared from Coconut Shells

The methylene blue and iodine indexes obtained are indicated in Table III. The results obtained show us that the iodine index of our activated carbons carbonized at 700°C varies from 338.9 to 487.9 mg· g^{-1}. Maximum values are obtained with phosphoric acid. Samples A$_2$ and A$_4$ having lemon juice as a chemical activator give identical results even though the impregnation process used is different. It can therefore be deduced that in the case of the ½ ratio used which implies a dilution of 50%, the chemical impregnation can be done before or after the pre-calcination because the iodine number remains the same. In fact, the iodine number increases with the concentration of the activating agent [21]. Lemon juice could thus provide better results if it were less diluted because remember that citric acid is a weak acid, and that lemon juice contains about 88.98% water. Thus the lemon would be a good activator if its concentration before the chemical activation of the activated carbon is optimized by removing a good part of the water it contains.

TABLE III: Iodine And Methylene Blue Indices of Activated Carbons Based on Coconut Residues

Samples	Iodine value (mg/g)	Methylene blue index (mg/g)
A_0	377.9	484.19
A_1	487.1	399.88
A_3	338.9	471.22
A_4	338.9	421.93

Activated carbons that have adsorbed more iodine than BM have more capacity to adsorb particles of size less than 2 nanometers and are called microporous carbons.

The samples made from lemon juice show good results for the methylene blue index. However, methylene blue makes it possible to characterize mesopores and macropores. It can therefore be said that coals prepared from lemon juice are microporous and mesoporous.

B. Activated Carbons Made from Teak Wood Shavings

By varying the temperature from 500 to 600° C. (the ratio being set at 1 and the carbonization time at 2 hours), it can be seen that the carbonization yield decreases considerably. Similarly, by varying the carbonization time from 1 hour to 2 hours (the carbonization temperature being set at 600° C. and the impregnation ratio at 1), it is noted that the carbonization yield decreases. [22] also showed that the pyrolysis mass yield decreases with pyrolysis temperature and residence time.

Finally, by varying the impregnation ratio from 1 to 1.5 (the carbonization temperature being set at 600° C. and the carbonization time at 1 hour), it is noted that the carbonization yield increases. [13] obtained similar results with phosphoric acid and coconut shells. For impregnation ratios ranging from 0.7 to 2, they noticed an increase in mass yield.

From Figure 1, it can be seen that under our experimental conditions, the carbonization time does not greatly influence the methylene blue adsorption efficiency. Indeed, the adsorption yield goes from 95.10 % to 89.75% when going from 2h to 1h at 600°C and an impregnation ratio of 1. The same observation is made for the impregnation ratio 1.5 when going from 2h to 1h, the adsorption yield goes from 60.48 % to 55.69 %. These results are consistent with those reported by [23] from walnut cakes.

However, in both cases, the adsorption yield is improved with increasing carbonization time. [24] reached similar conclusions with the activated carbons they prepared from industrial sisal (*Agrave sisalana) fiber waste by chemical activation with phosphoric acid.* . On the other hand, the impregnation ratio is a very important factor and greatly influences the adsorption yield of methylene blue on an activated carbon. When going from ratio 1 to ratio 1.5 (at 600° C. and for a carbonization time of 1 hour), the adsorption efficiency goes from 89.75% to 60.48%. Similarly, when going from ratio 1 to ratio 1.5 (at 600° C. and a carbonization time of 2 hours), the adsorption yield goes from 95.10% to 55.69%. Thus, the more the ratio increases, the adsorption efficiency decreases. This is consistent with results published by [25] who prepared activated carbon from used tires at 700°C by alkaline activation. They obtained a decrease in the BET surface, the iodine index and the methylene blue index when the impregnation ratio (mass of precursor/mass of KOH) goes from 1:1 to 1:2 then at 1:3. It is possible that the impregnation ratio had a positive effect on the adsorption yield up to 1 and then had a negative effect from 1.

[26] prepared activated carbon from wood by chemical activation with KOH. They reported an increase in the maximum adsorption capacity of methylene blue (obtained from the linear form of the Langmuir model 1) and in the BET-specific surface when the impregnation ratio changes from 0.25 to 1. [27] noted an increase in BET surface by varying the phosphoric acid/Lignin impregnation ratio from 0.7 to 1.4 at 450°C. For higher impregnation ratios, the BET surface decreased.

In industry, two factors are important for the production of activated carbon: the carbonization yield and the adsorption yield. It is noted that these two factors are generally inversely proportional under the conditions of our experiment, that is to say, the higher the carbonization yield, the lower the adsorption yield. The best compromise is offered with CACB3 carbon which is subsequently used for the study of isotherms.

The values of the absorbance obtained made it possible to find a linear relationship between the absorbance and the concentration of methylene blue in the solution. The correlation coefficient of 0.9991 obtained testifies to a good correlation between the absorbance and the concentration of methylene blue in the field of study.

Table IV presents the kinetic parameters obtained after plotting the pseudo-first-order (Fig. 3), pseudo-second-order (Fig. 4), and intraparticle diffusion (Fig. 5) models.

It can be seen that the best correlation coefficients are obtained for the pseudo-second-order model. This finding is made for all three activated carbons used for the tests. Moreover, the values of the calculated and experimental quantities at equilibrium are in agreement with this last model.

Thus, it appears that the adsorption of methylene blue on the activated carbons of teak wood chips would better follow the pseudo-second-order model. [8] has moreover established that the pseudo-second-order model makes it possible to verify the adsorption of Methylene Blue (MB).

TABLE IV: KINETIC PARAMETERS OF ADSORPTION OF METHYLENE BLUE

| Charcoal	Pseudo 1st order model	Pseudo 2nd order model	Diffusion intra-particle
CACB11	a= - 0.0752	a= 0.0360	a=9.1943
	b=4.4592	b=0.0039	b=213.94
	K1=0.0752	K1=0.0033	k=9.1943
	Qe=86.4183	Qe=277.78	C=213.94
	R^2=0.8475	R^2=0.998	R^2=0.9786
CACB9	a= - 0.0416	a= 0.0032	a=7.0835
	b=4.1255	b=0.0027	b=253.28
	K1=0.0416	K1=0.0004	k=7.0835
	Qe=61.8987	Qe=312.5	C=253.28
	R^2=0.7857	R^2=0.9995	R^2=0.9522
CACB5	a=- 0.0706	a= 0.0021	a=16,281
	b=5.0919	b=0.0028	b=346.79
	K1=0.0706	K1=0.6016	k=16.281
	Qe=162.6986	Qe=476.8904	C=36.79
	R^2=0.6589	R^2=0.9998	R^2=0.8088
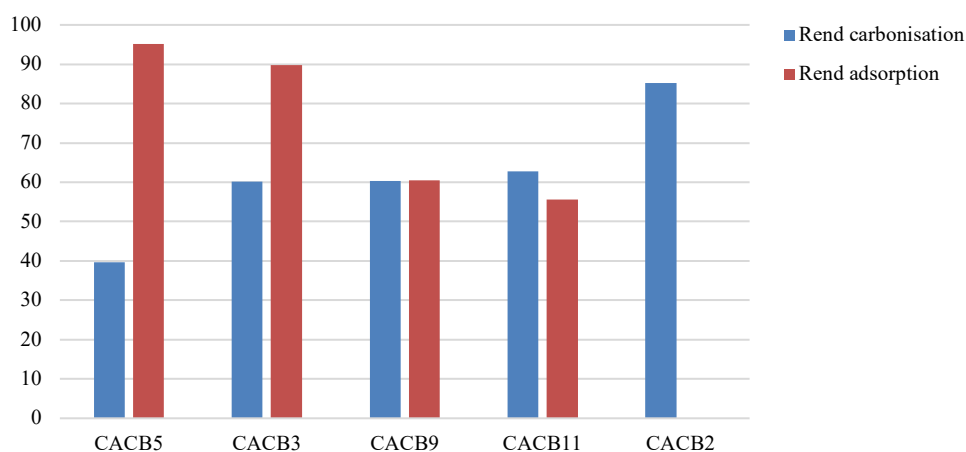
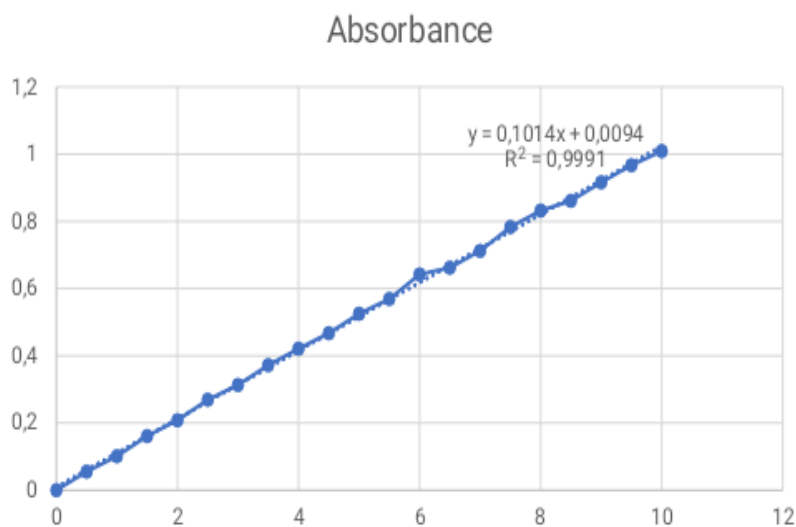Fig. 1. Carbonization and Adsorption Yields.
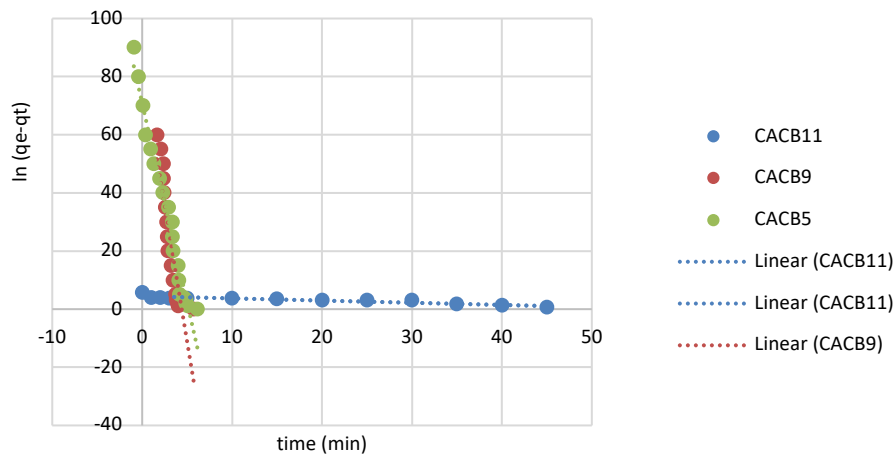

Fig. 2. Methylene Blue Calibration Curve.

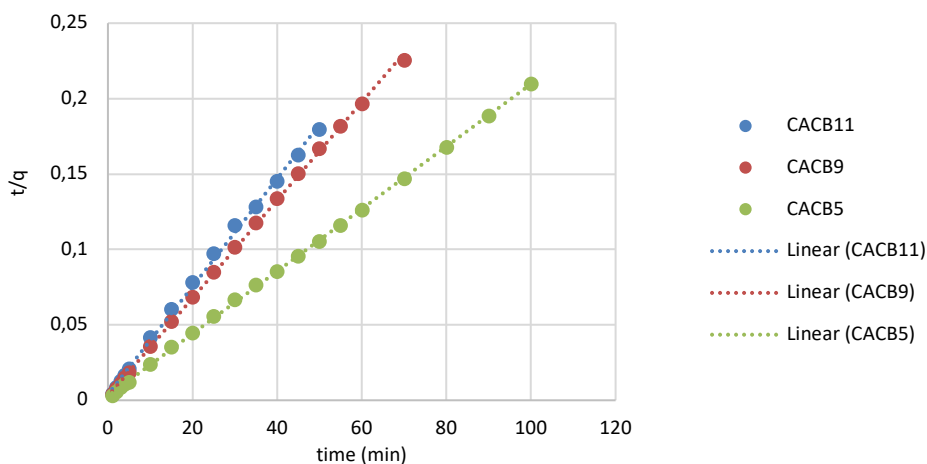Fig. 3. Plot of pseudo first order linear models.

Fig. 4. Plot of the pseudo second-order linear model.
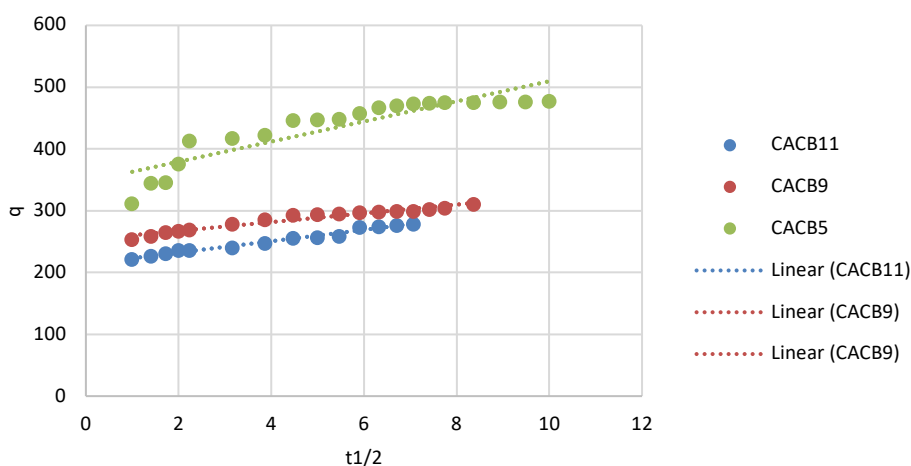

Fig. 5. Plot of the linear model of intraparticle diffusion.

The results obtained after 1 hour of contact of the activated carbons with BM are used to construct the adsorption isothems. The various calculations of the parameters are recorded in Table IV. From Table V, it appears that the best correlation coefficient is obtained for the Langmuir 2 model. It could therefore be concluded that the adsorption of methylene blue on activated carbons based on teak wood chips is made according to the assumptions of Langmuir with in particular a monolayer distribution.

For the adsorption isotherms, we see that the value of 1/n is less than 1 for the Frundlich model, which means that the isotherm is concave of type L. In addition, n is between 2 and 10, which means that adsorption is easy for the range of dye concentrations studied.

TABLE V: Constants Of Adsorption Isotherms

Models	Settings
Langmuir 1	Qm= 434.78 mg/g K= 0.2174 R^2= 0.9513
Langmuir 2	Q m = 769.75 mg/g K = 0.6842 R^2= 0.9831
Frundlich	Kf = 277.0505 n= 3.048 R^2= 0.9772

According to [28], the L-type isotherm is characteristic of microporous carbons and more precisely the shape obtained is characteristic of an activated carbon with a wide distribution of microporosity and a well-developed mesoporosity. Activated carbons from *Tectona grandis wood chips* can therefore be used to decolorize dyeworks effluents.

IV. Conclusion

This work first confirms the interest that researchers attach to the recovery of various lignocellulosic waste into activated carbon to solve water pollution problems. It comes specifically to give a new way of recovering coconut shells and wood chips. These can serve as raw materials for the preparation of activated carbons. Lemon juice has shown itself as a potential activating agent, inexpensive, accessible and practical to use for the local population, unlike phosphoric acid or sodium hydroxide. However, it would be necessary to seek to concentrate the citric acid by reducing the proportion of water initially contained in the juice used as an activator. Under the conditions described in this work, this activation can take place before or after carbonization. Finally, the optimal conditions obtained for the industrial preparation of activated carbons from teak wood chips are impregnation rate 1, carbonization duration 1 hour, and carbonization temperature 600 °C.

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