

# Removal of Violet Crystal from Aqueous Solution by Limon Peel Powders from Adamawa Region of Cameroon

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**Abstract:-** The pollution of wastewater from industrial discharges is a serious problem in several developing countries, particularly Cameroon. This pollution can cause damaging effects on the environment and human health. For this purpose, several physicochemical processes for the treatment of wastewater have been implemented, in particular that of adsorption. The present study consists in using a new natural adsorbent based on silt skin powders in order to treat industrial and artisanal discharges. The tests were carried out on Crystal violet which is a basic cationic dye. The influence of physicochemical adsorption experimental parameters was studied, namely contact time, mass, pH, concentration and temperature. These tests showed a remarkable elimination from the first 20 minutes. The kinetic models of pseudo-first and second order have been studied. The Langmuir and Freundlich isotherms were also studied. However, the experimental results showed that the adsorption of Crystal violet by the biosorbent reached an elimination rate of 81.36% at pH  $6.15 \pm 2$  and at a temperature of  $25 \pm 1$  ° C. The adsorption of Crystal violet has made it possible to understand that the pseudo-second-order kinetic model better describes the adsorption of confirmed crystal violet by a correlation coefficient  $R^2$  of greater than 0.999. The Freundlich isotherm better describes the adsorption of Crystal violet with a correlation coefficient greater than 0.90. Thermodynamic parameters such as  $\Delta G^\circ$ ,  $\Delta S^\circ$  and  $\Delta H^\circ$  have shown that the crystal violet adsorption mechanism on silt skin powders is physical, spontaneous and exothermic and between 293K and 333K. Therefore the skins of silt can be valued as adsorbents in general and in particular as biosorbent in the treatment of industrial effluents. Analysis of the FT-IR spectrum shows the presence of numerous functional groups such as phenol, amide, amine, carboxylic acid, alkene, alkyne.

**Key words:** - Lemon peel, Crystal violet, adsorption, Adamawa region.

## 1. INTRODUCTION

Water is a vital resource for man; his health and development depend on it. But because of the increase of its consumption and its use by the individuals and the industrialists, this one is subject of the pollution which thus generates wastewater which is rejected in an anarchic way and without treatment (AGENCE EAU / France, 1994), which contributes significantly to the contamination of the water table in particular and the environment in general. In developing countries, the activities of artisans such as

tanneries, dyeing companies ... are the ones that cause water pollution (Adjia et al., 2014); Thus marking whose dyes as major pollutant of these wastewater. Crystal violet, which is a basic cationic dye (Shawabkeh and Tutunji, 2003), once dissolved in water, is sometimes difficult to treat because they have a synthetic origin and a complex molecular structure that makes them more stable and difficult to be biodegradable (Forgacs et al., 2004). Several methods have been developed to treat this wastewater and are: physical, chemical and biological (Rangabhashiyam et al., 2013). But all these techniques seem very expensive for the developing countries, which do not control this water pollution, lack of less expensive and adequate treatment.

The technique of adsorption which is the most favorable method for the elimination of dyes in general and particular Crystal purple has become an analytical method of choice, very effective and simple in its use. The study of the present art, several solid materials (clays, zeolites, activated aluminas, sludge, biomasses, agricultural residues, industrial by-products and activated carbon, etc.) that can be used in processes for the depollution of wastewater (Faouzia, 2014). The valorization of agricultural residues without generating pollutants is a great challenge and is recommended for sustainable industrial development in order to preserve the environment (Khalfaoui, 2012). Waste resulting from the processing of raw materials generally has little or no market value (Faouzia, 2014). Thus, the use of these by-products as materials aims to valorize them and prevent the possible consequence that is harmful for the environment and health. Moreover, for the adsorption of dyes, the most sought after materials are those with a high content of tannin and lignin such as bark (orange) and sawdust (Faouzia, 2014).

So far, no study has been conducted on silt skins in wastewater remediation. The skins of lemons in general and in particular the skins of silt are one of the most important skins of the citrus fruit, because the silt appears as one of the most important in the ingredients of the kitchen, a garnish, like juice in lemonade, carbonated beverage and others (Bhatnagar et al., 2009). To do this, to avoid visual pollution by these skins of silt, they can be upgraded after extraction of the juices as abundant and less expensive adsorbents or biosorbents in wastewater treatment (Bhatnagara et al., 2010).

The present work makes it possible to prove the adsorptive potential of silt skins in the treatment of wastewater to improve the living conditions of the populations.

## 2. MATERIAL AND METHODS

### 2.1. Preparation of the biosorbent.

The silt skins used as biosorbents in the context of our work concerning the adsorption of Crystal violet in batch mode are obtained after peeling the silt fruits purchased from the village women in the local markets of Dang (N'Gaoundéré-Cameroun). These wet skins are washed several times with tap water and then rinse several times with distilled water. After washing, these skins of slimes are dried for 24 hours between 40 and 50° C inside a

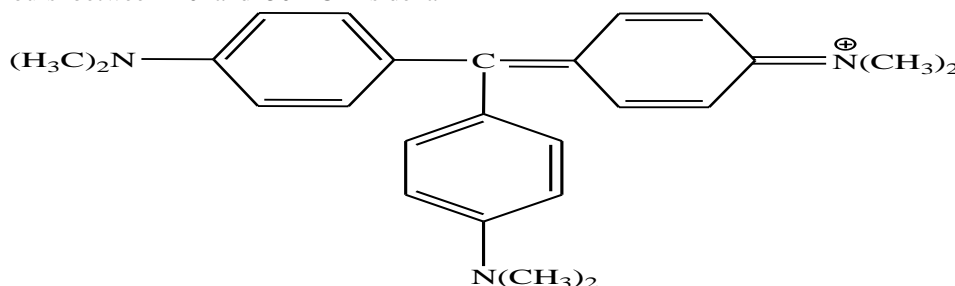


Figure 1: Structure of Crystal violet

### 2.3. PHYSICO-CHEMICAL CHARACTERISTICS OF THE BIOSORBENT

#### 2.3.1. Determination of the specific surface area by the methylene blue method (AFNOR, 1992)

The principle of this method is to determine the necessary amount of methylene blue to cover a mono-molecular layer of the outer and inner surface of biosorbent in water. A mass of approximately 1 g of finely ground biosorbent (200 µm) was mixed in a 100 ml beaker, 20 ml of distilled water to suspend by an AVW-ADVANCED type magnetic stirrer for a few minutes. Then, the latter will be assayed by a methylene blue solution of known mass concentration (drop by drop) until the persistence of the light blue halo which surrounds the central deposit of the spot formed on the wattman filter paper.

#### 2.3.2. Expression of results

The overall area is related to the volume of methylene blue added. Knowing that by definition, the overall area is proportional to the mass of methylene blue adsorbed per unit area of a given solid material. According to the theory, a molecule of methylene blue occupies an average surface area of 120 Å<sup>2</sup>, so it is possible to calculate the mass area (specific area) via the Avogadro number. The formula is as follows:

$$S_s = \frac{C \times V \times 6,02210^{23} \times 1,210^{-18} \text{m}^2}{M} \quad (1)$$

Where C (mol/l) is the molar concentration of V(L), methylene blue volume, M (g) is the mass of biosorbant.

MEMMERT brand electric dryer followed by grinding. After grinding, the powders of silt skins are obtained after sieving, the particle size of which is between 200 µm and 2 mm using a sieve of ISO-3310-1 mark. BODY: 200mm × 50mm, made in Germany.

### 2.2. Preparation of the adsorbate

Crystal violet in figure 1, a monovalent cationic basic dye with molecular formula C<sub>25</sub>H<sub>30</sub>N<sub>3</sub>Cl. In dye classification it is classified as C.I 42555 and class: basic dye 3. It is molecular weight of 407, 98 g/mol. Stock solution of Crystal violet 1000 mg/L was prepared in double-distilled water and the experimental solutions of the concentration were obtained by successive dilution.

#### 2.4. Determination of equilibrium pH.

The equilibrium pH will be determined by contacting 1 g of biosorbent (200 µm) with 100 ml of distilled water (pH = 6.7). The sample will be stirred at 300 rpm for 24 hours. The pH measurements will be made every 25min using a Schott Gerate pH meter until equilibrium, at the end of 24h, the equilibrium pH is measured again.

#### 2.5. Determination of pH of zero charges (pHpzc)

The pH<sub>pzc</sub> or pH of zero charge corresponds to the pH value for which the net charge of the surface of the adsorbents is zero (Wibowo et al., 2007). This parameter is very important in adsorbent phenomena especially when electrostatic forces are applied in the mechanism. The method for the determination of the zero charge pH consists in placing 50 ml of a solution of NaCl (0.01M) in closed vials after adjusting the pH of each (value between 2 and 12) by addition of NaOH solution (0.1N) or HCl (0.1N). 50 mg of biosorbent sample to be characterized are then added to each of the flasks. The suspensions are stirred at room temperature for 24 hours and the final pH is then determined. Then, we look at a graph ΔpH = f (pHi); the intersection of the curve with the axis passing through zero gives the pH<sub>pzc</sub>.

#### 2.6. Study of adsorption

The study of the adsorption of Crystal violet by the native silt skin powder was done in batch mode at room temperature. This study was done by shaking a series of volumetric flasks containing crystal violet solutions of varying concentration between 5 mg / L and 30 mg / L. The samples were made at time intervals of between 10 and 80

minutes, thus making it possible to determine the contact time between the adsorbate and the adsorbent. The solutions are removed from the stirrer after stirring for 30 minutes and then filtered using wattman filter paper and then proceed to read the optical density of the samples using an UV-visible spectrophotometer of VWR-ADVANCED brand. The adsorption study is carried out at initial pH conditions 6.15; at room temperature 25 ° C.

The study of the influence of pH is carried out by varying the pH values from 2 to 10 and the adjustment was done using the NaCl (0.1N) and HCl (0.1N) solutions. The elimination percentage is given by the following formula:

$$\frac{C_i - C_f}{C_i} \times 100 \quad (2)$$

C<sub>i</sub>: initial concentration of Crystal violet, C<sub>f</sub>: final concentration

The isotherms are given by varying the concentrations of the solution of the Crystal violet from 5 to 30 mg/L. The

calculation of the quantity adsorbed  $q_e$  (mg/g) according to the relation according to:  $q_e$  (mg/g) =  $\frac{(C_o - C_e) \times V}{m}$  (3)

Where C<sub>o</sub> is the initial concentration, C<sub>e</sub> is the equilibrium concentration, V(ml) is the volume of the concentration in purple Hook, m(g) is the mass of biosorbant.

### 3. RESULTS AND DISCUSSION

#### 3.1. Characterisation Physico-Chemical of Biosorbent

According to the **table 1**, the specific and very low surface area compared to the specific surface area of commercial activated charcoal based on 1158 m<sup>2</sup> / g skins obtained by the B.E.T method is found. The low surface area of our biosorbent can be explained either by the nature of our samples or by the method of determining this specific surface. As for the equilibrium pH, an equilibrium pH of about 4.9 is obtained. This pH balance allows us to know the exact pH of our material, which also has an acid character proving its membership in lemons.

Table 1: The specific Area and equilibrium pH

Specific area	Equilibrium pH
6,93	4,9

#### 3.2. IR-FT analysis

The **Figure 2** above shows a 3350 Cm<sup>-1</sup> band characteristic of the OH group of alcohol. Also a band between 2001-1943 Cm<sup>-1</sup> indicating the presence of a triple bond of the alkynes. The band lying between 1454-1416 Cm<sup>-1</sup> indicates the presence of aromatic double bonds and the alcohols vibration band (-OH). The peak at 1210 is characteristic of the phenol groups and located between 1187-1155 Cm<sup>-1</sup> indicating the deformation of the group -OH and -COOH, deformation vibration of COC of the ether group and the

presence of the phosphate group (PO) caused the activating agent which is phosphoric acid H<sub>3</sub>PO<sub>4</sub>. The peak located at 1000 Cm<sup>-1</sup> indicates the presence of the double bond CC of the alkenes. The peak at 872 Cm<sup>-1</sup> indicates the presence of the group NC- amides and 724 Cm<sup>-1</sup> indicates that of amine -NH. The peak appeared between 494-500 Cm<sup>-1</sup> shows the deformation of the halogenes. In fact, we can say through this spectrum FT-IR it presence of groups such as alcohol, aromatic, alkyne, alcene, amide, amine, phenol.

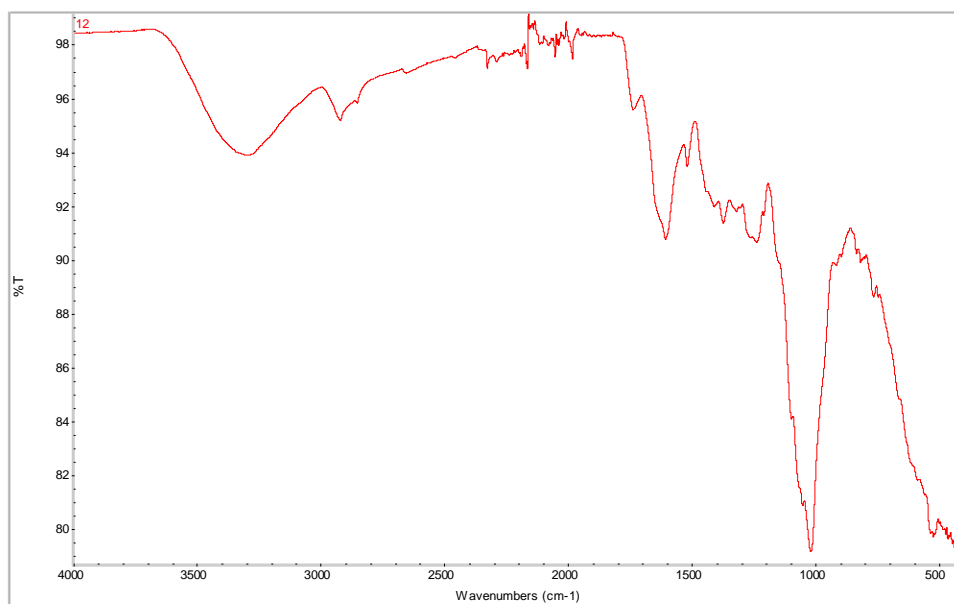


Figure 2: The FT-IR spectrum of the skin powder of citrus limonium (silt).

### 3.3. The pH of zero powder load of dried silt skins.

In **figure 3**, the pH at which the charge of the solid surface is zero is mentioned as the zero point of charge (pHpzc). Above pHpzc the external charge of the adsorbent is negative (Khormaei et al., 2007), the nature of our adsorbents can be acidic or basic depending on the pHpzc values, so the pHpzc depends on the origin of the precursor and of the preparation method which can be chemical and physical, the pHpzc is also a good indicator of the chemical

and electrical properties of the functional groups on the surface of our adsorbents (Baccar et al., 2012). According to this graph, we find the value of the pHpzc of our biosorbent of the order of 5.8. This result is in agreement with the other researchers like Singh and Sukla in 2015. However, the slight differences observed in the pHpzc values can be explained by the nature of our precursors, the method of preparation (Baccar et al., 2012).

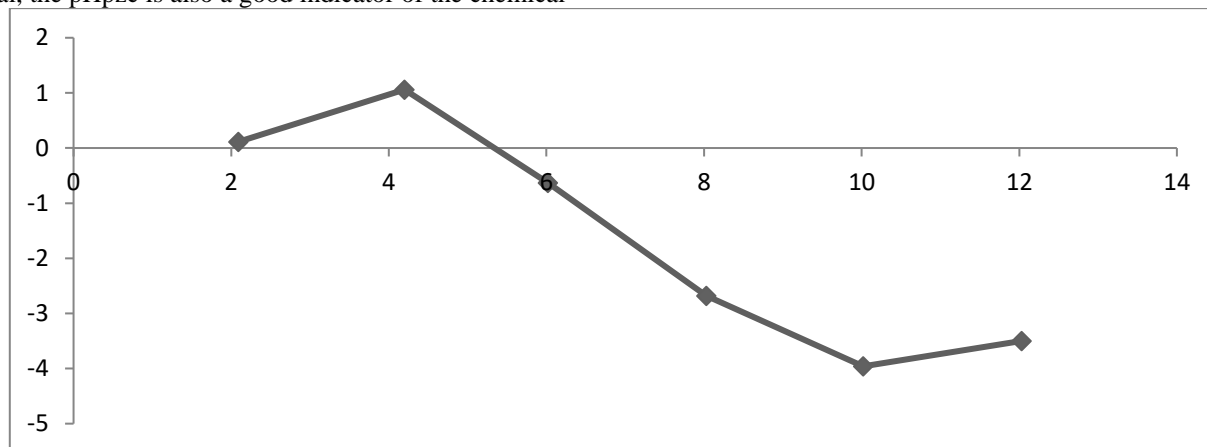


Figure 3: The pH of zero powder load of dried silt skins.

### 3.4. Establishment of the adsorption spectrum of Crystal violet in UV-visible

The adsorption spectrum of Crystal violet in UV-visible was made by a spectral sweep between 320 nm and 590 nm of a Crystal violet solution at 50 mg / L (Lin et al., 2011).

This spectrum in **figure 4** indeed shows us a maximum wavelength which lies in the range 557 nm and 590 nm characterized by a peak around 586 nm. So we considered this value of 586 nm for the rest of our work

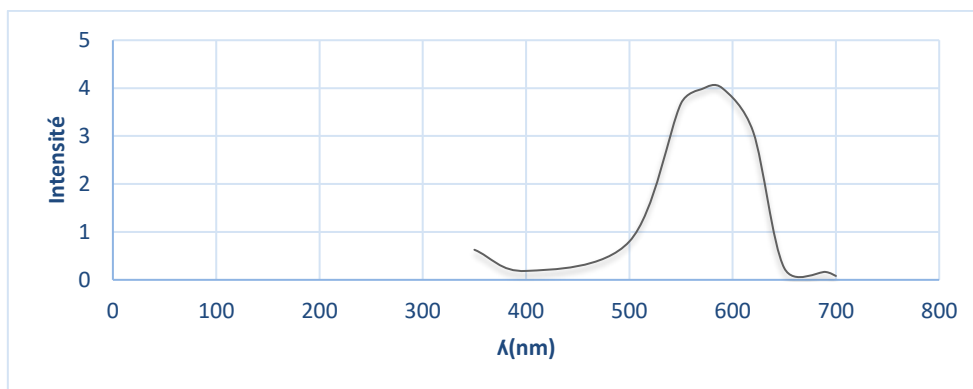


Figure 4: The UV-visible adsorption spectrum of Crystal violet

### 3.5. The influence of contact time

This **Figure 5** represents the percentage of eliminations as a function of the contact time, where the shape of the curves shown is typical of that of saturation but with a fairly high Crystal violet adsorption rate during the first 20 minutes before decreasing and then tending towards saturation (equilibrium plateau). This can be explained by the fact that initially the adsorption sites are vacant, therefore easily accessible to Crystal violet molecules, hence a higher rate of adsorption, in addition to the fact that

the concentration gradient between the solution and the liquid-solid interface is quite high at the beginning of the contacting of the solid and liquid phases.

However, after the initial period, adsorption becomes less due to slower diffusion of the species dissolved through the pores (Joel et al., 2007). The rapid removal of crystal violet molecules is of significant importance, as this will facilitate the use of reduced volumes of adsorbents and will ensure the efficiency and economy of the process.

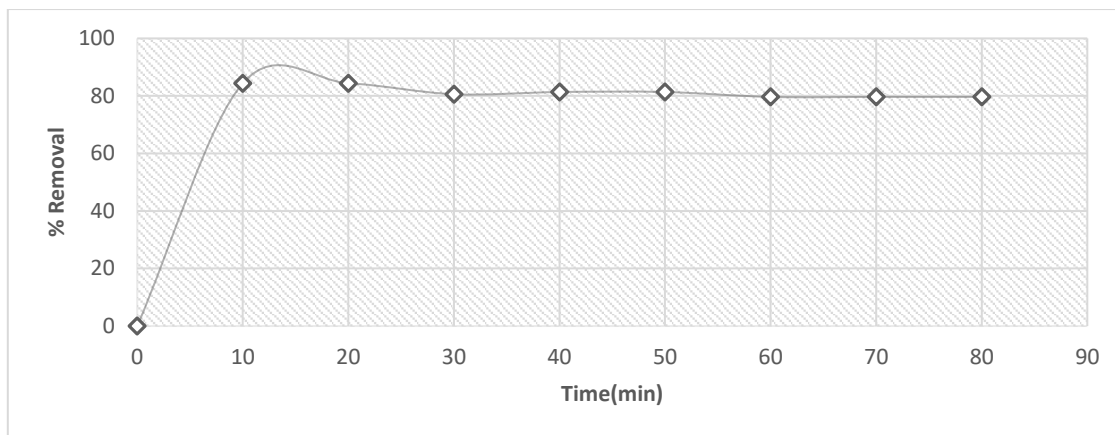


Figure 5: Influence of contact time on Cv adsorption ( $C = 5 \text{ mg / L}$ ,  $m = 0.1 \text{ g}$ ,  $\text{pH} = 6.15$ ,  $T = 25^\circ \text{C}$   $d = 200 \mu\text{m}$ ).

### 3.6. The influence of the mass of the biosorbant

It emerges from this **figure 6** that the adsorbed quantities decrease with increasing mass. Indeed, the interaction between the adsorbent molecules can cause the desorption of crystal violet molecules, narrow sites of the adsorbent (Manohar *et al.*, 2002). On the other hand, when the mass of biosorbant increases, an aggregation / agglomeration of the particles of the biosorbents is formed, thus causing a decrease in the specific surface area (Mohan *et al.*, 2006); this may explain the low adsorbed quantities of Crystal violet when the mass of the biosorbent increases. The

decrease in the adsorbed quantity would also be due to a high availability of the exchangeable adsorption sites or the specific surface (Rais, 2009). However, other authors have worked on the same dye and found the results similar to (Nour *et al.*, 2014, Patil *et al.*, 2011).

Indeed, the amount adsorbed for a mass 0.1 g is maximum for the biosorbent, this value is of the order of 3.9448 mg / g. Thus, the mass of 0.1g for the adsorbent will serve for the continuation of our work. But one of the factors that can influence the retention of Crystal violet molecules is its pH.

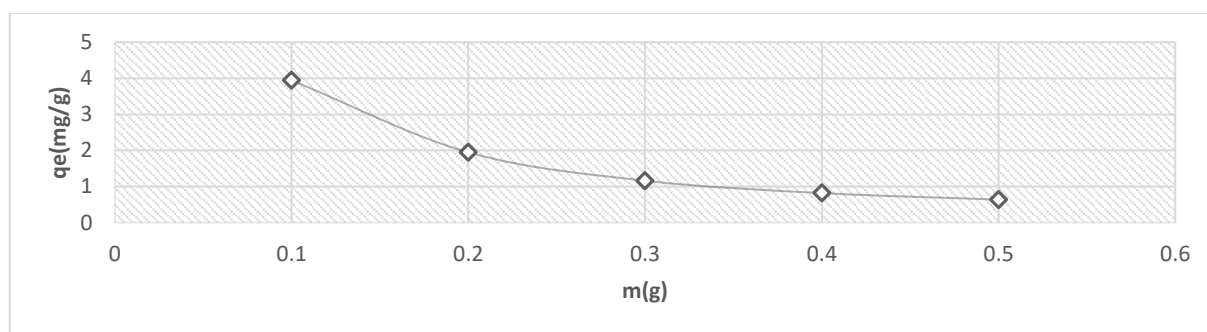


Figure 6 : Influence of the mass of the three biosorbents on Crystal violet adsorption ( $C = 5 \text{ mg / L}$ ,  $t = 30 \text{ min}$ ,  $\text{pH} = 6.15$ ,  $T = 25^\circ \text{C}$   $d = 200 \mu\text{m}$ ).

### 3.7. pH effect

It is good that the pH is a very important parameter intervening in the processes of adsorption, for this we varied the pH over a range going from 2 to 12. According to this **figure 7**, it is clear that the adsorption of the Crystal violet molecules by our biosorbent increases up to pH 5; this would be due to the biosorbent surface which is negatively charged according to the  $\text{pH}_{\text{pzc}}$  value, this can also be explained because at low pH values, the surface of

the adsorbents would be surrounded by  $\text{H}^+$  ions which increases the Crystal violet (cationic pollutant) ions interact with the adsorbent sites. Above pH 5 the adsorbed amount of Crystal violet by the biosorbent decreases slightly to pH 7 caused by the decrease of  $\text{H}^+$  ions, against the high pH, the  $\text{H}^+$  concentration decreases which generates good interaction between dye ions and surface sites (Khalfaoui, 2012). Similar results have been found by (Sakr *et al.* 2014).

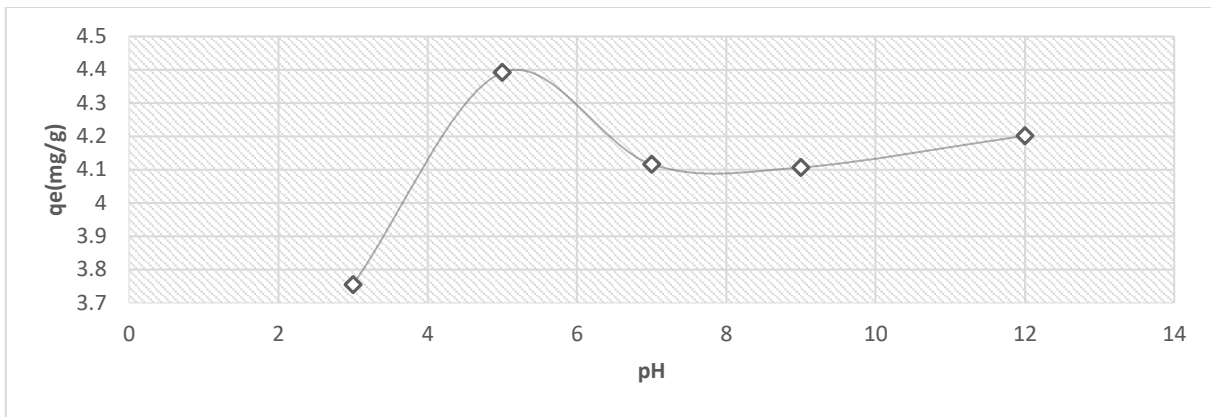


Figure 7 : Influence of pH on the Crystal violet adsorption capacity (C = 5 mg / L, m = 0.1 g, t = 30 min, T = 25 ° C. d = 200 μm).

### 3.8. Influence of concentration

In order to study the effect of Crystal violet concentration on the percentage of elimination by the non-activated biosorbent (cf. **figure 8**), adsorption experiments were carried out in a concentration range varying between 5 and 30 mg / l, for a mass 0.1 g of biosorbent, at a pH of 6.15 and at room temperature. To do this, we see that for our biosorbent the percentage of elimination increases with the crystal violet concentration until reaching a maximum value after a period of 20 min, or 81.36% before being stabilized. This could be explained by the fact that initially the adsorption sites of the biosorbents were widely

available to adsorb the Crystal violet molecules of low concentration or else a large availability of the active sites leading to an increase in the concentration gradient and the rate of diffusion of the analyte to the surfaces of the biosorbent. On the other hand, when the concentration of the analyte increases, the adsorption sites decrease and the percentage of eliminations decrease and become constant regardless of the Crystal violet concentration and this can be explained by the saturation phenomenon of the adsorption sites. on the surface of our biosorbents (Rais *et al.*, 2017, Sayed *et al.*, 2015).

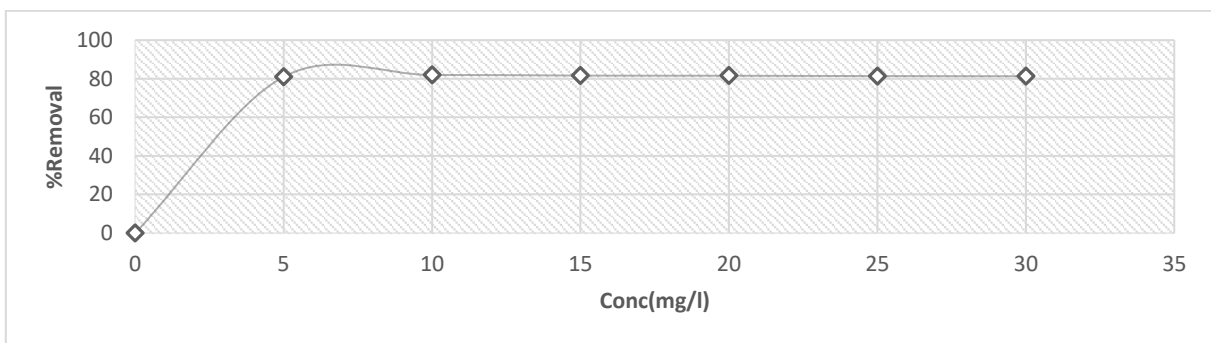


Figure 8 : The influence of the initial concentration on the adsorption of Crystal violet by the adsorbent (m = 0.1 g, pH = 6.15, t = 30 min, T = 25 ° C. d = 200 μm)

### 3.9. Influence of temperature

**Figure 9** shows that the adsorption of CV by our adsorbent increases with temperature. The experiments were carried out for temperatures of 303, 313, 323 K, for a concentration of 5 mg / l and for a mass of 0.1 g of biosorbent. The increase in temperature first favored the diffusion of the Crystal violet molecule through the boundary layer, then on the surface of the adsorbents and finally towards the inside of the pores of the adsorbents. Increasing adsorption with temperature can also be

explained by increasing the numbers of adsorption sites generated by breaking internal bonds at the surface of the adsorbents.

Another explanation would be the favorable effect of temperature on the irreversible adsorption of these adsorbed Crystal violet molecules (Ryoo *et al.*, 1999) or the desolation of Crystal violet molecules and an increase in active centers for adsorption. (Manohar *et al.*, 2002). This result is similar to that obtained by Mouthe in 2017.

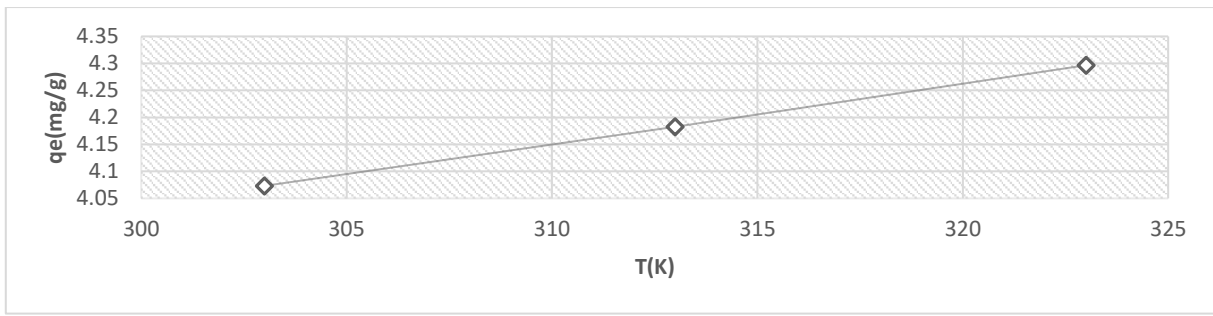


Figure 9: Influence of the temperature on the adsorption of Crystal violet (C = 5 mg / L, m = 0.1 g, pH = 6.15, t = 30 min, T = 25 ° C d = 200 μm).

### 3.10. Isotherms adsorption

From **Table 2** and **Figure 10**, we observe that the Freundlich model agreement with the experimental data is satisfactory for the adsorption of Crystal violet by our biosorbent. The R<sup>2</sup> coefficient value presented in the following table shows that the Freundlich isothermal linear model is best for describing the adsorption of Crystal violet

– It is also characterized by localized site adsorption;

by the adsorbent. This model of Freundlich allows us to conclude that:

– Crystal violet adsorption by our biosorbent consists of an exponential distribution of adsorptive site energies at the surface of biosorbent (Cooney, 1990);

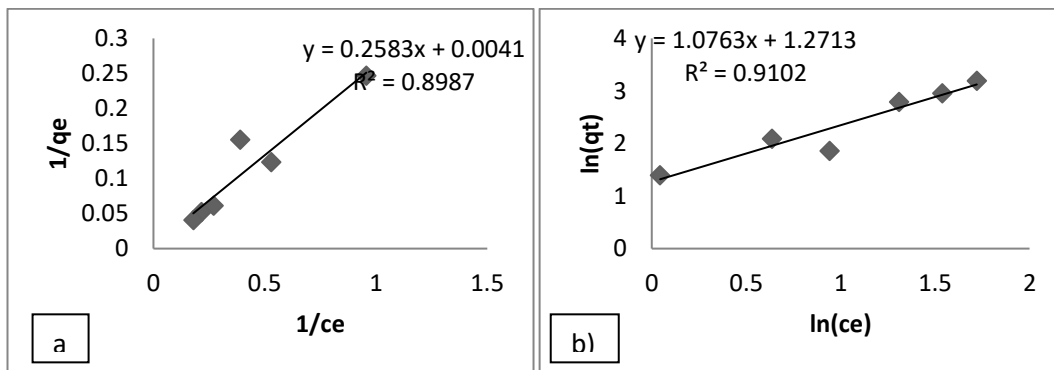


Figure 10: a) Langmuir isotherm and b) Freundlich isotherm.

**Table 2:** Summary of the isotherms of adsorption of Langmuir and Freundlich

Langmuir isotherm			Freundlich isotherm		
$q_{max}(mg/g)$	$K_L(1/mg)$	$R^2$	$K_F$	$\frac{1}{n}$	$R^2$
3,8714	63,0010	0,8987	3,5654	1,0763	0,9102

### 3.11. Kinetics of adsorption.

The validity of the kinetic models is examined from the value of R<sup>2</sup> of the linear regression coefficient reported in **Table 3** above. It can be seen that the application of the pseudo-second-order kinetic model makes it possible to obtain regression coefficients greater than 0.90 in **figure 11**. Moreover, it is found that the values of Q<sub>e</sub> (exp) and Q<sub>e</sub>

(cal) are very close to one another for the biosorbent. This indicates that the adsorption is done in two steps:

- The first step is the diffusion of Crystal violet molecules to the surface of the biosorbents
- The second step is the interaction of Crystal violet molecules on the surface of the biosorbents.

Table 3: Summary of the kinetic parameter pseudo-first and second order.

Kinetics pseudo-first order				Kinetics pseudo-second order			
R <sup>2</sup>	Q <sub>e</sub> (exp)	Q <sub>e</sub> (cal)	K <sub>1</sub> (min <sup>-1</sup> )	R <sup>2</sup>	Q <sub>e</sub> (exp)	Q <sub>e</sub> (cal)	K <sub>2</sub> (g.mg <sup>-1</sup> min <sup>-1</sup> )
0,9013	3,7643	0,8175	0,0483	0,999	73,7643	3,745	0,5862

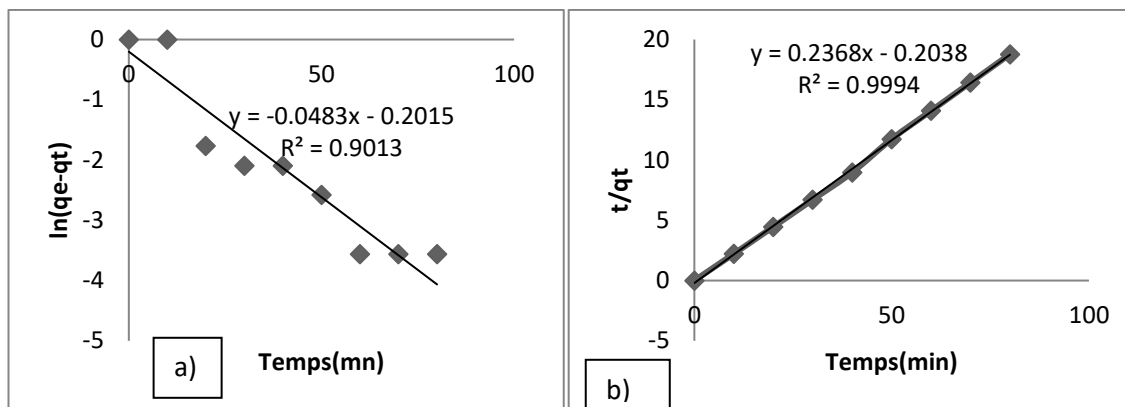


Figure 11: a) Kinetic pseudo first order; b) Kinetic pseudo second order

3.12. Thermodynamic study.

These **Figure 12** and **table 4** reveals that, the positive sign of enthalpy shows that the reaction between the crystal violet molecule and the biosorbent surface is exothermic. The increase in temperature thus serves to increase the diffusion of the crystal violet molecule to the surfaces of the adsorbent (Chun and Chen, 2002), the temperature then acting as a catalyst.

Positive values of ( $\Delta rS^\circ$ ) indicate an increase in adsorption disorder; this is not possible because the adsorption involves a reduction of the adsorbed Cv molecule. ( $\Delta rS^\circ$ ) would then describe the total phenomenon including diffusion (Patil et al., 2011, Senthilkumar et al., 2006).

Negative values ( $\Delta rG^\circ$ ) show that the reaction is spontaneous. It can be seen that the values of ( $\Delta rG^\circ$ ) decrease with increasing temperature. In addition, the values of ( $\Delta rG^\circ$ ) are between -20 and 0Kj / mol, which allows us to justify that the adsorption of Crystal violet on the surface of the adsorbent is physical. Increasing the amount adsorbed with temperature indicates that the system needs energy to overcome the kinetic activation energy. The low values of ( $\Delta rG^\circ$ ) indicate that the bonds are low in energy. So the intermolecular forces are those involved in the adsorption of Crystal violet by the adsorbents.

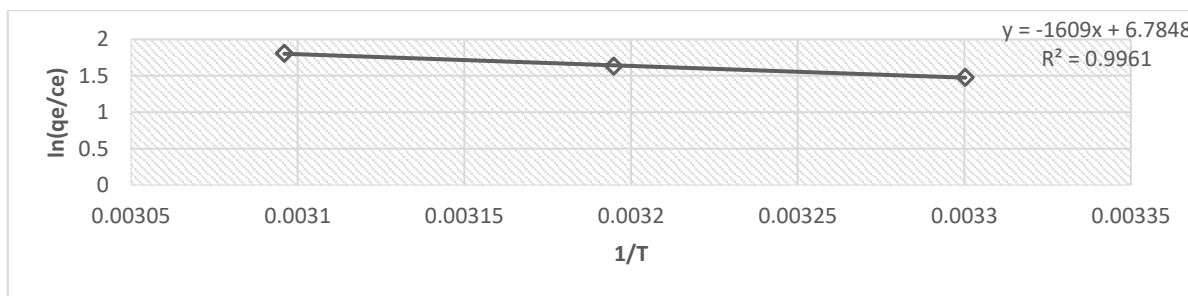


Figure 12: Linear form of thermodynamic parameter.

Table 4 : Summary of the values of constant thermodynamics

R <sup>2</sup>	Températures	$\Delta rG^\circ$ ( $\frac{Kj}{mol}$ )	$\Delta rH^\circ$ ( $\frac{Kj}{mol}$ )	$\Delta rS^\circ$ ( $\frac{Kj}{molK}$ )
0,9961	303 K	-3,38	13,38	0,056
	313 K	-4,148		
	323 K	-4,708		



#### 4. CONCLUSION

The main objective of this work was to prove the natural adsorbent potential of silt skin powders in the treatment of wastewater containing Crystal violet to improve the living conditions of the populations.

In the light of the results, it appears that the properties of the silt skins have a very good Crystal violet retention capacity confirming a high affinity of Crystal violet to be adsorbed on the biosorbent. Linear regressions showed that kinetics are controlled by the pseudo-second-order kinetic model. This is clear and confirmed by the values of the correlation factors corresponding to each model. The effect of temperature on the retention of Crystal violet molecules by non-activated silt skin powders has shown that the process is exothermic and physical. The negative values of the Gibbs energy ( $\Delta G < 0$ ) indicate that the adsorption process of this dye by the studied system is spontaneous.

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