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Preparation and Characterisation of adsorbent prepared from sewage sludge for removal of methylene blue

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ABSTRACT/RESUME

Abstract: *The objective of this study is to valorize an urban waste, the sludges from the wastewater treatment plant of Boumerdes/Algeria for the removal of methylene blue dye in aqueous solution by adsorption. The material made from this sludge has been characterized by various techniques: X-ray fluorescence microscopy (XRF), X- ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and scanning electron microscope (SEM) to obtain information concerning their structures and their compositions.A systematic study of various parameters such as: effect of adsorbent dosage, contact time and initial dye concentration was carried out in order to optimize the ideal conditions for a good adsorption of the studied pollutant; especially the kinetics of adsorption.The exploitation of experimental results using different kinetic models and adsorption isotherms showed that the sludge has similar adsorption characteristics to those of activated carbon, which perfectly confirms the applicability of the activated sludge in the wastewater treatment.*

I. Introduction

Water is a vital source for humanity. Multiple uses use this complex and fragile environment: food needs, domestic, industrial, agricultural and tourist uses. Human activities produce wastewater containing fecal micro-organisms and various contaminants (organic matter, nitrogen, phosphorus, mineral and organic micro-pollutants ...) whose load often exceeds the self-purifying power of the receiving waters (water courses ...), inducing a risk for human health and ecosystems [1]. Wastewater is collected and transported to sewage treatment plants via the sewer system. At the outlet of a sewage treatment plant (WWTP), there is purified water (rejected into the natural environment) and wastewater, consisting especially of sewage sludge containing mineral and organic substances. Sewage sludge, an inevitable by-product generated in wastewater after treatment operations, is a kind of colloidal sediment containing harmful pollutants of pathogens, microorganisms and heavy metals [2] In a few figures, the production of sludge in Algeria is estimated at 400 000 T of MD on the horizon 2020, producing 16 000 T / year of nitrogen and 20 000 T / year of phosphorus, could thus to contribute to the fertilization of 200 000 ha of agricultural land (2.5% of the Algerian productive land) [3]. In the face of stricter statutory pressure and increasing public opposition, a low cost innovative and environmentally convivial alternative to sludge removal is needed [4]. With the growing resource in matter and environmental protection requirements associated with sustainable development it has become necessary to explore all possibilities for the reuse and recycling of industrial and urban wastes and by-products, in particular in the fields of chemistry and engineering. This is a way to reduce management costs in special factories [5-6]. In addition, waste reuse represents a sustainable solution to the scarcity of raw materials to be used for energy production and various end products [7-8]. The intensive use of dyes in the various industrial sectors (textiles, tannery, printing, etc.) causes environmental deterioration. These products transported by wastewater present significant risks to aquatic ecosystems; they are therefore classified as major pollutants [9-10]. The rejections of colored effluents into the nature are not only disagreeable to the environment but also affect many biological cycles [11]. These releases present a real danger to humans and their environment because of their stability and low biodegradability [12, 13]. It is therefore essential to limit pollution as much as possible by introducing a suitable treatment process and integrating a fading unit [14]. Several treatments have been used to reduce the harmful effect of effluent rejected. Traditional processes such as biological processes produce unsatisfactory results, due to the composition of these toxic and colourant rejects which are difficult to biodegrade [15]. These dyes are removed by economically acceptable methods using several techniques: biological treatment for biodegradables [16, 17], coagulation, adsorption on activated carbon [18, 19], by electrochemical method [20] and by the membrane processes [21]. A wide variety of physical, chemical and biological techniques have been developed and tested in the treatment of dye-laden effluents. These processes include flocculation, precipitation, ion exchange, membrane filtration, irradiation and ozonation. However, these procedures are costly and lead to the generation of large quantities of sludge or the formation of derivatives [22]. Among the processes for the treatment of liquid discharges, adsorption remains a relatively used and easy to implement technique. The removal of dyes in aqueous solutions by adsorption on various solid materials, Researchers have thus shown that a wide variety of materials of natural or biological origin had the ability to fix large quantities of organic pollutants present in water [23]. At present, activated carbon is considered to be one of the most versatile adsorbents and many studies show its effectiveness, but its use is limited because of the difficulties of its regeneration and its high cost [24]. Activated carbon is the most widely used adsorbent because of its high adsorption capacity of organic materials [25]. Several studies have shown a high efficiency in the treatment of waste from the textile industry, from certain rustic materials, or by-products of the industry, such as charcoal [26], sunflower stems [27], sugar cane waste [28], olive kernels [29], seed shells [30], sunflower seeds [31], cane-trash of sugar cane [32], almond shells [30], peach cores [33], grape seeds [34], apricot kernels [32], cherry kernels [34], walnut shells [35], corn husks [36].In the present work, we were interested in the idea of testing and valorizing an adsorbent material based on sewage sludge from the Boumerdes region, which is abundant in our country for the removal of a cationic dye, methylene blue. This adsorbent was used in the natural state. Various experimental parameters were analyzed: mass of adsorbent, initial concentration of dye and contact time. The adsorption capacity of raw sludge was studied using Langmuir, Freundlich and Tempkin isotherms.

II. Materials and methods

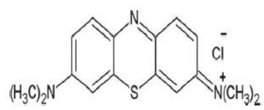
II.1. The adsorbent:

The sewage sludge used in this study (sludge taken out of the filter press) was gathered from the urban wastewater treatment plant located in Boumerdes/Algeria. The samples were in the open air and then in an oven at 105 ° C, crushed by a mechanical crusher to obtain sewage sludge powder After that, the sample was sieved To retain only the fraction between 100 µm and 200 µm. Resulted samples of sewage sludge were washed several times with distilled water, dried at 105°C, crushed, sieved once again and kept in the air-tight plastic bag until they are used..

II.2. Preparation of the adsorbate:

The textile dye used in this study is methylene blue(BM) which is a cationic dye most commonly used in dyeing cotton, wood and paper, [37, 38] it exists as a dark green powder in several hydrated forms: Monohydrate, Dihydrate, Trihydrate and Pentahytrate, [39] The most common is trihydrate [40]. Several researches have been published on the possibility of elimination of methylene blue by different adsorbents [41, 42, 43.44]. The following table summarizes the main physico-chemical properties of the dye.

Table 1. General characteristics of methylene blue (MB).

Name	Methylene blue (MB)
Family	Basic dye
chemical formula	C16H18N3SCl
Chemical name	3,7-bis-(dimethylamino) phenazathionium
Molecular Weight	319.86 g/mol
λ_{max}	664 nm
Solubility in water	high
Steam voltage	low
Structure	

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The solution was prepared by dissolving the amount of methylene blue (MB) in distilled water. The study of its visible UV spectrum at wavelengths between 200 and 800 nm, Using a spectrophotometer type T90 + .UV / Vis spectrometer PG Instruments Ltd, a dye solution at a concentration of 20 mg / l was used to determine the wavelength corresponding to the maximum of absorbance, λ_{max} = 664 nm.

II.3Adsorption experiments:

The adsorption experiments were performed in batch mode. The adsorption of the cationic dye (MB) on the studied adsorbent was performed by introducing a quantity of 0,2g in suspension of dried sludge in solutions of 50 ml of the MB of initial concentration C_0 (20mg. L^{-1}). The mixture was subjected to agitation of 300tr/min at room temperature. The samples were performed every 5 minutes. The coloured solution was separated from the adsorbent. The samples collected were filtered on Millipore membrane of type 0.45 µm. The absorbance of the filtered solution was measured using spectrophotometer type T90 + UV/Vis spectrometer PG Instruments Ltd at the wavelength corresponding to the maximum absorbance of the sample (λ_{max} = 664 nm).

Adsorption capacity was calculated by Eq. (1) [45]

$$q_e = \frac{(C_0 - C_e)V}{m} \quad (1)$$

Removal efficiency was calculated by Eq. (2) [46]

$$R\% = \frac{C_0 - C_e}{C_0}\,100 \quad (2)$$

Where q_e: adsorption capacity (mg.g^{-1}), C_0: initial dye concentration (mg l^{-1}), C_e: equilibrium dye concentration (mg. l^{-1}), V: volume of the solution (l), m: mass of the adsorbent (g), R: removal efficiency (%).

II.4. Characterization methods:

II.4.1. X-ray fluorescence spectroscopy:

The chemical analysis of the dried sewage sludge were performed using the Bruker-Axs: S8 TIGER sequential spectrometers (wavelength dispersve). The result were grouped in table 2.

II.4.2. Fourier transform infrared spectroscopy (FTIR):

Fourier Transform infrared spectroscopy (FTIR) analysis was performed in the range 500-4000cm-^{1}, thanks to a JASCO FT-IR 4100 spectrometer. (Figure1).

II.4.3. X-ray Diffraction (XRD):

To identify the predominant phases of the material studied, X-ray diffraction analyses were performed using the Diffractometer PANalytical XPERT-PRO with a PIXcel-1 D detector sensor. The result of the analysis was given in Figure 2.

II.4.4. Scanning electron microscopy (SEM):

The physical morphology of the sludge was observed using a JEOL. JSM-6830 scanning electron microscope coupled with dispersive energy analysis (EDX) (Energy dispersive X-ray spectroscopy (EDXS).)

III. Results and Discussion:

III.1. Chemical characterization of the material

The results of the semi quantitative chemical analysis of the raw dried sludge expressed as mass percentage (%) were reported on table 1.They show that SiO_2 was the main constituent of the dried sludge with a content of 32.08%, the presence of aluminium oxide, lime and iron oxide were in the order of 7.49%, 5.85% and 4.15% respectively. The phosphorus content in the raw sludge was 2.52% which gives it the character of fertilizer for agricultural purposes. A relatively small presence of the other elements (Na_2O, MgO, SO_3, K_2O, TiO_2, MnO, and Cl) was identified.

Table 2. Chemical composition of dried sewage sludge.

Elements	Percentage%
SiO_2	32.08
Al_2O_3	7.49
Fe_2O_3	4.15
K_2O	1.15
Na_2O	1.17
CaO	5.85
MgO	1.45
TiO_2	0. 32
SO_3	1.87
Cl	0.23
P_2O_5	2.52
MnO	0.12
LOI	42.08

III.2. FTIR:

The FTIR spectra obtained for the sample was shown in Figure 1. The analysis of this spectrum reveals different absorption bands of the functional groups of the raw sludge. The maximum positions of the sample are noticed at 3388.60 cm^{-1}, 2924.31 cm^{-1}, 1651.19 cm^{-1}, 1537.76 cm^{-1}, 1425.25 cm^{-1}, 1034.92 cm^{-1}, the spectrum has wide and stacked bands in the region 3600-3200 cm^{-1}, which are due to elongation of the O–H bonds. The bands at 3388.60 cm^{-1} can be attributed to the presence of –OH and –NH groups while the peak at 2924.31 cm^{-1} can be assigned to the C-H aliphatic vibration while peaks between 600 cm^{-1} and 1000 cm^{-1} can be attributed to aromatic vibration NS C-H [47]. The bands at 1651.19 cm^{-1} to 1537.76 cm^{-1} and 1425.25

cm^{-1} represent the carboxylic acid groups C = O and the stretching of the C-O carboxylate groups. The band at 1034.92 cm^{-1} can be assigned to groups of alcohol. [48]. Absorption peaks in the region of wavelengths below 800 cm^{-1} can be attributed to nitrogenous Bioligands [49].

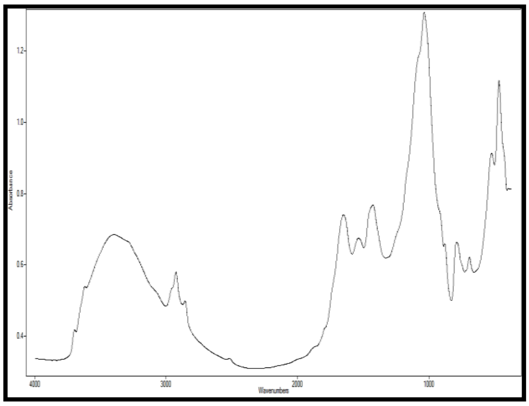

Figure1. *Fourier transform infrared spectrum of raw sludge.*

III.3. X-ray Diffraction (DRX):

The analysis of the X-ray diffraction spectrum of the dried sludge shows the presence of different crystalline phases including significant quantities of quartz and calcite. In addition, we note a significant percentage of 53.5% of organic matter.

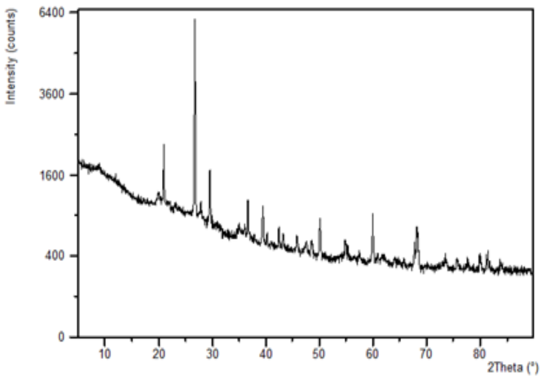

Figure2. *XRD patterns of raw sludge.*

III.4. Scanning electron microscopy (SEM):

Scanning electron microscope (SEM) was used to observe the texture of the material made from raw sludge. According to Figure 3, which shows the image of the sample, clearly visible grain aggregates were observed and the pore structure was not very significant.

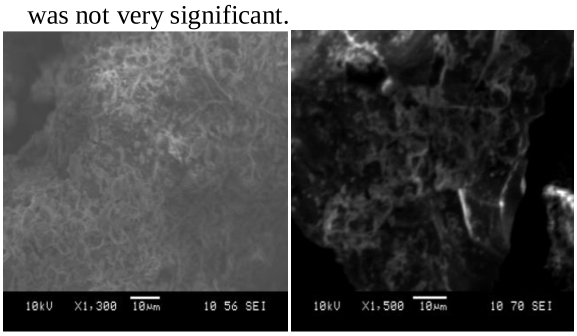

Figure 3. *Scanning electron microscope of raw sludge.*

III.5. Effect of adsorbent dosage:

In order to examine the influence of the solid / liquid ratio, the initial amount of the adsorbent is varied while keeping the dye concentration in the solution constant (20 mg / l). The study of the influence of the MB mass on the adsorbed quantities of this pollutant was represented by the curve of figure 4.

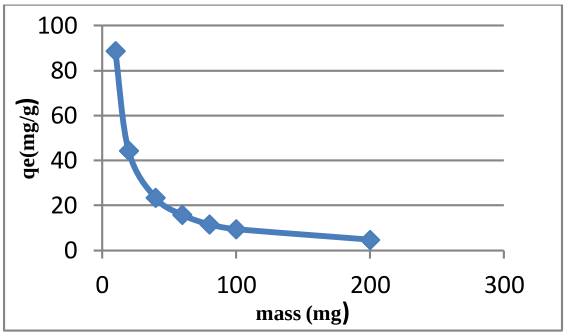

Figure 4. *Effect of adsorbent dose*

According to Figure 4, there was a significant effect of the adsorbent mass on the adsorbed amount of the dye. It was noticed that for a concentration of 20 mg/L of methylene blue during a contact time of 120min, an increase in the mass of sludge from 10 to 200 mg involves a decrease in the adsorbed amount expressed in milli gram per gram of the dye adsorbent. Indeed, the decrease in the concentration of suspension raw sewage sludge causes in the dispersion of the grains in the aqueous phase. As a result, sorbantes surfaces will be more exposed. This will facilitate the accessibility of a wide number of free sites from the support to the molecules. We also notice from the analysis of the results shown in Figure 5 that the percentage of removal of methylene blue increases with the increase in the mass of the adsorbent to stabilize at a value close to 0.2 g of the dried raw sludge.

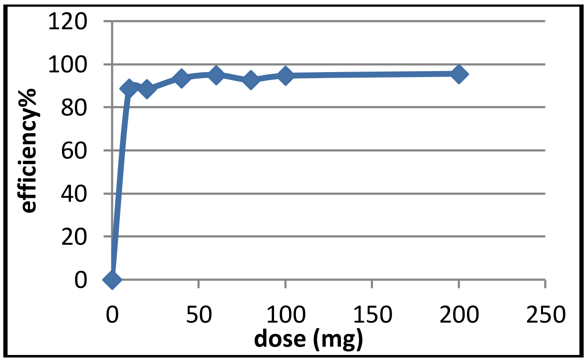

Figure 5. *Effect of sludge dose on methylene blue removal efficiencies*

III.6. Effect of contact time:

To determine the time required to attain the MB adsorption equilibrium, experiments were realized on a volume of 50 ml of a MB solution at a concentration of 20 mg / l, at initial pH and with a report of 4 g /l of dried sewage sludge. At regular

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time intervals, samples were performed. After filtration, the samples were analyzed by UV / Vis Spectrophotometer at λ = 664 nm. Figure 6.

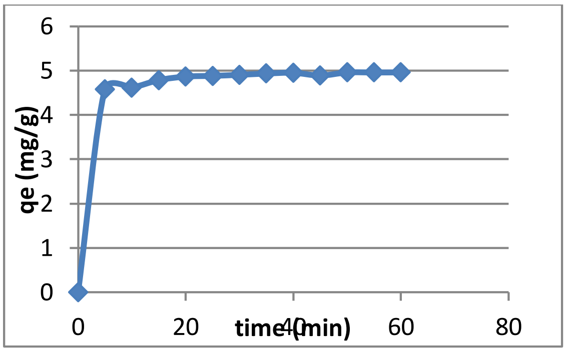

Figure 6. *Effect of contact time*

Analysis of the kinetic curve of methylene blue retention by raw sewage sludge shows that the process of adsorption of the dye was manifested in two distinct stages: the first part of the kinetics corresponds to a rapid phase, where the fixation of the Methylene blue was carried out within the first 20 minutes of adsorption while the second stage was of average speed until reaching the saturation level. The rapid adsorption kinetics during the first few minutes of reaction, can be explained by the existence of easily accessible sites probably located on the outer surface of the adsorbent material, followed by a second slow stage interpreted by an internal diffusion of the dye molecules in the pores difficult to reach, or inside the organic matter before reaching an adsorption equilibrium where all the sites become occupied. In all tests, the contact time was maintained at an hour as maximum, time needed to obtain the adsorption equilibrium of this dye.

III.7. Effect of the initial dye concentration:

Figure7. Represents the evolution of the initial concentration of the MB as a function of time. it was noticed that the increase in the initial dye concentration involves in an increase in the amount adsorbed to the surface of the adsorbent material used. It was also noticed that the initial speed of adsorption increases with the concentration, this to indicate that if the initial concentration of methylene blue in the solution was low, it was adsorbed only on the surface to form a monolayer during a very short time [50] and when the initial concentration is high, there will therefore be more molecules that will diffuse to the surface of the sites of the adsorbent particles that consequently the retention becomes more important [51, 52].

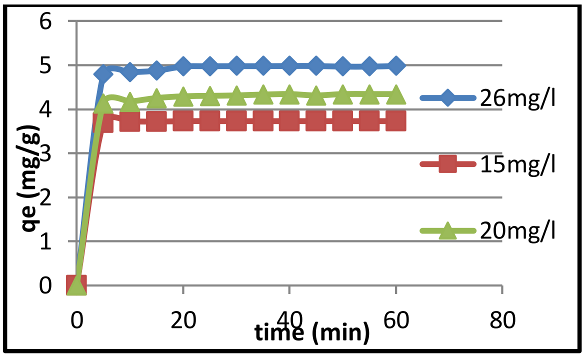

Figure 7. *Effect of the initial dye concentration*

IV. Adsorption isotherms:

Adsorption isotherms play an important rule in the determination of maximum adsorption capacities and in the design of new adsorbents; it is therefore indispensable in our study to determine them. The results of the equilibrium methylene blue adsorption study on the sewage sludge were treated according to the linear equations of Langmuir, Freundlich and Tempkin. The purpose of this this linearization was to be able to verify the model according to which the adsorption takes place and to deduce the maximum adsorbed quantities as well as the affinity of the adsorbate for the adsorbent.

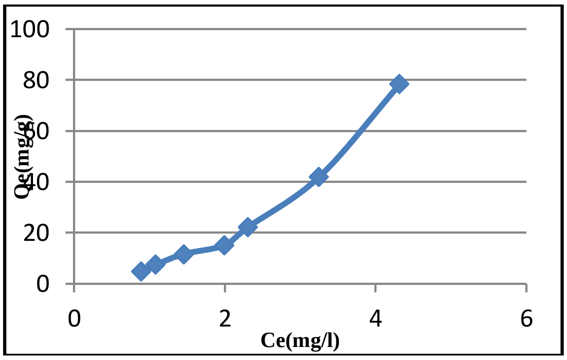

Figure 8.*Adsorption Isotherm of Methylene Blue*

IV.1. Langmuir isotherm :

$$q_e = \frac{q_m b C_e}{1 + b C_e} \qquad (3)\ [53]$$

Linearization of equation (3) gives equation (4):

$$\frac{1}{q_e} = \frac{1}{q_m} + \frac{1}{q_m b C_e} \qquad (4)$$

Where q_m is the maximal adsorption capacity (mg. g^{-1}), b is the Langmuir constant.

IV.2. Freundlich isotherm:

$$q_e = K C_e^{\frac{1}{n}} \qquad (5)\ [54]$$

Linearization of equation (5) gives equation (6):

$$log q_e = log K + \frac{1}{n} \log C_e \qquad (6)$$

Where n and K are the Freundlich constants.

IV.3. Tempkin isotherm :

The linear form of the Tempkin isotherm is given by the following equations [55]:

$$q_e = \frac{RT}{b}\ln(A_T C_e) \qquad (7)$$

$$q_e = \frac{RT}{b_T}\ln\ A_T + \left(\frac{RT}{b}\right)\ln C_e \qquad (8)$$

$$q_e = Bln\ AT + Bln\ C_e \qquad (9)$$

Where AT is Tempkin isotherm equilibrium binding constant, (L/g) b_T is Tempkin isotherm constant, R is the gas constant (J/mol/K), T (K) the absolute temperature. B is the Constant related to heat of sorption (J/mol).

Adsorption equilibrium constants and linear regression coefficients obtained from Langmuir, Freundlich and Tempkin isotherm models are presented in Table 3. The linear plots of the Studied models are shown in Figure 8.

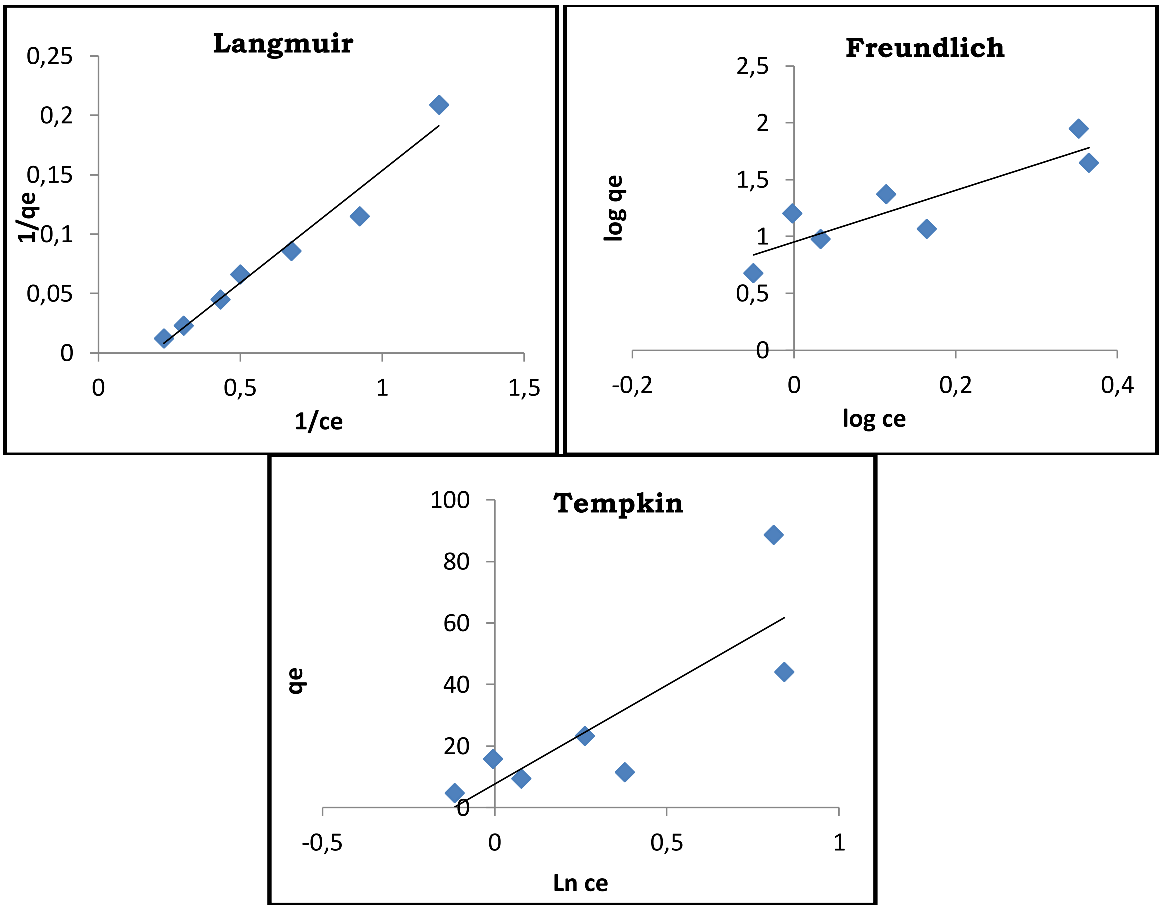

Figure 9. *Linear plots of studied isotherm models at constant temperature 22 ℃ and pH=7.*

Table 3. Parameters of the studied isotherm models

Langmuir			**Freundlich**		
q_m(mg/g)	b(L/mg)	R^2	$K((mg/g)/(mg/L)^{1/n})$	n	R^2
5.29	-5.40	0.964	2.585	0.440	0.779
Tempkin					
K_T	b(L/mg)	R^2			
1.126	64.27	0.683			

According to the results given in table 3 we observed that the Langmuir linear model is the most credible model that better describes the adsorption compared to the Freundlich and Tempkin isotherms with a highest value of R^2 = 0.964 by the raw sludge. Therefore, the adsorption of the methylene blue onto the dried raw sewage sludge surface was performed principally on monolayer homogeneous surface with a maximal adsorption capacity of 5,29 mg/g.

V. Conclusion

The main objective of this study is focused on the use of sewage sludge, an abundant and low-cost material by an efficient method capable of limiting the pollution generated by an industrial cationic dye case of methylene blue. The adsorption process removal of MB dye from aqueous solutions has been investigated under different operating conditions of pH, temperature and agitation speed. The results show that the adsorption of this effluent is influenced by several factors, allowed us to fix them at 60min for contact time, ambient temperature 22°C, pH 7 and the adsorbent mass of 0,2 g. The modeling of experimental data revealed that the adsorption has obeyed to the Langmuir isotherm with a correlation coefficient of 0.964 and a maximal adsorption capacity of 5.29 mg/g. Finally The different results obtained in this study are promising and encouraging to consider has allowed us to suggest that the sewage sludge used is effective in the removal of methylene blue and that this new material produced has similar adsorption characteristics to those of activated carbon confirms the use of sludge as an economic industrial adsorbent in the field of wastewater treatment.

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