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Elaboration of Benin clay based geopolymer: Application to congo red adsorption in an aqueous medium

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Abstract

In this work, clay based geopolymer was prepared and applied for anionic dye (congo red) removal in water. Clay (kaolin) from Adjozounme in Ketou from Benin was used as aluminosilicate material and NaOH solution was alkaline activator in the molar ratio n(NaOH)/n($NaAlO_2$)/n(Na_2SiO_3, $5H_2O$) = 0.3/0.3/0.8 respectively. Then we added 0.5g of CTAB and 1.5g of metakaolin obtained by calcining kaolin at 800°C. Thus, two geopolymer samples were developed by varying the polymerization time (48 hours and 72 hours). X-ray diffraction (XRD) analysis showed that the two geopolymer sample could be amorphous. Measurements of parameters such as geopolymer mass, pH and kinetics revealed that this congo red can be removed up to 80% from water with a maximum adsorption capacity of 129.31mg/g under optimal conditions of 60 minutes of contact at pH ≤ 4. Adsorption is described by pseudo-second order kinetics. The implementation of this sector is essential to reduce the costs of treating industrial effluents.

Keywords: Geopolymer, Kaolin, anionic dye, congo red, Adsorption.

Introduction

Scientific progress in the fields of chemistry, clothing or plastic art, textiles, printing, foodstuffs, medicine, etc., has led to the discovery and abundant use of a multiplicity of dyes. The majority of these dyes are compounds that are difficult to biodegrade, often toxic or harmful to living beings[1,2]. They then constitute micropollutants whose bioaccumulation could have harmful consequences on the body. The reduction or even the total elimination of these micropollutants is therefore a necessity due to the environmental danger they constitute in the short or long term. Congo red is an anionic (acid) dye belonging to the azo family. Initially used with great success in the textile industry, in histology (for the staining of eosinophil cells) in mycology (study of fungi), congo red is gradually abandoned because of its toxicity. Today, it is more used as a pH indicator[3]. Like dyes from the azo family, congo red is toxic. By contact with the skin, it causes burns, it is fatal at high doses of ingestion and carcinogenic by bioaccumulation[4]. The interest of our work is to find an optimal process for eliminating this dye from textile effluents in order to be able to take advantage of its great initial success. Operational treatment systems on a laboratory or industrial scale exist including physicochemical, chemical and biological processes[5]. But these treatments are for the most part very expensive, failing or not very effective in the elimination of micropollutants[6].

Given the economic difficulties experienced by developing countries such as Benin, in terms of sanitation and wastewater management, the use of other treatment techniques that are less expensive and simpler to manage is essential. Thus, the use of amorphous geopolymers based on clay (kaolin) from Benin as an adsorbent could then constitute a simple, sustainable, efficient and more economical alternative for the elimination of dyes from water. And this, because of the adsorption properties developed by this material[7] and the great availability of clay in Benin[8].

It is with this in mind that we carried out, on the one hand, a methodological study of the development of an amorphous geopolymer based on clay from Benin; then, on the other hand, a parametric study of adsorption by examining the effect of factors such as the mass of adsorbent, the pH, the contact time on the bleaching power of the material. The application of kinetic models and the study of adsorption isotherms made it possible to characterize the phenomenon of adsorption of Congo red on the geopolymer.

Materials and methods

Work equipment: The raw clay (kaolin) was taken from a working quarry in Adjozounmè, which is a locality in the commune of Kétou whose soils contain clay deposits[8].

The geographical coordinates of the sampling point are: latitude 07°20'55.8" North and longitude 002°34'04.6" East.

In our work, we used as aluminosilicate material: Adjozounmè clay (designated by Adj1) and as basic activator, sodium hydroxide (NaOH)[9,10]. To increase the silica content of the geopolymer, we added sodium silicate (Na_2SiO_3) during activation[11,12]. Furthermore, we used cetyltrimethylammonium bromide (CTAB) as a blowing agent to increase the specific surface of the geopolymer during curing[13,14]. Congo red (RC) is the anionic dye chosen as the adsorbate for this application. A DR 3900 UV-visible spectrophotometer is used for the absorbance measurements.

Work methods: Geopolymer elaboration: In order to eliminate as much as possible the impurities contained in the raw clay, the clay samples taken were subjected to a pre-treatment according to the current approach of grinding-sieving, decarbonation[15] and extraction of the clay fraction. The technique used for the synthesis of geopolymers is the sol-gel process[16]. The kaolin recovered following the previous operation is calcined at 800°C for 2 hours in the oven to obtain metakaolin[10]. In a second phase, an alkaline activating solution of 100mL is prepared according to the molar ratio n(NaOH)/ n($NaAlO_2$)/n(Na_2SiO_3,$5H_2O$)=0.3/0.3/0.8 respectively[10,17]. Then 0.5g of CTAB then 1.5g of met kaolin were added to this mixture, with stirring, successively. Thus, two identical mixtures were produced and stirred (at 180 revolutions/min) using the mini-flocculator, at room temperature, one for a time of 48 hours and the other for 72 hours. At the end of each stirring time, the suspension is removed and centrifuged at 3000 revolutions/min for 10 minutes to recover the precipitate. The latter is washed several times with distilled water until an almost neutral pH is obtained for the supernatant. The precipitate obtained is dried in the oven at 65°C for 24h. A slightly compact amorphous powder is collected and finely ground using a ceramic mortar.

Material Characterization: A sample of the clay fraction and of each geopolymer produced (48 hours and 72 hours) was taken and packaged in a well-sterilized plastic tank bearing the respective indications Adj1, ADJ-G48 and ADJ-G72. These samples were sent to Italy to the ELETTRA Synchrotron where they were subjected to the X-ray diffraction test (XRD), analyzed on the MCX beam line (Material Cristallography bay X Ray), at the wavelength λ = 1.03Å with an energy of 12 KeV, 1D detector.

Procedure for carrying out adsorption tests: After calibration of the dye (Congo red), three parameters were examined, namely: the effect of the mass of the adsorbent, the effect of the pH of the reaction medium and the kinetics of the adsorption reaction. For each parameter, three tests are carried out; mean values and standard deviations are calculated. For the measurements of the various parameters, the volume and concentration of Congo red solution are set at 50mL and 50 mg/L respectively. The colored solution is separated from the adsorbent by centrifugation at 4000rpm for 5min; the absorbance of the supernatant is measured with a UV-visible spectrophotometer at the wavelength of the maximum absorbance of the Congo red solution (λ=500nm)[18]. The residual concentration of the dye is determined using the equation (equation 1) deduced from the calibration curve. The adsorption percentage of RC is evaluated using the formula:

$$R(\%) = \left(\frac{C_o - C_r}{C_o}\right).100 \quad (1)^{19}$$

Where C_o is the initial concentration in mg/L of the CR, C_r the residual concentration, obtained after 24 hours.

The adsorption capacity of RC by the geopolymer is the quantity of dye adsorbed at equilibrium time per gram of adsorbent; it is calculated from the following equation (equation 2):

$$Q_e = \frac{(C_o - C_r).V}{m} \quad (2)^{20}$$

With C_o: the initial concentration in mg/L of the Congo red solution; C_r: the residual concentration measured in mg/L; V: volume used in mL; m: the dry mass in mg of the geopolymer.

The effect of adsorbent mass was studied in a range of geopolymer mass ranging from 10 to 200mg; the solutions were stirred for 24 hours. The influence of pH on the adsorption of RC was studied in a pH interval ranging from 2 to 12; each dose of 50mL of 50mg/L dye solution is adjusted to one of the defined pH values, added with the optimum mass of adsorbent and stirred for 24 hours. Adjustments were made with a decimolar solution of hydrochloric acid or sodium hydroxide.

The effect of time as a function of the adsorbate concentration was studied by introducing into RC solutions of different concentrations (0.5 to 150mg/L) the optimal mass of geopolymer deduced. Its solutions are stirred at 175rpm and samples are taken at 15-minute intervals, for one hour, then, at 30-minute intervals, for one hour 30 minutes; these are centrifuged and the residual concentrations are determined after measuring the absorbances with a visible UV-spectrophotometer.

The kinetic data were analyzed by two kinetic models (first-order and second-order kinetic models) and by two isotherms (Langmuir isotherm and Freundlich isotherm); the characteristic parameters were determined using Excel and ORIGIN-PRO software.

Results and discussion

Geopolymers production: Figure-1 shows the kaolin recovered from the raw clay sample and the metakaolin obtained by calcining the kaolin at 800°C.

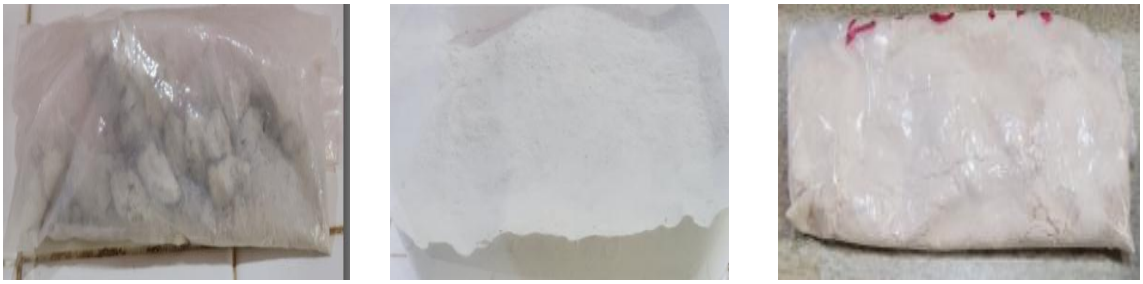
A slightly basic pH between 7.3 to 7.5 was reached after 10 to 12 rounds of washing with abundant distilled water. Here, two geopolymer materials are obtained for the 2 reaction times: ADJ-G48 for the 48-hour geopolymer and ADJ-G72 for the 72-hour geopolymer. They are visibly amorphous white powders (Figure-2)

Characterization of kaolin and geopolymer: Figure-3 showed the diffractogram of the clay sample (kaolin) from Adjozounmè (Adj1). The peaks of this diffractogram indicate that the clay material has a crystalline structure. The chemical analysis by fluorescence of this sample gave the results presented in Table-1. These results reveal that this Adjozounmè clay is rich in silica (SiO_2) and in alumina (Al_2O_3) with percentages of 44 .54% and 37.19% respectively.

These values are very close to those found by Laibi and al. (39.37% silica and 30.90% alumina) on the characterization of Etigbo clay which has a very high kaolinite content (78.1%)[8]. We could then say that the clay of Adjozounmè has the necessary constituents to serve as raw material for the elaboration of geopolymer material.

Figure-4 shows the results of the XRD of the samples of the two geopolymers ADJ-G48 and ADJ-G72. The presence of a large marked bump in the angular range $10° < 2\theta < 25°$ of these diffractograms and the disappearance of the pointed rays could mean that geopolymer is a semi-amorphous material[21]. However, the intensity of the bumps is mitigated by the existence of slight peaks due to the presence of unreacted silicate and quartz residues. What's more, the hump of ADJ-G72 ($10° < 2\theta < 25°$) is the same width as that of ADJ-G48; this could show that with 48 to 72 hours of preparation, a semi-amorphous geopolymer material can be obtained.

The results of the characterization of the elaborated geopolymer materials showed that the adopted elaboration approach makes it possible to obtain amorphous geopolymers, therefore endowed with adsorbent properties. This result is similar to that obtained by Lahcen et al. in a research on the synthesis of a mesoporous geopolymer material based on Moroccan clay rich in kaolinite. By using the surfactant CTAB, the authors obtained anamorphous geopolymer material whose pore volume is more improved than that of the clay raw material[22].



a) Unground reclaimed kaolin b) ground kaolin c) Metakaolin obtained

Figure-1: Extraction of the clay fraction and formation of metakaolin.

a) ADJ-G48 dry in the oven b) ADJ-G48 grind c) ADJ-G72 dry in the oven d) ADJ-G72 grind

Figure-2: The two geopolymers produced.

Table-1: Results of chemical analysis by fluorescence of sample Adj1.

Clay sample	Proportions of the different constituents (in %)									Total
	Si	Al	Fe	CaO	Mg	K	Na	S	CO_2	
Adj1	44.54	37.19	2.57	0.00	0.30	0.27	0.01	0.00	14.77	99.65

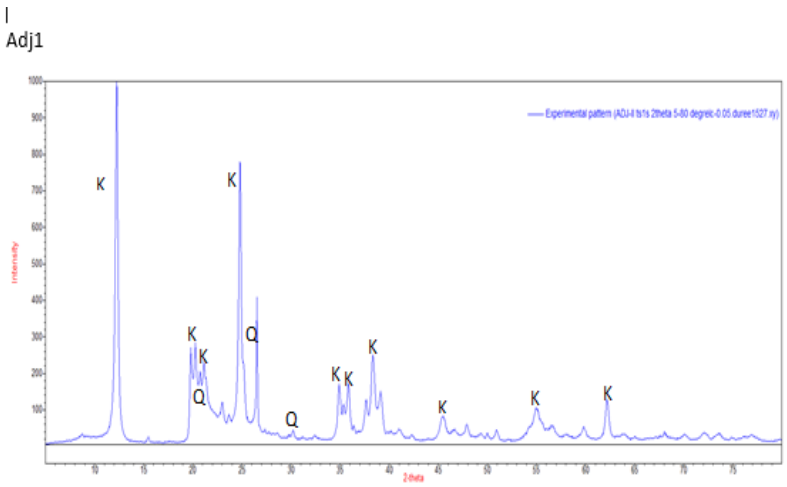


Figure-3: X-ray diffractograms of kaolin Adj1 (K = kaolinite, Q = quartz).

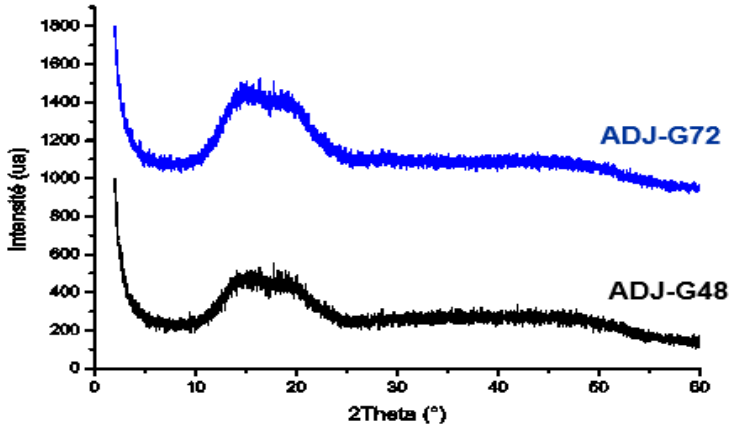


Figure-4: X-ray diffractograms of ADJ-G48 and ADJ-G72.

Application to the adsorption of congo red: The 72-hour geopolymer (ADJ-G72) was used for the adsorption tests. The standard deviations calculated from the test results being low, this assumes that the data is homogeneous and therefore usable. The average values are then used for the studies. The calibration curve Abs = f(C) in Figure-5 exhibiting fairly good linearity with a trend equation: $y = 0.0098\ x + 0.018$ and a correlation coefficient $R^2 = 9992$.

By setting y = Abs and x = C_i we obtain the relation Abs = 0.0098. C_i + 0.018

Let: $C_i = \frac{(Abs - 0{,}018)}{0{,}0098}$ (3)

Abs = absorbance. This relationship is subsequently used to determine the residual RC concentration from the measured absorbance.

The effect of geopolymer mass: The variations in yield as a function of the mass of adsorbent are given by the curve in Figure-6. The yield increases with the mass between 0 and 80 mg of geopolymer, beyond 80mg, an equilibrium is observed in which the reaction no longer evolves regardless of the mass of adsorbent. From this result, we can retain that for 50mL of dye solution at 50mg/L, the optimal mass of adsorbent is $m_{optimal}$ = 80mg with a yield of 66%.

pH_{pzc} determination: The point of zero charge (pzc) defines the conditions of the solution (in particular the pH value) for which the surface density of the positive charges (cations) is equal to that of the negative charges (anions). The pH_{PZC} makes it possible to anticipate the nature of the surface electrostatic forces of the depollutant.

To determine the pH_{pzc} we used the method described by Crini and Badot[23]. It consists of placing 50cm^3 of 0.01M NaCl solution in beakers and adjusting the pH of each to a value between 2 and 12 by adding 0.1M NaOH or HCl solution. In each beaker, the optimum mass of geopolymer is added to it.

The suspensions must be kept under constant stirring, at room temperature, for 48h, in order to determine the final pH. The curve pH_f - pH_i = f (pH_i) is then plotted. The point of intersection between the curve and the straight line of equation y = 0 indicates the pH at the zero point of charge (pH_{pzc}). The optimum mass being set at 80mg and the initial pH values being adjusted to the nearest hundredth for volumes of 50mL of the 0.01 M sodium chloride solution, the curve pHf – pH_i = $_f$ (pH_i) is given in Figure-7. From this curve, it can be seen that the final pH is very close to the initial pH in the interval[6,8]. So, we can say that the pH at the zero point of charge is around 7.

The effect of pH: The determination of the residual concentrations and the calculation of the yields led to the curve R%= f (pH) given in Figure-8. This curve shows that the yield is higher when the medium is more acidic; it tends towards 78% when the pH is equal to 2. It decreases as the medium becomes more basic. From this result, we can say that the optimum pH of this adsorption reaction is less than or equal to 4. Adsorption would therefore be possible at ordinary temperature, but with a better yield in very acidic media.

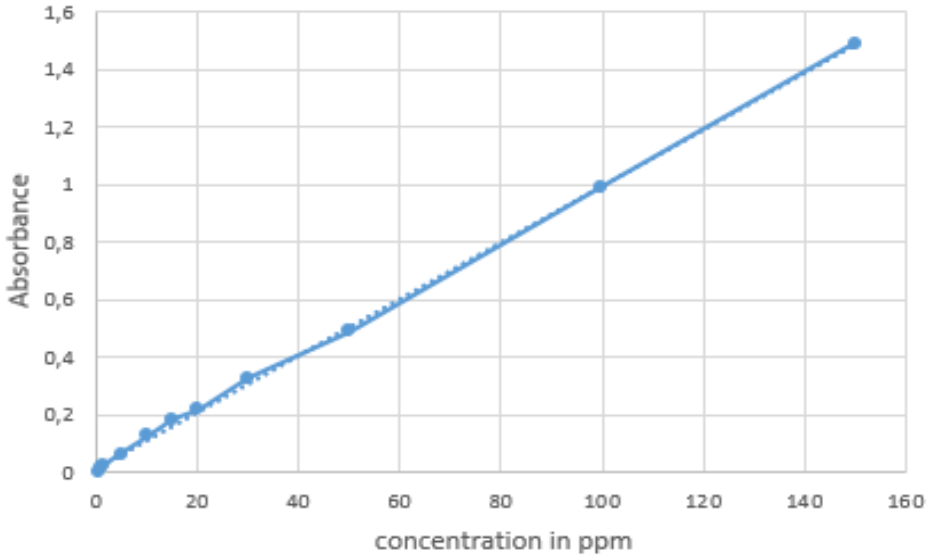


Figure-5: RC Calibration Curve (λ =500nm): y =0,0098x + 0,018; R^2 = 0,9992.

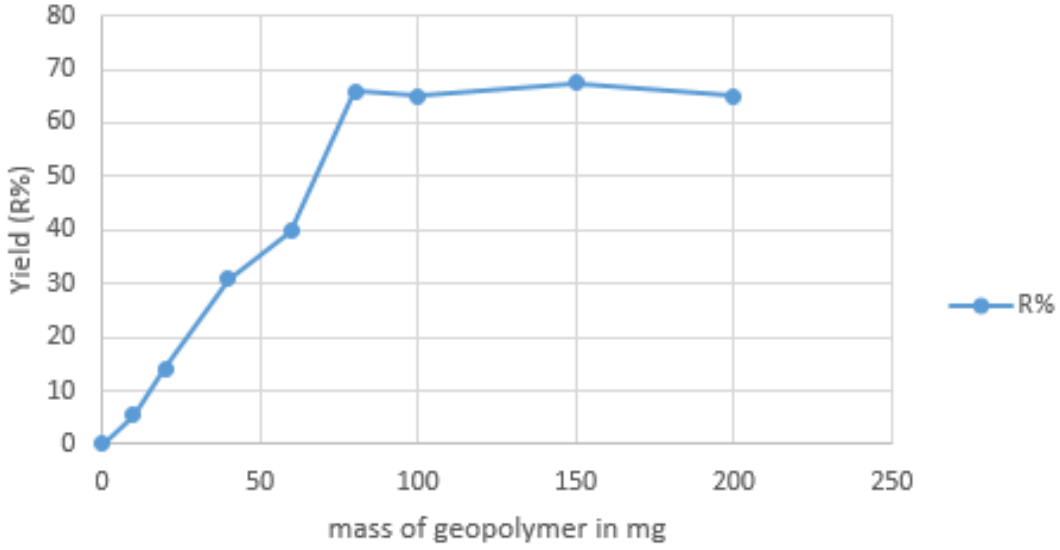


Figure-6: Effect of geopolymer mass on Congo red adsorption.

Adsorption kinetics: The kinetics of the reaction were studied at room temperature (approximately 28°C) and under the optimal conditions previously determined, namely: pH = 4 and mass of adsorbent m = 80mg for each volume of 50mL of dye at a concentration given (10; 20; 50 or 100mg/L). Figure-9 gives the evolution of the yield as a function of time for the four different concentrations. It can be noted that the reaction, although not instantaneous, is rapid from the first minutes of contact (between 0 and 40 minutes) then an equilibrium is observed beyond 60 minutes for all the four concentrations. However, the steady state is quickly reached (around 45 min) for high concentrations (50 and 100mg/L). For concentrations of 10 and 20mg/L, equilibrium is reached after 90 and 60 minutes respectively. It can therefore be said that the rate of the reaction increases with the concentration. Moreover, solutions of medium concentration (20 and 50mg/L) give a higher yield (78.57% and 75.10% respectively). Furthermore, there is a regression in yield beyond 120 minutes of contact; this may explain the drop in yield when the contact time becomes too long.

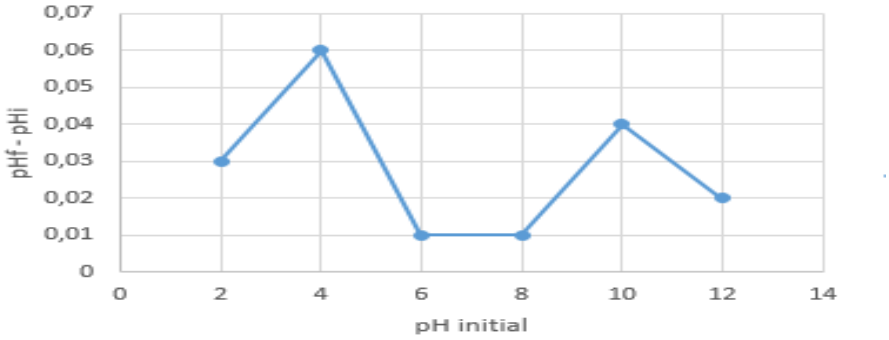


Figure-7: Curve $pH_f - pH_i = f(pH_i)$ for the zero point of charge.

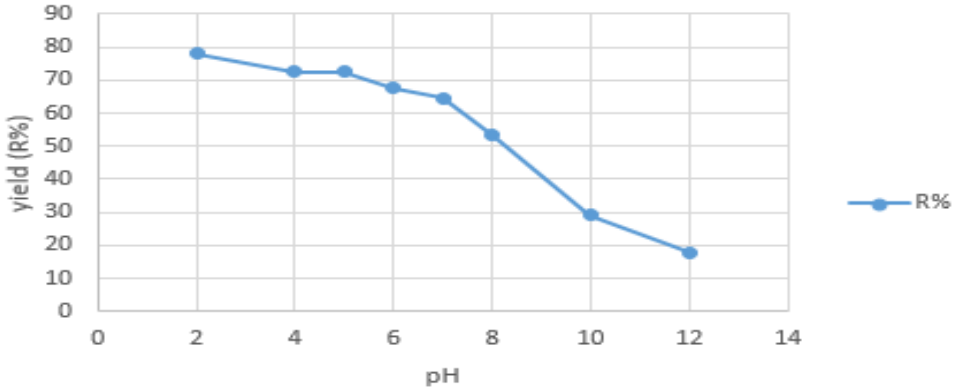


Figure-8: Effect of pH on Congo Red Adsorption by Geopolymer.

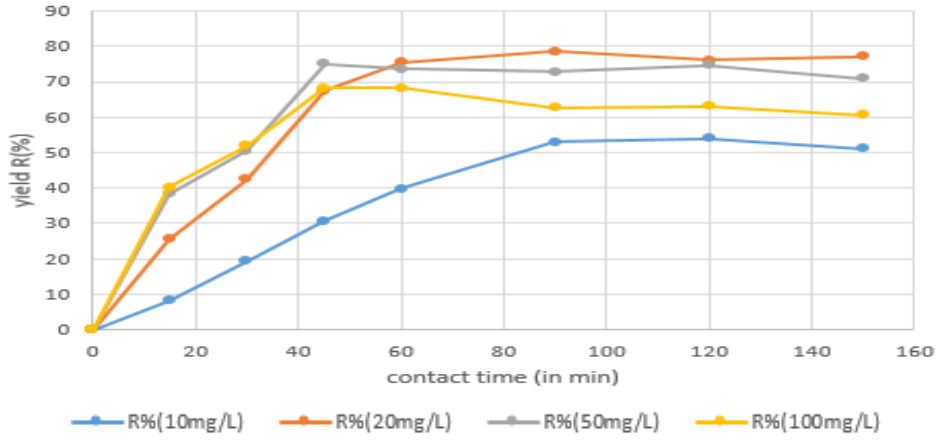


Figure-9: Adsorption yield as a function of time and congo red concentration.

From these analyses, we can retain that the process of adsorption of the congo red by the geopolymer is not an instantaneous reaction nor a very slow one. The optimal reaction time can be between 45 and 90 minutes depending on whether the concentration varies from 10 to 100mg/L.

The order of adsorption kinetics: The order of the reaction is a parameter revealing the influence of the contact time on the retention of an adsorbate; it provides more precision on the reaction mechanisms[24]. The most common reaction orders are:

The pseudo-first order: in this case, the rate of reaction is proportional to (q_e - q_t) according to the Lagergren equation.

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \quad (4)^{24},$$

With: k_1: the pseudo-first order rate constant (in min^{-1}), q_t: the adsorption capacity at date t ($mg.g^{-1}$), q_e: the adsorption capacity at equilibrium ($mg.g^{-1}$).

After integration of t = 0 to t, and of q_t = 0 to q_e, we obtain the linear form of this equation which is:

$$\text{Log}(q_e - q_t) = \log(q_e) - k_1 t \quad (5)^{24}.$$

Pseudo-second order: Here the reaction rate depends on the amount of adsorbate retained on the surface of the adsorbent and the amount adsorbed at equilibrium[25]. The pseudo-second order equation is:

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \quad (6)^{25}$$

With: k_2: pseudo-second order rate constant ($g.mg^{-1}.min^{-1}$), q_t: the adsorption capacity at date t ($mg.g^{-1}$), q_e: the adsorption capacity at equilibrium ($mg.g^{-1}$),

By integration we obtain the following linearized form

$$\frac{1}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (7)^{25}$$

The results of the adsorption kinetics made it possible to calculate the adsorption capacities as a function of the contact time for each concentration. The application of the pseudo-first-order and pseudo-second-order kinetic models by the ORIGIN-PRO Software enabled us to obtain the values of the maximum capacities (Q_e), of the regression coefficients (r_1 and r_2) and of the pseudo-first order and second order constants (k_1 and k_2) that are noted in Table-2.

In view of these results, it appears that for both orders, the retention capacity increases with the concentration. Moreover, the values of the regression coefficients are higher for the pseudo-second order model. From these observations, we can conclude that the adsorption mechanism would indeed be of the pseudo-second order model. Therefore, the reaction rate depends on the amount of adsorbate retained on the surface of the adsorbent and the amount adsorbed at equilibrium.

Adsorption isotherms: The adsorption isotherm is a very important parameter for the characterization of the textural structure of the adsorbent. In the expression of isotherms, the most frequent models are the Langmuir model and that of Freundlich[26]**.**

The Langmuir isotherm: Langmuir's theory is based on the following assumptions: i. The adsorption is localized, that is to say that the dye molecules are adsorbed on well determined sites. ii. The adsorption is monolayer, i.e. each site can retain only one molecule of dye. iii. There are no interactions between the adsorbed molecules.

Its equation is: $q_e = \frac{q_m . k_L . C_e}{1 + k_L . C_e}$ (8)[26]

With C_e: the equilibrium concentration of the solute in solution ($mg.L^{-1}$); q_e: the adsorption capacity at equilibrium ($mg.g^{-1}$); q_m: the maximum adsorption capacity ($mg.g^{-1}$); k_L: Langmuir's constant ($L.mg^{-1}$).

Table-2: Order constants of the adsorption reaction of Congo red by the geopolymer.

	Pseudo-first order			Pseudo-second order		
Concentration ($mg.L^{-1}$)	Qe ($mg.g^{-1}$)	k1 (min^{-1})	r_1^2	Qe ($mg.g^{-1}$)	k_2 ($L.mg^{-1}.min^{-1}$)	r_2^2
10	3.88 ± 0.42	0.01560 ± 0.00362	0.9648	5.52 ± 0.01	0.00222 ± 0.00001	0.9990
20	10.14 ± 0.50	0.03128 ± 0.00100	0.9700	12.54 ± 0.02	0.00263 ± 0.00002	0.9927
50	23.38 ± 0.88	0.04862 ± 0.00757	0.9701	26.96 ± 0.05	0.00232 ± 0.00003	0.9824
100	40.05 ± 1.04	0.06555 ± 0.00851	0.9818	44.60 ± 0.09	0.00213 ± 0.00003	0.9732

The Freundlich isotherm: The Freundlich model is based on an empirical equation representing the variation of energies with the quantity of adsorbate retained. It applies in many cases, in particular the case of multilayer adsorption with the possibility of interactions between the adsorbed molecules.

Its equation is: $q_e = k_f . C_e^{1/n}$ (9)[26]

With q_e: the adsorption capacity at equilibrium ($mg.g^{-1}$); n and k_f: the Freundlich constants. C_e: equilibrium concentration of the solute in solution ($mg.L^{-1}$); Adsorption is good if $0.1 < n < 0.5$ and weak if $n > 1$.

The various constants k_L, k_f and the regression coefficients r^2 as well as the maximum adsorption capacity q_m are determined by plotting these equations using the ORIGIN-PRO software.

Figure-10 shows the adsorption isotherms according to the Langmuir and Freundlich models. The values of the Langmuir and Freundlich parameters are listed in Table-3. A comparison of the regression coefficients of the Langmuir model (r_1^2= 0.9593) with those of the Freundlich model (r_2^2 =0.9983) reveals that the process of adsorption of congo red on the geopolymer can be described by either of the two isotherms. However, a Freundlich isotherm is better indicated. In this case, it can be said, based on the Freundlich isotherm that the dye molecules adsorb on several layers (multilayer adsorption) with the possibility of interaction between the adsorbed molecules.

It should be noted that the Freundlich constant (n) is greater than 1 and the maximum yield obtained under optimal conditions is around 80%; which means that the material fails to remove almost all of the dye regardless of its concentration. This state of affairs can be explained by the nature of the bonds between the dye molecules and the solid (adsorbent). Indeed, the dye (congo red) being anionic, therefore has more affinity to the cationic sites; whereas the cationic sites of the geopolymer are OH groups, therefore, the retention of the dye is therefore done by electrostatic interactions (of the hydrogen bond type) which are weak bonds. It therefore follows an easy desorption of the anionic dye. This reversible character of the reaction and the type of chemical bond confirms, on the one hand, the Freundlich isotherm which supposes a multilayer and reversible fixing of the adsorbate on the adsorbent and justifies, on the other hand, the reduction of the yield observed when the contact time is too long. This result is comparable to that of Fardjaoui in his research on the application of LTA zeolite to the adsorption of anionic textile dyes (bezanyl yellow, nylomine green). He finds that the Freundlich model better explains this adsorption process[27]. However, the pseudo-second-order kinetic model that characterizes this adsorption process generally assumes a chemisorption mechanism[19]. Given the proximity of the values of the regression coefficients of the Langmuir and Freundlich isotherms, it would be imprudent to make a choice with certainty. Then, the determination of the thermodynamic parameters and of the diffusion process could make it possible to elucidate this state of affairs.

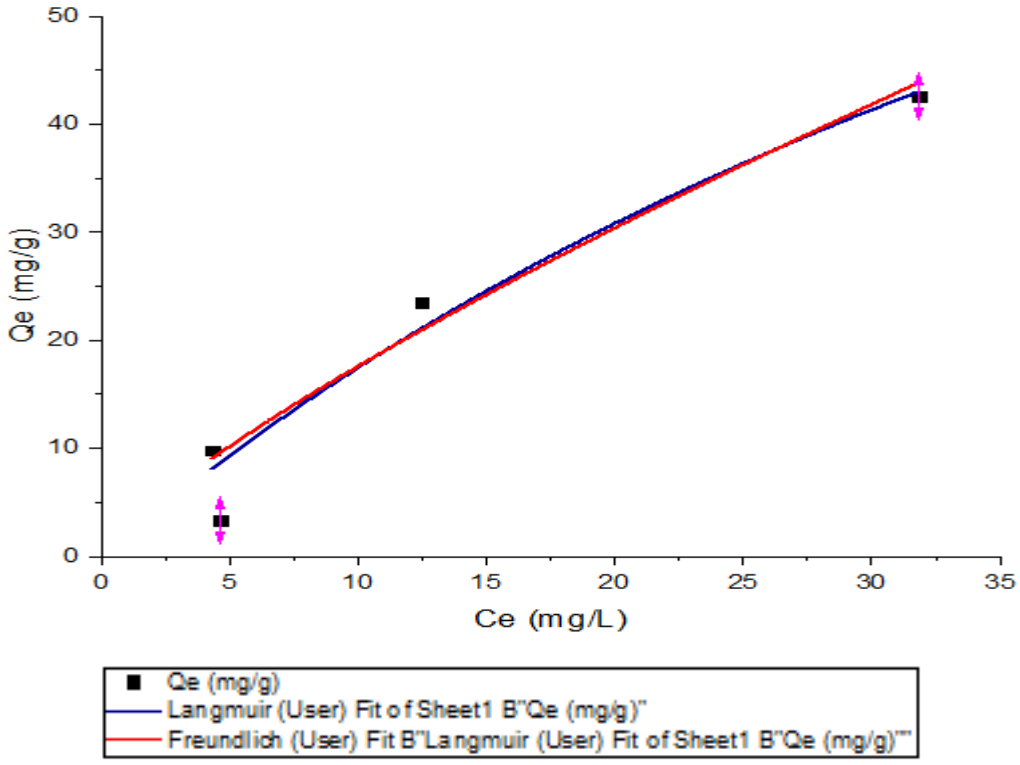


Figure-10: Isotherms of the adsorption of congo red by the geopolymer according to the Langmuir and Freundlich models.

Table-3: Langmuir and Freundlich constants of the adsorption reaction of congo red by the geopolymer.

Langmuir Isotherm			Freundlich isotherm		
Q_m (mg.g^{-1})	K_L (L.mg^{-1})	r^2	K_F (mg$^{(1-n)}$.Ln.g^{-1})	n	r^2
129.3083±0.94389	0.01566 ± 0.01592	0.9593	2.88591±0.01109	1.27199 ± 0.00203	0.9983

Conclusion

Our work was carried out in the interest of finding a method of treating colored water by an economically affordable and quantitatively available material in our environment. The geopolymer material made from Benin clay has the ability to absorb an anionic dye (Congo red). The adsorption tests and the parameters involved have shown that optimization of the adsorption phenomenon is possible in a frankly acid medium (pH ≤ 4) and with a contact time of approximately one hour at room temperature. However, the Freundlich elimination capacity (n=1.27199) being very high (n>1), research must continue on the improvement and adaptation of the adsorbent properties of the material to the adsorption of anionic dyes in general on the one hand and the optimization of thermodynamic parameters on the other hand. In perspective, an implementation of the treatment chain must be studied and applied to effluents from textile industries and hospital effluents from the city of Cotonou and its surroundings. This study must include the management of the sludge generated.

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