Removal of Anionic Dye from Textile Industries' Effluents by using Tunisian clay as adsorbent. Electrophoretic and Streaming potential investigations

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Abstract

The treatment of textile effluents is still a great challenge and constant research is done to find sustainable efficient processes. Natural clays are promising adsorbents for removal of pollutants, particularly dyes from effluents. But there is a need of better understanding the mechanisms of adsorption, especially in the case of anionic dyes. In this study, we investigated the adsorption capacities for RR120 anionic dye, of raw clays such as Tabarka (kaolinite and illite), Fouchana (smectite, kaolinite and illite), a standard kaolinite (KGa-2), and a standard palygorskite (PFl-1). Batch experiments were performed by varying different parameters. Electrophoretic mobility and streaming induced potentials (SIP) measures were done to investigate the clays surface charges and the dye-clay interactions during adsorption. The results showed that although palygorskite exhibits the highest porosity and specific surface, its capacity to adsorb RR120 is the lowest compared to the other clays, whatever the pH and ionic strength conditions, whereas KGa-2 shows the highest adsorption capacity. The adsorption process was exothermic, spontaneous and feasible, and favoured at ambient temperature, although to a lesser extend for PFl-1. Electrokinetic measurements confirm that adsorption occurs onto clay surfaces and are in accordance with isotherm modelling pointing out that the adsorption is limited. The kaolinite shows the best adsorption potential for RR120 anionic compared to smectite and fibrous clay and may be a sustainable alternative for the treatment of dyeing wastewaters.

Keywords: Anionic dye; adsorption; clay; electrokinetic; streaming potential; electrophoresis

Introduction

The effluents from textile dyeing industries contain various chemicals, among them dyes which have not been retained during the dyeing process (Gürses et al. 2006; Allen and Koumanova 2005; Volmajer Valh et al. 2009**)**. The dyes are of various compositions, among them reactive dyes are widely used in the textile industry and are particularly difficult to degrade. Their estimated half life time in the environment is of several years (Zaharia and Zuteu 2012). The treatment of these effluents is a challenge even today, on the one hand because it is difficult to totally remove the color, and on the other hand because of the costs of treatment. Adsorption is a widely used method and many studies are being invested in the search for alternative, inexpensive, available and effective adsorbents. Another important point is the sustainability of the treatment. Keeping in mind these objectives, natural clays are a promising material because of their low cost, large availability in many countries, their potential reuse, and their widely recognized qualities already used in depollution (Khalfa et al. 2016; Alshameri et al. 2014). In fact, their structure provides them an active surface for the adsorption of organic and inorganic pollutants. In the natural state, they have great adsorption capacities for metals, cationic or neutral molecules. Previous studies have shown their potential to discolour effluents containing reactive, thus anionic dyes, which should be difficult owing to their negative permanent structural charge (Abidi et al. 2014, 2015). In this work, it is proposed to study and compare the decolorizing potential of clays of different species by focusing on an anionic dye used in the textile industry. The adsorption processes are studied from experiments in batch, and by electrokinetic methods.

Materials and methods

Materials

Two natural clays from the North-East of Tunisia and two standard clays were used as adsorbent. Two of them are kaolinite-rich clays, the commonly called Tabarka clay mainly composed of kaolinite (62%) and illite (38%), and the standard kaolinite (KGa-2) from the Source Clay Repository of the Clay Minerals Society. An other adsorbent clay, the commonly called Fouchana clay, is rich in smectite (60%) but contains also kaolinite (30%) and illite (10%). The last clay is standard fibrous structured clay: it is palygorskite (PFl-1) from the Source Clay Repository of the Clay Minerals Society. These clays are almost pure and contain minor amounts of accessory minerals (Abidi 2015). The Cation Exchange Capacity (CEC) values of Fouchana, PFl-1, Tabarka and KGa-2 are respectively 34.3, 19.5, 15 and 3.3 meq 100 g^{-1}, and their specific surface values are respectively 80, 136.4, 47 and 23.5 m^2 g-1. Only KGa-2 exhibits an isoelectric point at pH 4.8, whereas the other clays keep their negative surface charge in the 3-10 pH range. The anionic dye Reactive Red 120 (RR 120) commonly used in the textile industry is chosen as adsorbate in this study. It is a bifunctional azo-based chromogens dye incorporating bis (monochloro-s-triazine). Its structure is given elsewhere (Abidi et al. 2015) and the molecular weight is M =1338 g mol^{-1}. The absorption maximum wavelength of RR 120 in the visible range is λ=535 nm, which was used to analyse the supernatant of the anionic dye aqueous dispersions.

Methods

Adsorption experiments

The adsorption of Reactive Red 120 (RR 120) onto Fouchana, PFl-1, Tabarka and KGa-2 clays was investigated in aqueous solution in a batch system. Various parameters were varied such as the contact time adsorbent-adsorbate, the ionic strength, the pH of the aqueous phase, the initial dye concentration and the temperature. The experiments were performed at temperature ranging from 20 to 50°C with constant solution volume (V) of 100 mL and with the same mass of adsorbent (m = 3g). Samples were collected and centrifuged for 15 min at 3600 rpm. The remaining amount of the dye in the supernatant solution was analysed using a spectrograph (Jobin Yvon, TRIAX 320), and by monitoring the absorbance changes of the dye aqueous solutions at λ_{max} = 535 nm. The amount of the adsorbed dye per unit mass of clay at time t, q_t (mg g^{-1}) was calculated according to (Eq 1):

$$q_e = (C_0 - C_e) \times \left(\frac{V}{m}\right) \qquad \textbf{Eq (1)}$$

where C_0 and Ce are the initial and the equilibrium liquid phase concentrations of dye solution (g L^{-1}), respectively, qe is equilibrium dye concentration on adsorbent (mg g^{-1}), V is the volume of dye solution (L), and m (g) the mass of clay sample.

Electrokinetic measurements

Electrokinetic effects are the result of interrelations between mechanical and electrical forces at the boundary between a solid and a solution of electrolyte (Jada and Chaou, 2002; Jada et al. 2002). According to Jacobasch et al. (1985), adsorption processes can be investigated directly with the help of electrokinetics and make it possible to recognize reactions on the surface during measurements (Ridaoui et al. 2006; Jada and Salou 2002; Jada and Verraes 2003). Moreover, pH changes have an effect on the zeta potential (ζ), which also gives information regarding the nature of the solid surface in contact with a polar medium (Siffert et al. 1994; Jada et al. 1999).

In the present work, we have used two ways to determine the electrokinetic potential: either by applying an externally electrical field causing movement of the solid particles in a stationary liquid phase (electrophoresis, EP) (Jada and Salou 2002), or by applying a mechanical force which creates an electric current (Jada et al. 2006). This current builds up in turn a potential difference between two electrodes situated at the ends of the capillary known as streaming induced potential (SIP) On one hand, The EP measurements allow the monitoring of the particles' surface charge as function of the aqueous phase pH, and thus determining the Isoelectrical point (IEP). On the other hand, the SIP measurements enable recording the particles' surface charge evolution as function of an added titrant.

Streaming induced potential measurements

The measuring cell of the apparatus (Müteck PCD03) is composed of a cylindrical polytetrafluoroethylene (PTFE) container with a PTFE piston inside. An amount of 10 mL of clay aqueous dispersion was filled into the gap (0.5 mm) between the container wall and the piston, and various amounts of titrants RR 120 dye aqueous solution were then added. The resulting streaming induced potential (SIP) was measured between two gold electrodes located at the top and the bottom of the gap (Jada et al. 2006). The evolution of the SIP value as function of the added titrant reflects the modification of clay particle surface charge during the titration, and thus informs on the adsorption or not, of the titrant onto the titrated species. The titration of the clay particles by the RR 120 dye molecules were done at natural pH, ambient temperature, with initial concentrations of 0.48 mg L^{-1} for the RR 120 dye solution and 100 mg L^{-1} for the clay suspension.

Electrophoretic mobility measurements

The measurements of the electrophoretic mobility (Ue) of the clay particles were performed in a clean electrophoresis chamber, filled with a dilute clay aqueous dispersion prepared either in the absence or in the presence of the adsorbed RR 120 dye molecules. During the electrophoretic measurements, the clay aqueous dispersions were submitted to an electrical field for few seconds. The applied tension in the electrophoretic cell was 100 V, and the pH of the clay aqueous dispersion was adjusted between 2.5 and 12.5 by adding aliquots of either HCl or NaOH solutions prior the mobility measurement. At least three experiments were achieved for each sample. It should be emphasised that no sedimentation was observed during the measurements, even at low pH values, due to the low particle sedimentation rate. The mobility Ue is calculated according to Eq (2):

$$\mathbf{Ue\ (cm^2/V\ s) = 10^{-4}\ v\ (\mu m/s)/E\ (V/cm)} \qquad \textbf{Eq (2)}$$

Where v (µm s^{-1}) is the clay particle velocity measured at ambient temperature by using the Zetaphoremeter II apparatus (Jada and Erlenmeyer 2012), and E (V cm^{-1}) is the electric field applied to the clay.

Results and discussion

Effect of contact time, ionic strength, initial dye concentration and pH

In all cases, at natural solution pH and at ambient temperature, there is an increase in the amount of adsorbed dye RR120 (qe: mg g^{-1}) when ionic strength increases from 0 to 1 M (Fig. 1) due to screening of the clay particles' surface charge by the presence of many nearby cations (Newcombe and Drikas 1997). There is also a rapid increase with contact time between 10 to 60 min due to the abundant availability of active sites on the clay surface. The plateau is reached after 60 min, and KGa-2 exhibits a greater amount of dye adsorbed for 1 M ionic strength (2.4 mg g^{-1}) compared to Tabarka (1 mg g^{-1}), Fouchana (0.9 mg g^{-1}) and PFl-1(0.6 mg g^{-1}).

When the initial concentration of RR 120 dye (Ci) increases from 10 to 120 mg L^{-1} there is a rapid increase in the amount of dye adsorbed for low Ci values, and a plateau is reached for higher concentrations of dye (Fig.2). For KGa-2 clay the Ci value at which a plateau is reached is higher than the other clays, with the minimum value for PFl-1. This indicates that KGa-2 offers more active sites available for the adsorption and that saturation occurs with higher Ci values. On the contrary, in the case of PFl-1 saturation occurs at lower Ci values indicating less active sites or a less efficient process because of less accessibility of the sites (Murat 2014).

When the pH of the solution decreases from 9 to 3, there is a significant increase in the amount of RR 120 dye adsorbed onto clays. This is particularly true from PFl-1 showing qe values for pH =3 (0.26 mg g^{-1}) twice as much as for pH =7 (0.78 mg g^{-1}). KGa-2 exhibits an isoelectric point at pH = 4.8, which means that the clay's surface charges become more positive at lower pHs; that explains a higher adsorption capacity of the clay for anionic dye. Although Fouchana, Tabarka and PFl-1 clays do not exhibit an isoelectric point at the studied pH range the increase in the amount of dye adsorbed at low pH may be explained in the same way as for KGa-2. Indeed, all these clays exhibit amphoteric surfaces that become more positively charged at low pH, which leads to favourable attraction of the anionic dye molecules, and thus enhanced adsorption. It should be also noted that at low pHs, the dye molecule is subject to de-protonation of one of the diaminochlorotriazine-groups (pKa = 4.4), which could also lead to attraction between molecules (Errais et al. 2012), and enhances adsorption onto clay.

When comparing the different adsorption capacities of the clays, it appears that KGa-2 kaolinite is the most efficient, followed by the Tabarka mixture of kaolinite and illite, then by the Fouchana smectite-rich clay, and finally by the fibrous clay PFl-1. This tends to indicate that the kaolinite mineralogical species plays a major role in the adsorption of anionic dye, whereas illite (in Tabarka) and smectite (in Fouchana) are more limited and do not enhance the adsorption capacity of the clay material when mixed with kaolinite. Palygorskite, which is widely used as adsorbent of pollutant due to its high structural porosity and high specific surface, shows the lowest efficiency. This means that high porosity and specific surface do not enhance the adsorption of RR120 dye. Thus, the explanation would be in the nature of the surfaces and the distribution of particle surfaces' charges that are different between kaolinite and the other clays.

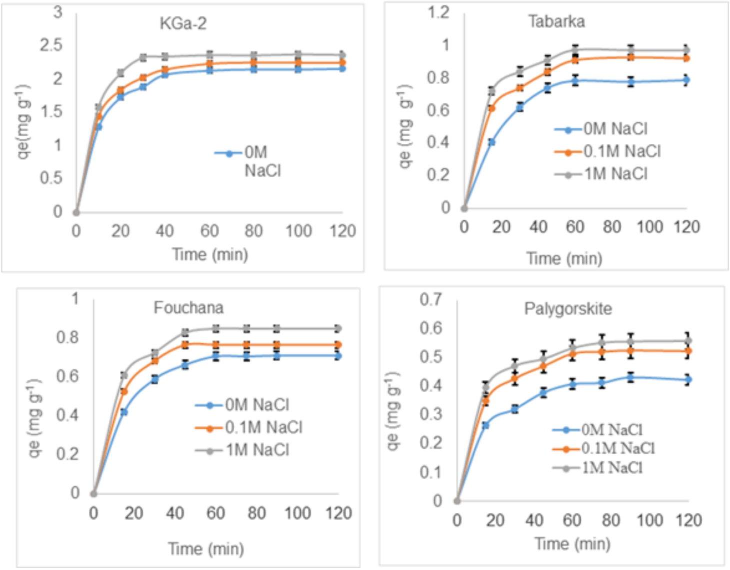

Fig. 1 Effect of ionic strength and contact time on the adsorption of RR 120 dye onto clays

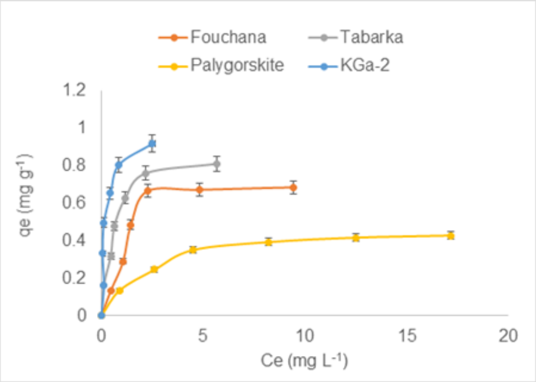

Fig. 2 Effect of initial dye concentration on the adsorption onto clays

Adsorption isotherms

Two isotherm models were considered to model the data, and to investigate the adsorption mechanism: the two parameters Langmuir and Freundlich isotherms models. They were chosen because they are the most widely applied sorption isotherms, and because they describe satisfactorily the experimental isothermal data (Rahman et al. 2013; Chaari et al. 2015). The Langmuir adsorption isotherm (Langmuir 1916) assumes that sorption takes place at specific homogeneous sites within the adsorbent. The linear form of Langmuir isotherm is given in Table 1, where C_e is the equilibrium concentration (mg L^{-1}), qe the amount adsorbed at equilibrium (mg g^{-1}), qm the adsorption capacity (mg g^{-1}) and b is the energy of adsorption (Langmuir constant (L mg^{-1})). The Freundlich adsorption isotherm is based on the adsorption with non-uniform distribution of adsorption heat onto heterogeneous surfaces (Freundlich 1906; Adamson and Gast 1997) as well as multilayer sorption as given by equation in Table 1, where K_F and n are Freundlich constants and were calculated from the slope and intercept of the Freundlich plots (log(qe) versus log(Ce)). The parameters calculated from these models, the regression coefficients (R^2), and sum of squared errors of prediction (SSE) are listed in Table 1.

Table 1 Isotherm equations, constants, correlation coefficients (R^2), and sum square errors (SSE) for RR 120 adsorption onto Tabarka, Fouchana, KGa-2 and Palygorskite clays

Isotherm model	Clays	Model parameters				
		qe experimental (mg g^{-1})	qm (mg g^{-1})	K_L (L mg^{-1})	R^2	SSE
Langmuir $\frac{C_e}{q_e}=\frac{1}{q_m b}+\frac{C_e}{q_m}$	Tabarka	1.2	1.0	0.53	0.98	0.041
	KGa-2	2.4	2.1	1.004	0.97	0.26
	Fouchana	0.8	0.6	0.049	0.99	0.12
	Palygorskite	0.4	0.45	0.04	0.99	0.22
Freundlich $\log(q_e)=\log(q_F)+\frac{1}{n}\log(C_e)$			K_F (mg g^{-1})	n	R^2	SSE
	Tabarka	1.2	0.5	1.17	0.84	0.24
	KGa-2	2.4	0.7	3.16	0.91	0.77
	Fouchana	0.8	1.03	0.6	0.95	0.09
	Palygorskite	0.4	1.53	1.34	0.97	0.13

We considered first the highest R^2 value, and second the smallest SSE value, to choose the best model to describe the experimental isotherm. Considering these criteria and the good correspondence of calculated and experimental q_e, the results indicate that the Langmuir model is the best to describe the adsorption of RR 120 onto all clays, indicating monolayer coverage of the surfaces.

Thermodynamic parameters of adsorption

The changes in the free energy of sorption reaction (ΔG°) were calculated for different temperatures (Table 2). ΔG° is given by Eq (3). ΔG^0 is the standard free energy change (J mol^{-1}), R is the universal gas constant 8.314 (J $mol^{-1}K^{-1}$), T is the absolute temperature (K), and K the equilibrium constant given by Eq (4). ΔH^0 and ΔS^0 are the enthalpy and entropy of sorption reaction respectively.

$$\Delta G^\circ = -RT \ln K_0 \quad \textbf{Eq (3)} \qquad \ln K_0 = (\Delta S^0/R) - (\Delta H^0/RT) \quad \textbf{Eq (4)}$$

Table 2 Standard free energy (ΔG^0), at different temperatures, enthalpy (ΔH^0) and entropy (ΔS^0) for adsorption of RR 120 dye onto the Tabarka, KGa-2, Fouchana and Palygorskite clays

T (K)		293.15	303.15	313.15	323.15
ΔG^0 (kJ mol^{-1})	KGa-2	-4.738	-4.359	-4.81	-3.514
	Tabarka	-4.283	-4.027	-3.898	-3.219
	Fouchana	-2.457	-7.52	-2.72	-0.146
	Palygorskite	0.645	0.969	1.251	1.507
ΔH^0 (kJ mol^{-1})	KGa-2	-16.3			
	Tabarka	-13.9			
	Fouchana	-10			
	Palygorskite	-7.8			
ΔS^0 (J mol^{-1})	KGa-2	-39.3			
	Tabarka	-32.7			
	Fouchana	-30.7			
	Palygorskite	-28.8			

The results show in all cases a decrease in the adsorption capacity of RR 120 dye when the temperature increases from 293 K to 323 K, indicating that ambient temperature is more favourable to adsorption of RR120 dye on the studied clays. The negative values of ΔG° in case of KGa-2, Tabarka and Fouchana in the studied temperature range indicate that the adsorption is still feasible and spontaneous at higher temperature (Salam et al. 2010). But in the case of PFl-1 the ΔG° values are always positive which indicates that the adsorption process is not as feasible and spontaneous as for the other clays (Table 2). The ΔH0 and ΔS0 values are all negative, which indicates that the process is exothermic and that it induces more order at the liquid-solid interface. It is to note that ΔS0 value for KGa-2 is the most negative, thus the dye molecules adsorbed onto kaolinite are more organized at the solid-liquid interface than in the case of palygorskite.

Electrophoretic mobility

Fig. 3 shows the variation of the electrophoretic mobility, Ue, of the various clay particles as function of the aqueous phase pH, in the presence of RR120 dye molecules. In all instances, the increase of the aqueous phase pH leads to an increase in the magnitude of Ue, which, at pH≥9 reaches its maximum value, -2.23 10^{-4}; -1.93 10^{-4}; -1.73 10^{-4}; -1.55 10^{-4} cm^2 sec^{-1} $Volt^{-1}$, respectively for, Tabarka; KGa2; Palygorskite; and Fouchana clays. In the presence of RR 120 dye molecules adsorbed on various clay surfaces, the increase in the magnitude of Ue, with the pH, is due to the contribution of two negatively charged groups: one clay surface group (mainly silanol groups), the number of which increases with the pH, and a negatively charged sulfonate group belonging to the dye molecule, the number of which increases mainly with the adsorbed amount of the dye on the clay surface.

Note that the RR120 dye molecule contains mainly aromatic, amine and sulfonate groups. Thus, the adsorption of the dye molecules on the clay surface can involve electrostatic interactions that occur between negative and positive species, as well as non-covalent π interactions which occur between electron-rich π system and a metallic (cationic or neutral), or anionic species. All these interactions involved in the dye adsorption from water onto the clay solid surface can be tuned by varying various parameters such as the aqueous phase pH, the ionic strength, the temperature, the initial dye concentration, the nature of the electrolyte, etc. In addition, an important reduction in the Ue values of the bare clay occurs mainly at pH values ≤ 9 (Fig. not shown). The comparison of the data before and after adsorption of RR120 dye leads to the hypothesis that it is likely that the RR120 dye molecules adsorb on the clay surface, either via their ammonium groups which are deprotonated at pH values above pH=9, or via their sulfonate (SO_3^-) groups which interact with the clay surface positive sites, reducing hence the surface charge of this latter.

Streaming induced potential

Fig. 4 shows the variation of the normalized values of the Streaming Induced Potential (SIP) of various clays upon the addition of increasing amounts of the RR 120 dye molecules. Recall that the SIP measured during the piston movement, results from the separation of the counter ions from the RR 120 dye molecules covered clay particles adsorbed on the cylindrical container wall of the Müteck PCD03 measuring cell. In addition, the evolution of the SIP value as function of the added RR 120 dye, reflects the modification of clay particle surface charge during the titration, and thus informs on the adsorption or not, of the dye molecules onto the clay surface. Initially, i.e. before any addition of the dye molecules to the aqueous clay dispersions, the measured SIP values were negative, as resulting from the negatively charged surfaces for various clays. This excess negative charge is due to isomorphic substitutions in the crystal lattice, and protonation/ deprotonation of the aluminols and silanols at the amphoteric clay surfaces. However, upon the addition of increasing amounts of the dye solution to the clay aqueous dispersion, the SIP magnitudes increased, i.e. become more negative (Fig. 4) in the case of Fouchana, KGa-2 and Tabarka, and then levelled out, whereas a slight decrease in the SIP magnitude is observed in the case of palygorskite clay. The negative charge increase as observed in Fig. 4, for Fouchana, KGa-2 and Tabarka clays, indicates that the anionic dye added negative charges, by its adsorption on positive charges of the amphoteric surfaces of the clays. These positive charges are gradually neutralized by electrostatic interactions occurring between the positive charges of the minerals, and the dye's sulfonate groups (SO_3^-), enhancing hence the net negative surface charge of the clay particles. At the plateau values, as shown in Fig. 4, the SIP becomes constant because of the complete neutralization of the positive sites of the clay minerals and/or to the formation of barrier by the dye molecules, limiting hence the adsorption process. The SIP magnitudes, for Fouchana, KGa-2 and Tabarka samples, are higher than that observed for the palygorskite clay in the whole range of the added RR dye solution, reflecting hence higher adsorption amounts, for the former clays in accordance with the adsorption experiments. These differences in behaviours of various clays are probably related to the nature of their surfaces and compositions.

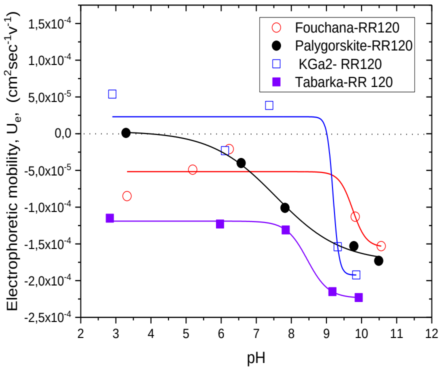

Fig. 3 Variation of the electrophoretic mobility, Ue, with the aqueous phase pH, for RR 120 dye molecules covered various clay aqueous dispersions

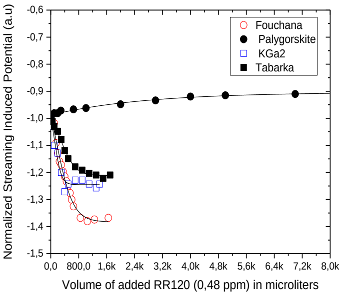

Fig. 4 Variation of the Streaming Induced Potential (SIP) with the volume of the added RR 120 dye molecules to the various clay particles in aqueous dispersions

Conclusion

The behavior of four natural clays was investigated in the adsorption of the RR120 anionic dye. The studied clays are a pure kaolinite (KGa-2), a kaolinite dominant clay mixed with illite (Tabarka), a smectite dominant clay mixed with kaolinite and illite (Fouchana) and a pure fibrous clay (palygorskite PFl-1).Their difference lies in their mineralogical composition and nature, but they all exhibit a negative permanent charge in their natural state, and amphoteric crystal edges. In addition, palygorskite exhibits a highly porous structure contrary to the other clays, and thus a higher specific surface.

The results show that although palygorskite provides a large surface to the adsorption, it has the lowest capacity for adsorption of RR120 dye, contrary to the pure kaolinite KGa-2 which exhibits the lowest specific surface and shows the highest adsorption capacity for anionic dye. Tabarka shows also a better adsorption capacity than Fouchana and palygorskite. Electrokinetic measurements indicate that adsorption occurs on the clay surfaces, which process should be difficult owing to the negative surface charge of the clays and the anionic character of the dye. The process is spontaneous, feasible and exothermic according to ΔG°and ΔH°values, although not favoured at high temperature. In case of palygorskite the positive values of ΔG° confirm that the adsorption process is difficult, which indicates again that porosity and specific surface are not relevant parameters in the adsorption of anionic dye. Moreover, isotherm modeling indicates that the coverage of the dye molecules on the clay surfaces is limited. All the results showed that the kaolinite rich clay is a better effective adsorbent for removal of the anionic dye in aqueous solution at ambient temperature, a low pH and a high ionic strength, and that the process without treatment of the clay may be a sustainable alternative for the treatment of dyeing waste waters.

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