Sorption of the Direct Black 22 dye in alluvial soil

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ABSTRACT

The semiarid region of Pernambuco has a large water deficit, leading the population to explore groundwater resources such as alluvial aquifers. The state of Pernambuco also stands out for having the second-largest textile manufacturing center in Brazil. However, the direct discharge of textile effluents from the region's industries has intensified the negative impacts on water conservation and alluvial soils. This work characterized the sorption of Direct Black 22 dye (DB22) in two layers of alluvial soil in the Capibaribe-PE basin. Batch experiments (kinetics and sorption isotherms) allowed the evaluation of the retention potential and mobility of this compound in this environment. Sorption kinetics were verified for stirring times of 0.25; 0.5; 1; 2; 4; 6; 8; 10; 12; 24; 48 and 72 hours and the experimental data was adjusted to the first- and second-order kinetic models. The isotherm occurred with concentrations of 1; 5; 10; 15; 20; 32.5 and 40 mg.L^{-1} and experimental data was adjusted to the linear, Freundlich and Langmuir models. The sorption kinetics of DB22 was best described by the second-order model, while the Freundlich and Linear models properly fitted sorption isotherms for Layers 1 and 2. The organic matter contents and the cation-exchange capacity of the soil layers influenced the sorption of the dye. The superficial layer privileges dye retention phenomena, while in the subsurface layer dye mobility phenomena prevail.

Keywords: contamination, emerging pollutants, groundwater, isotherms, semiarid, wastewater.

Sorção do corante têxtil Direct Black 22 em solo aluvionar

RESUMO

O semiárido pernambucano é uma região que apresenta grande déficit hídrico, levando a população a explorar os recursos hídricos subterrâneos como os aquíferos aluvionares. O estado de Pernambuco também se destaca por conter o segundo maior polo de confecções têxteis do
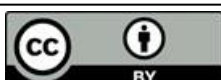
Brasil. No entanto, o lançamento direto de efluentes têxteis provenientes das indústrias da região tem intensificado os impactos negativos na conservação da água e dos solos aluvionares. O objetivo deste trabalho consiste na caracterização da sorção do corante Direct Black 22 (DB22) em solos de duas camadas de um aluvião na bacia do Capibaribe-PE. Os ensaios de Bacth (cinética e isotermas de sorção) permitiram avaliar o potencial de retenção e mobilidade desse composto nesse ambiente. A cinética de sorção foi verificada com os tempos de agitação de 0,25; 0,5; 1; 2; 4; 6; 8; 10; 12; 24; 48 e 72 horas e os dados experimentais foram ajustados aos modelos cinéticos de primeira e segunda. A isoterma ocorreu com concentrações de 1; 5; 10; 15; 20; 32,5 e 40 mg.L^{-1}, e os dados experimentais foram ajustados ao modelo linear, de Freundlich e de Langmuir. A cinética de sorção do DB22 foi melhor descrita pelo modelo de segunda ordem e as isotermas de sorção para Camadas 1 e 2 ajustaram-se adequadamente aos modelos de Freundlich e Linear. Os teores de matéria orgânica e a capacidade de troca catiônica das camadas de solo foram propriedades que influenciaram na sorção do corante. A camada superficial privilegia fenômenos de retenção do corante enquanto que a camada subsuperficial privilegia fenômenos de mobilidade do corante.

1. INTRODUCTION

Water availability in quantity and quality for multiple uses is an inherent concern of the population living in semiarid regions in northeastern Brazil. However, despite this problem, part of this region stands out as the second-largest textile manufacturing center in Brazil. The Pernambuco municipalities of Caruaru, Toritama, and Santa Cruz do Capibaribe are the main cities of this textile hub (Oliveira and Braga, 2014).

Alluvial deposits are geological formations that commonly occupy the beds of temporary rivers in the northeastern semiarid region. These deposits act as natural water reservoirs, allowing water supply during critical scarcity periods (Brito *et al.*, 2018). Despite being an essential water supply source, these formations are the destination of indiscriminate wastewater disposal by the region's textile industries. This considerably raises the risk of environmental contamination, as the release of textile effluents promotes the entry of various pollutants into the environment (Ali *et al.*, 2009; Shehzadi *et al.*, 2014; Konicki *et al.* 2017). For example, Shalaby *et al.* (2016) point out that the wastewater from these industries is a complex mixture of dyes and heavy metal compounds, and Vikrant *et al.* (2018) highlighted that 15% of the dyes are lost in effluents during the dyeing process.

Among the materials used by the textile industries, dyes, which are emerging pollutants, offer a high possibility of environmental contamination (Montagner *et al.*, 2017), being potentially hazardous for both the environment and human health (Munagapati *et al.*, 2019; Nazir *et al.*, 2020), even at low concentrations. There is also a lack of legislation to regulate their maximum allowable concentration in effluents (Souza and Souza, 2016). Therefore, to enable the development of studies concerning the analysis of groundwater vulnerability and conservation of its quality, it is fundamental to assess retention capacity and mobility of dyes in soils of alluvial deposits (Qu *et al.*, 2008; Dawodu and Akpomie, 2016; Silva Júnior *et al.*, 2019; Alves *et al.*, 2020).

Furthermore, Direct Black 22 (DB22), which is an azo dye characterized by the presence of the double bond between two nitrogen atoms (-N = N-) (Graciani, 2014), stands out as one of the most-used dyes among the textile industries of Pernambuco's Agreste (Amorim *et al.*, 2013). Investigations regarding the behavior of azo dyes, such as DB22 in alluvial deposits of the Capibaribe watershed, are still minimal. Therefore, considering these facts, this research

evaluated DB22 sorption through sorption kinetics and isotherm experiments in two soil layers of an alluvial deposit of Pernambuco semiarid region.

2. MATERIALS AND METHODS

2.1. Study Area

The study area is located in the Poço da Lama Ranch in the city of Brejo da Madre de Deus, bordering the city of Santa Cruz do Capibaribe in Pernambuco's State, Brazil (Figure 1). Soil samples were taken from the dry bed of the Capibaribe River, which is formed by a deposit of alluvial sediments. The aquifer in this area is widely exploited as a source of groundwater resources. The region has an average rainfall of 600 mm/year and potential evapotranspiration of 1900 mm/year, with semiarid climatic conditions and native vegetation cover of Caatinga (Braga *et al.*, 2015). Geologically, the study area is located in the Borborema Province, with crystalline rocky features and hydrogeology of fissural domain (Beltrão *et al.*, 2005).
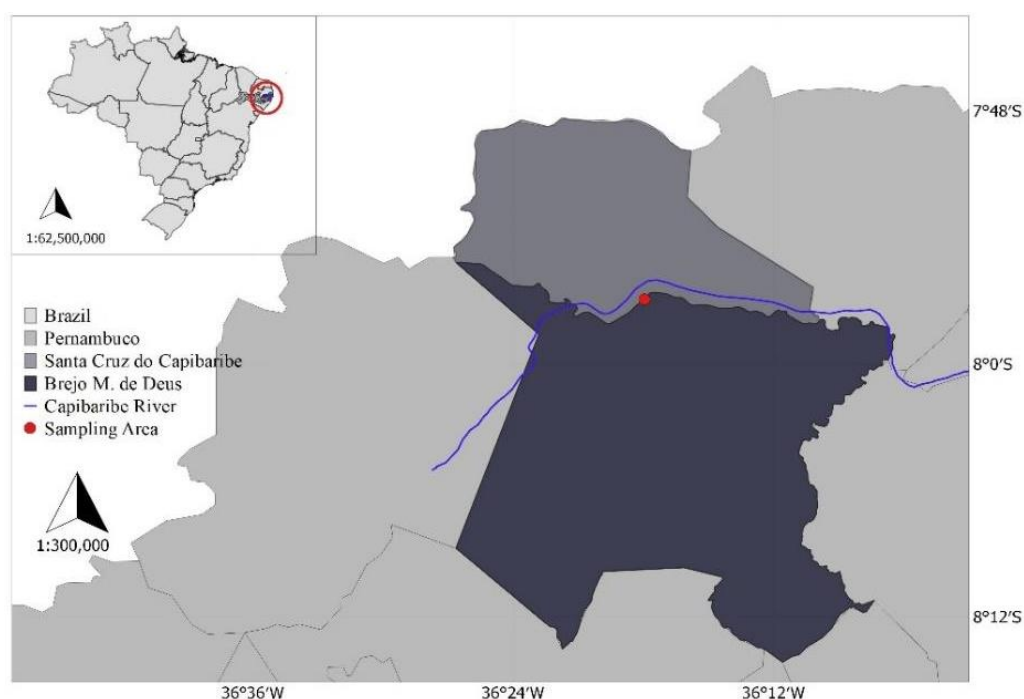

Figure 1. Location of the cities of Brejo da Madre de Deus and Santa Cruz do Capibaribe.

2.2. Physical and Chemical Properties of Soils

Batch experiments were performed with soil samples collected at two depths of the alluvial deposits. The first soil belongs to the most superficial layer (Layer 1), with a thickness of approximately 50 cm. The second soil belongs to the subsequent layer (Layer 2), which is about 60 cm thick. Some physicochemical properties of these soils were determined by Rabelo (2018), and are presented in Table 1.

According to Table 1, Layer 1 (superficial) has higher clay (7.03%) and organic matter (2.1 7%) contents than Layer 2, which presented a clay portion of 2.34%. These soils were classified as Loamy Sand and Sand, respectively. Vimonses *et al.* (2009) and Yagub *et al.* (2014) verified that information about the clay content in sorption studies is essential because this characteristic can influence the sorption of the DB22 dye. The pH values in H_2O were higher than the pH in KCl. These results are attributed to the effect of the KCl solution, which, in contact with the soil, induces cation exchange, with consequent increase of acidity (Plieski *et al.*, 2004; Ebeling *et al.*, 2008). Layer 1 (3.75 $m^2.g^{-1}$) has a specific surface greater than Layer

2 (2.56 m^2.g^{-1}). The iron (Fe) content in Layer 1 was higher than in Layer 2. This element can be presented in different phases and, consequently, with varying states of oxidation. Studies such as Siqueira *et al.* (2008) and Parsa and Chianeh (2012) prove the use and efficiency of iron oxide compounds in textile dye adsorption. In terms of silicon and aluminum oxides, they facilitate electrostatic attraction and cation exchange between the soil and the dye molecule (Freitas *et al.*, 2015).

Table 1. Particle-size distribution and soil classification.

Physicochemical Properties		Layer 1	Layer 2
Clay (%)		7.03	2.34
Silte (%)		10.55	3.52
Sand (%)		82.42	94.14
Textural class		Loamy Sand	Sand
Organic Carbon average (g.g^{-1})		1.26%	0.97%
Organic Matter average (g.g^{-1})		2.17%	1.67%
Specific Surface (m^2.g^{-1})		3.75	2.46
pH water		8.26	6.00
pH KCl		6.87	5.48
	Ca	2.70	0.75
	Mg	1.80	1.25
	Na	2.40	0.62
Exchangeable Bases (cmolc/dm^{-3})	K	0.10	0.07
	Al	0.00	0.00
	H	0.41	0.57
Sum of Bases (cmolc/dm^{-3})		7.0	2.7
Cation Exchange Capacity (cmolc/dm^{-3})		7.4	3.3
Saturation of Bases (%)		94	83
Fe (mg.kg^{-1})		13897±445	7928±443
Al (mg.kg^{-1})		64760± 1852	58802±1847
Si (mg.kg^{-1})		384913± 11468	416429± 11468

2.3. Point of Zero Charge

The surface charge of both soil layers was evaluated to define the pH value at which the adsorption of ions that determine H$^+$ and OH$^-$ potential is the same without interfering in the specific pH range of the environment. The methodology for the determination of the Point of Zero Charge (PZC) followed the recommendation proposed by Regalbuto and Robles (2004). The pH range where PZC occurred was used as an experimental adjustment parameter in the sorption experiments.

2.4. Direct Black 22

The dye used for this study was Direct Black 22 (DB22, CAS 6473-13-8), which was chosen because it is widely used by the textile industries of the Confection Center in Pernambuco's Agreste (Amorim *et al.*, 2013). Direct Black 22 is suitable for dyeing cellulosic fibers such as cotton, wool, viscose, rayon, and paper. It has the appearance of black powder and water solubility of 40 g.L^{-1} (at 90°C). Its molecular structure can be seen in Figure 2; molecular weight is 1083,97 g.mol$^{-1,}$ and melting point is higher than 360°C. The DB22 used in this study is from Henan Coreychem Co., Ltd, with 98% purity, and it was manufactured in China.
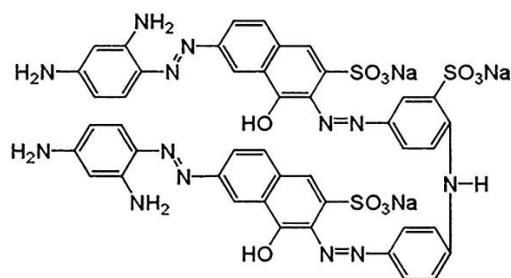
Figure 2. Molecular Structure of Direct Black 22.

2.5. Preparation of hydrolyzed dye

The DB22 solution was prepared with 65 mg of the dye diluted in 2 L of distilled water and was hydrolyzed according to Santos (2012) methodology. At this stage, the initial pH of the dye solution prepared with distilled water was adjusted to 11.00 with a 20% (w/v) concentration of the NaOH solution. The NaOH used is from the Merck company with a 98% degree of purity, and it was manufactured in Brazil. This solution was heated to 80°C for 60 minutes and stirred on a magnetic stirrer. At the end of this period and the solution cooling, the pH was adjusted until the PZC with HCl (0.1N) solution was obtained. The HCl used is from the Merck company with a 37% degree of purity, and it was manufactured in Brazil. This type of hydrolysis procedure is typically used in the preparation of dyes for dyeing garments in the textile industry. Thus, this solution served to analyze the behavior of the characteristic dye solution of textile industries in the study region.

2.6. Batch tests

Sorption tests were performed in triplicate and were based on the method described by Martins and Mermoud (1998) and Silva Júnior *et al.* (2019). For the kinetic study, 50 ml of a 32.5 mg.L^{-1} solution of DB22 and 5g of soil in a 250 ml Erlenmeyer were added. The samples were then placed in a shaking incubator and shaken for 0.25; 0.5; 1; 2; 4; 6; 8; 10; 12; 24; 48 and 72 hours. At the end of each experiment, the supernatant liquid was filtered through 0.45 µm membranes, and DB22 concentration was determined on a UV/VIS spectrophotometer with a wavelength of 481 nm.

The obtained data were used to calculate the amount of dye adsorbed per gram of residue, according to Equation 1.

$$St = \frac{Co - Ce}{M} \tag{1}$$

Where Co is the initial dye concentration in solution (mg.L^{-1}); Ce is the equilibrium concentration of the dye in solution (mg.L^{-1}); V is the volume of the solution (L); M is the dry mass of the adsorbent in solution (mg); St is the sorption in time (mg.kg^{-1}).

The curves resulting from the sorption kinetics were evaluated by fitting the data to the First and Second-Order models according to Equations 2 and 3, respectively (Yaneva and Koumanova, 2006).

$$log\,log\,(S_{e1} - S_t) = logS_{e1} - \frac{k_1}{2,303}t \tag{2}$$

$$\frac{1}{S_{e2} - S_t} = \frac{1}{S_{e2}} + k_2 t \tag{3}$$

Where S$_{e1}$ is the sorption capacity in equilibrium (mg.kg^{-1}) and k$_1$ is the first-order sorption constant rate (h^{-1}). The term S$_{e2}$ is the second-order sorption capacity in equilibrium (mg.kg^{-1}) and k$_2$ is the second-order sorption constant rate (kg.mg^{-1}.h^{-1}).

After defining the equilibrium time in the kinetic study, sorption isotherm experiments were performed to evaluate sorption capacity as a function of dye concentration. Samples were prepared in the same way as for sorption kinetics. DB22 concentrations used for this experiment were 1; 5; 10; 15; 20; 32.5 and 40 in mg.L^{-1}. The isotherm mathematical models used were the linear, Langmuir and Freundlich. These equations are efficient in determining the parameters of dye sorption in the soil (Caliman *et al.*, 2009). The Linear, Langmuir and Freundlich models are presented in Equations 4, 5 and 6, respectively.

$$S = K_D C_e \tag{4}$$

$$S = \frac{K_L S_0 C_e}{1 + K_L C_e} \tag{5}$$

$$S = K_F C_e^{1/n} \tag{6}$$

Where S is the amount of dye adsorbed in mg.kg^{-1} of soil, Ce is the dye concentration in the equilibrium solution (mg.L^{-1}), K_D is the soil-solution partition constant expressed in L.kg^{-1}; K_L represents the Langmuir constant expressed in L.mg^{-1}, S_0 is the maximum sorption capacity in mg.kg^{-1}, K_F is the Freundlich's constant expressed in (kg^{-1}mg$^{1-1/n}$L$^{1/n}$), and *n* is a dimensionless parameter.

3. RESULTS AND DISCUSSION

3.1. Point of Zero Charge
The determination of the PZC value is an essential step in this study. This information will assist in adjustments in the solutions' pH during the batch test. Studies such as Lucena (2018) prove the influence of this electrokinetic characteristic on dye sorption. The results of the point of zero charge (PZC), which was 5.02 for Layer 1 and 6.05 for Layer 2, indicate that the pH is acidic for both soils.

3.2. Sorption Kinetics
Sorption kinetics tests provide valuable insight into the behavior of dye sorption in soils. First and second-order kinetic models were used to help understand the behavior of the interaction between the pollutant and the solid soil matrix. The results of DB22 sorption kinetics for both soils can be seen in Figure 3.
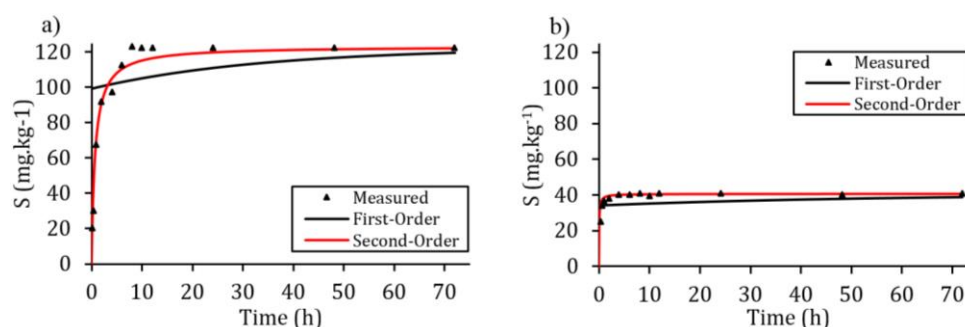

Figure 3. Sorption kinetics curve of the studied soils: a) Layer 1; b) Layer 2.

According to the results of sorption kinetics in Figure 3, Layer 1 required 8 hours to reach equilibrium and adsorbed approximately 120 mg of DB22 per kg of soil. Layer 2 achieved equilibrium in about 4h, and during this time, adsorbed approximately 40.86 mg.kg^{-1}. Layer 1 therefore presents more sorption sites, which, according to Yagub *et al.* (2014), increases dye

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sorption, and so adsorption on Layer 1 was thereby higher.

According to the obtained equilibrium times, it is possible to consider the dye sorption as an instantaneous phenomenon, in which chemical equilibrium is quickly reached. This consideration is based on kinetic studies in which equilibrium is achieved within no more than 24 hours (Bukallah *et al.*, 2007; Hong *et al.*, 2014; Dawodu and Akpomie, 2016). Regarding the equilibrium time, the results are in agreement with what is presented by Caliman *et al.* (2009), who concluded that the stirring time of 8 hours is the most appropriate to obtain the adsorption of an azo dye in a soil that has similar particle-size characteristics as the studied soil. Albanis *et al.* (2000) observed slower sorption, between 4 and 6 hours, for an azo dye in fly ash mixtures on a soil of low organic-matter content.

These results indicate that DB22 has a higher potential to be sorbed in Layer 1 than in Layer 2. This phenomenon can be explained by the higher presence of clay in Layer 1 (7.03%) than in Layer 2 (2.34%), and because the textile dye has a high ability to interact chemically with minerals in the soil (Duarte-Neto *et al.*, 2014; Santori *et al.*, 2011; Souza *et al.*, 2016). According to Table 1, Layer 1 has a higher OM content (2.17%) than Layer 2, resulting in a potentially higher reactivity between the soil and the DB22. According to Linhares *et al.* (2010), OM is a factor that influences the higher sorption of organic substances.

First-order kinetic model values were obtained from a linear regression between $\ln (S_e - S_t)$ by time (h). Regression coefficients for the first-order kinetic model were 0.39 and 0.12 for Layer 1 and Layer 2, respectively (Table 2). These low values demonstrated that DB22 sorption for the studied soils does not follow the first-order kinetic model.

The plots of t/S_t as a function of time (h) for the DB22 sorption with the soils of both layers at a concentration of 32.5 $mg.L^{-1}$ resulted in straight lines. Regression coefficients were higher than 0.999 for both soils (Table 2). This result indicates that the sorption kinetics of DB22 follow the second-order kinetic model.

Table 2. Kinetic parameters adjusted for the First and Second-order models for both soil layers.

Soil	First-order model			Second-order model		
	S_{e1} (mg.kg^{-1})	k_1 (h^{-1})	r^2	S_{e2} (mg.kg^{-1})	k_2 (kg.mg^{-1}.h^{-1})	r^2
Layer 1	23.17	0.0295	0.3955	123.4568	0.011511	0.9996
Layer 2	5.7902	0.0191	0.1249	40.6504	0.2420	0.9998

The sorption capacities' values in equilibrium corroborate the higher sorption affinity of DB22 with Layer 1 because for both adjusted models the values of S_{e1} and S_{e2} were higher. Qu *et al.* (2008) and Caliman *et al.* (2009) also noticed similar results in their studies with azo dyes, where the second-order model represented the process more faithfully. They found S_{e2} equal to 281 and 224 $mg.kg^{-1}$, respectively. The summary of the parameters obtained from the adjustments of the First and Second-Order kinetic models for the DB22 kinetics data and the studied soils are presented in Table 2.

3.3. Sorption Isotherms

Sorption isotherms are extremely important in the determination of maximum adsorption capacity of the studied soils, and in understanding the nature of the sorption process. The curves of DB22 sorption isotherms for the studied soils are presented in Figure 4. This study used Linear, Langmuir and Freundlich isotherm models to adjust the experimental data.

The adjustment of the isotherm to the Linear model is obtained from the regression between S_t and C_e, which resulted in linear regression coefficients of 0.99 for Layer 1 and 0.97 for Layer 2. This indicates that this Sorption isotherm model is suitable for both soils. K_D value

for Layer 1 was 5.28 L.kg^{-1}, and for Layer 2 was 0.99 L. kg^{-1}. K_D is a parameter often related to pollutant mobility in the soil and/or sediment. High K_D values indicate strong sorption and low values indicate weak interaction between the dye molecule and the solid matrix surface. Thus, low K_D values indicate high mobility and contamination risk of the surface and groundwater by leaching. Analyzing the K_D of the studied soils, Layer 1 has a higher ability to retain DB22, and Layer 2 has a higher leaching potential.
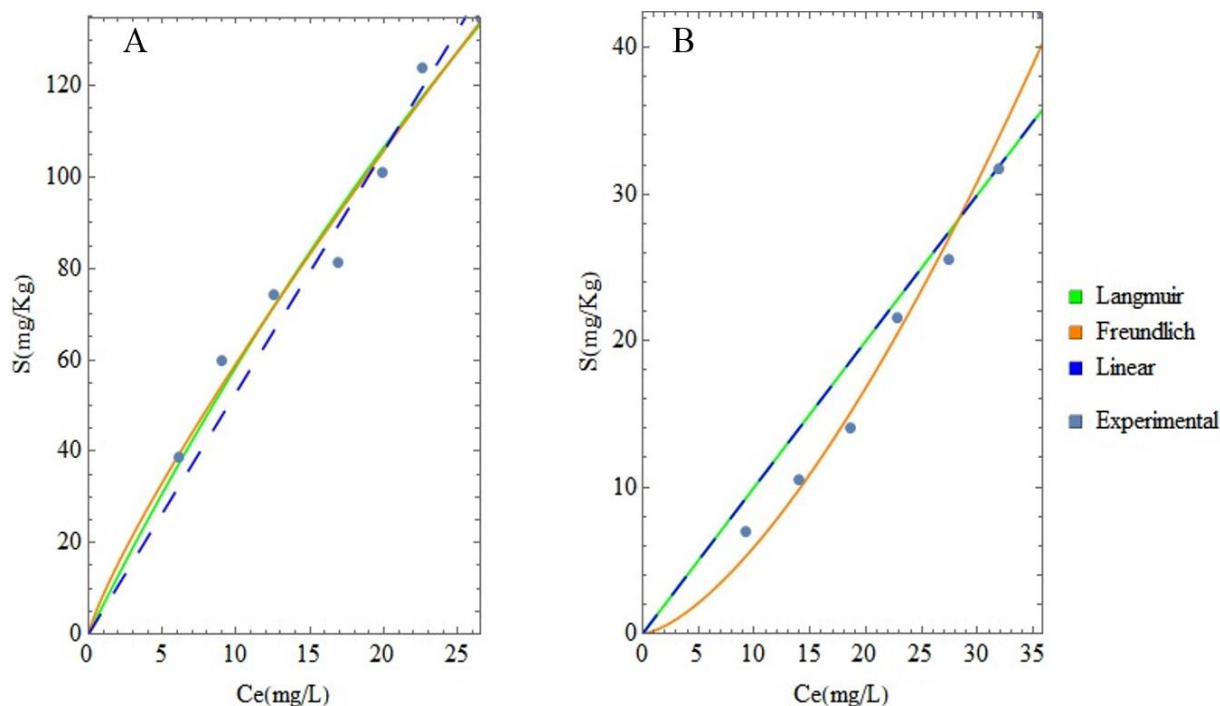

Figure 4. Langmuir, Freundlich, and Linear model adjustments for Layer 1 (A) and Layer 2 (B).

Analogous results were also found by Bukallah *et al.* (2007) and Dawodu and Akpomie (2016) in analyses performed with dye and soil with particle-size characteristics similar to the ones presented in this study. According to Ribeiro *et al.* (2007), most processes that cause contaminant attenuation in groundwater are more active in the biologically active soil zone, as a result of their higher clay and organic matter content.

Analyzing Figure 4, it is observed that the isotherms are better adjusted to the Freundlich model. The Langmuir model reasonably represents the experimental data of Layer 1, but it does not characterize Layer 2 well. The parameters of the linear, Freundlich, and Langmuir models are shown in Table 3. The values of the determination coefficients (r^2) of the Freundlich model for both layers are greater than 0.99. Therefore, the best adjustments of the experimental data for Layers 1 and 2 resulted from the application of the Freundlich model, as verified by Bukallah *et al.* (2007), Qu *et al.* (2008), and Dawodu and Akpomie (2016). The K_F values (Table 3) were 8.64 (kg^{-1}mg$^{1-1/n}$L$^{1/n}$) and 0.18 (kg^{-1}mg$^{1-1/n}$L$^{1/n}$) for Layers 1 and 2, respectively. The sorption capacity of DB22 is strongly correlated with the organic matter content and clay fraction, which contributes to the DB22 sorption, as these values are quantitatively higher in Layer 1. This finding agrees with Ketelsen and Meyer-Windel (1999), and Qu *et al.* (2008), who reported a positive correlation between the studied dyes and the soil clay content. Regarding K_F values, the results agree with other studies conducted with azo dyes such as Qu *et al.* (2008) and Uğurlu (2009).

Caliman *et al.* (2009), Morris *et al.* (2008), and Germán-Heins and Flury (2000) performed the adjustments according to Langmuir models for isotherms of a system composed of an azo dye and sandy soil. These authors obtained K_L (L.mg^{-1}) of 0.00365, 0.0124 and 0.0157,

respectively. The low values indicate that the binding energy between sandy soils and dyes of the same type as DB22 is weak, indicating high mobility.

Table 3. Langmuir and Freundlich isotherms parameters for the alluvial soil's layers of the Capibaribe River dry bed.

Models		Layer 1	Layer 2
Linear	$K_D(L.kg^{-1})$	5.286672	0.999598
	r^2	0.993927	0.979776
Freundlich	$K_F(kg^{-1}mg^{1-1/n}L^{1/n})$	8.640709	0.186793
	n	0.836228	1.501927
	r^2	0.996656	0.996152
Langmuir	$K_L(L.mg^{-1})$	0.011056	6.67E-05
	S_0	588.2885	15008.5
	r^2	0.996125	0.979655

4. CONCLUSIONS

A second-order model best described the sorption kinetics of DB22, and the Freundlich and Linear models properly adjusted the sorption isotherms for Layers 1 and 2. The organic matter content and the cation exchange capacity of both soil layers influenced the dye sorption. The superficial layer privileges dye retention phenomena while the subsurface layer favors dye mobility.

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