Disciplinarum Scientia. Série: Naturais e Tecnológicas, Santa Maria, v. 23, n. 3, p. 29-39, 2022.
Recebido em: 29.10.2022. Aprovado em: 20.12.2022.

ISSN 2176-462X

DOI: 10.37779/nt.v23i3.4374

ADSORPTION OF METHYLENE BLUE USING FISH SCALES AS BIOSORBENT[1]

ADSORÇÃO DE AZUL DE METILENO USANDO ESCAMAS DE PEIXE COMO BIOSORVENTE

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ABSTRACT

Access to water is fundamental for several sectors, such as human supply, irrigation, livestock, transport, energy, and manufacturing. However, the growing consumption associated with population increase, socio-economic and technological development, urbanization, and climate change, has substantially changed the availability and quality of this resource, mainly due to wastewater with dyes. Thus, the present study aims to evaluate the adsorption capacity of the tilapia fish scales (FS) as a biosorbent in removing the methylene blue dye (MB). The adsorption tests were carried out in batch mode, evaluating the effect of the MB concentration (10 - 40 mg L^{-1}), through the study of equilibrium, considering the Langmuir and Freundlich adsorption isotherms and kinetic adsorption of pseudo-first-order (PFO) and pseudo-second-order (PSO), with the biosorbent concentration of 0.7 g L^{-1}. It was observed that the more significant removal was achieved at 10 mg L^{-1} of MB (50.87%). Regarding the kinetic and equilibrium adsorption, all systems showed considerable adjustment to the PFO model and Freundlich isotherm model, respectively, coming into equilibrium after 15 minutes. Therefore, it is emphasized that the fish scale waste has great potential for application as a biosorbent to remove synthetic dyes, meeting the theme of sustainable development.

Keywords: Biosorption, Equilibrium, Kinetic, Sustainable Development.

RESUMO

O acesso à água é fundamental para diversos setores, como abastecimento humano, irrigação, pecuária, transporte, energia e manufatura. No entanto, o crescente consumo associado ao aumento populacional, desenvolvimento socioeconômico e tecnológico, urbanização e mudanças climáticas, tem alterado substancialmente a disponibilidade e qualidade desse recurso, principalmente devido aos efluentes com corantes. Assim, o presente estudo tem como objetivo avaliar a capacidade de adsorção das escamas de tilápia (FS) como biossorvente na remoção do corante azul de metileno (AM). Os testes de adsorção foram realizados em modo descontínuo, avaliando o efeito da concentração de AM (10 - 40 mg L^{-1}), por meio do estudo de equilíbrio, considerando as isotermas de Langmuir e Freundlich e a cinética de adsorção de pseudo-primeira ordem (PPO) e pseudo-segunda ordem (PSO), com concentração de biossorvente de 0,7 g L^{-1}. Assim, observou-se que a maior remoção foi alcançada com 10 mg L^{-1} de AM (50,87%). Em relação à adsorção cinética e de equilíbrio, todos os sistemas apresentaram considerável ajuste ao modelo PPO e ao modelo isotérmico de Freundlich, respectivamente, entrando em equilíbrio após 15 minutos. Por conseguinte,

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ressalta-se que o resíduo de escama de peixe tem grande potencial de aplicação como biossorvente para remoção de corantes sintéticos, atendendo ao tema do desenvolvimento sustentável.

Palavras-chave: *Biossorção, Equilíbrio, Cinética, Desenvolvimento Sustentável.*

INTRODUTION

The great increase in the world population in the last decades, added to the growing development of new products and technologies, has generated a very great need for water consumption by the population in general, but by industries (CICEK, 2003). These high-water resources consumption leads to a large generation of wastewater contaminated with a variety of products, such as heavy metals, organic dyes, and inorganic residues (FIGUEIREDO *et al*., 2020; KAMOUN *et al*., 2020; KESKIN *et al*., 2021). A dye widely used in the textile industry is methylene blue (MB) ($C_{16}H_{18}ClN_3S$), evaluated as a basic type of dye, with a heterocyclic aromatic molecular structure (ÖZACAR; ŞENGIL, 2003). Methylene blue is a cationic dye belonging to the class of dyes called thiazine (PANG *et al*., 2017; VINOD *et al*., 2010). In addition, MB is responsible for causing harmful effects on human health and the environment (ANFAR *et al*., 2017), but it is industrially distributed in the dyeing of cotton and silk fabrics (HAMEED *et al*., 2007).

To reduce environmental impacts and deal with the increase in water resources contamination, numerous researchers have been looking for solutions to remove or immobilize these contaminants (AHMED *et al*., 2017; AHMED *et al*., 2021; BHATNAGAR *et al*., 2011; CHAI *et al*., 2021; XU *et al*., 2021). Among these, the use of adsorbents for the capture of compounds, such as organic dyes, can be highlighted. These methodologies are favorable, as they do not generate toxic by-products, and low-cost sorbents of natural origin (biosorbents) can be used, such as rice husks, clays, agro-industrial residues, biomass, and fish scales (CHOWDHURY *et al*., 2012; DUARTE NETO *et al*., 2018; EL MOUHRI *et al*., 2021; KONGSRI *et al*., 2013; PAL; MAITI,2020; VIGNESHWARAN *et al*., 2021). Fish scales are waste from the fishing industry and correspond to about 4% of the waste generated by this industry (HUANG *et al*., 2011; LV *et al*., 2021), which is considered an environmental hazard without application in other industries. In addition, it is worth noting that the fish-derived food market has grown at high speed in recent decades (COPPOLA *et al*., 2021) Fish scales are materials that have their composition structures derived from calcium phosphates (hydroxyapatite) and collagen, which makes it possible the exchange of ions and the adsorption of different compounds to its structure (ATHINARAYANAN *et al*., 2020; HUANG *et al*., 2011).

Therefore, to contribute to the reuse of discarded waste and, at the same time, make it useful for the removal of organic pollutants, the present work aims to evaluate the efficiency of methylene blue dye adsorption in an aqueous solution, using tilapia fish scales as biosorbent. Furthermore, determine the parameters of the Langmuir and Freundlich isotherms to identify the best model that fits the experimental data, as well as the kinetic parameters.

MATERIALS AND METHODS

MATERIALS

For the adsorption experiments, methylene blue (MB, Neon, PA, Brazil) was used as adsorbate in a batch regime. The concentration was evaluated at 10, 20, 30, and 40 mg L^{-1}. The concentration of the MB solution was measured based on the constructed calibration curves with an adsorption wavelength of 666 nm. Fish scales (FS) from *Oreochomis niloticus* were collected manually at room temperature (25 ± 2 ºC), washed in distillate water to remove impurities, and then dried in a muffle furnace (60ºC for 4 h). Thereafter, the FS was ground in a ball mill (CT- 241 Mill), using alumina spheres (Al_2O_3), for 15 min and sieved (#45 - Dp < 2 mm).

ADSORPTION EQUILIBRIUM

The adsorption tests were evaluated in a batch regime (DOĞAR *et al.*, 2010). A volume of 100 mL of the four MB solution previously prepared was analyzed, using 0.7 g L^{-1} of FS to each one, for 210 min, under a magnetic stirrer (150 rpm). At pre-set time intervals (0, 5, 15, 30, 45, 60, 75, 90, 120, 150, 180, and 210 min), 2 mL samples were taken, filtered (0,22 µm, Millex GP), and analyzed by a UV-vis spectrophotometer (Cary 100Scan, United States), to calculate the amount of MB adsorbed. A duplicate analysis was conducted. The removal percentage of the MB - R(%), the maximum adsorption capacity (q_e), the Langmuir ($Q_{eq,L}$), and Freundlich ($Q_{eq,F}$) isothermal models are presented in Equations (1)-(4), respectively (CHEN; WANG, 2007).

$$R(\%) = \frac{C_0 - C_t}{C_0}.100 \quad (1)$$

$$q_e = \frac{V.(C_0 - C_t)}{m} \quad (2)$$

$$Q_{eq,L} = \frac{Q_{máx}.K_L.C_{eq}}{1 + K_L.C_{eq}} \quad (3)$$

$$Q_{eq,F} = K_F.C_{eq}^{\frac{1}{n}} \quad (4)$$

Where: C_0 the initial MB dye concentration (mg L^{-1}); C_t the MB dye concentration at time t (mg L^{-1}); *V* is the solution volume (L); *m* is the mass of adsorbent (g); Q_{max} is the maximum adsorption capacity, related to the monolayer adsorption capacity (mg g^{-1}); K_L the Langmuir constant (L mg^{-1}); K_F the

Freundlich constant [(mg g^{-1}) (mg L^{-1})$^{-1/n}$]; C_{eq} is the equilibrium concentration of the solute in solution (mg L^{-1}); and *1/n* the heterogeneity factor (dimensionless).

An essential characteristic to be evaluated in the Langmuir isotherm is the separation factor or equilibrium parameter (R_L) indicating the isothermal shape and behavior allowing the determination if the isothermal is favorable, linear, or if the process is irreversible, according to Equation (5) (NASCIMENTO *et al.*, 2014):

$$R_L = \frac{1}{1 + K_L . C_0} \quad (5)$$

Where: $R_L > 1$: favorable adsorption; R_L: linear; $0 < R_L < 1$: favorable adsorption; and $R_L = 0$: irreversible process.

ADSORPTION KINETICS

Kinetics models were used to evaluate the main mechanism of the adsorption process (Chemical reaction, diffusion-controlled and mass transfer), highlighting the pseudo-first-order (PFO) and pseudo-second-order (PSO), according to Equations (6) and (7), respectively (ÖNAL 2006):

$$q_t = q_1 \left[1 - \exp(-k_1 . t)\right] \quad (6)$$

$$q_t = \frac{t}{(1/k_2 q_2^2) + (t/q_2)} \quad (7)$$

Where:

q_1: theoretical value of adsorption capacity, considering a pseudo-first order (mg g^{-1});

k_1: kinetic constant of pseudo-first order (min^{-1});

q_2: theoretical value of adsorption capacity, considering a pseudo-second order (mg g^{-1});

t: time (min^{-1})

k_2: kinetic constant of pseudo-second order (g mg^{-1} min^{-1}).

STATISTICAL ANALYSIS

The kinetic parameters were determined by adjusting the models to the experimental data, using non-linear regression. The values were obtained using Statistic 10 (StatSoft, United States), considering the Quasi-Newton methods.

RESULTS AND DISCUSSION

ADSORPTION TESTS AND ADSORPTION CAPACITY

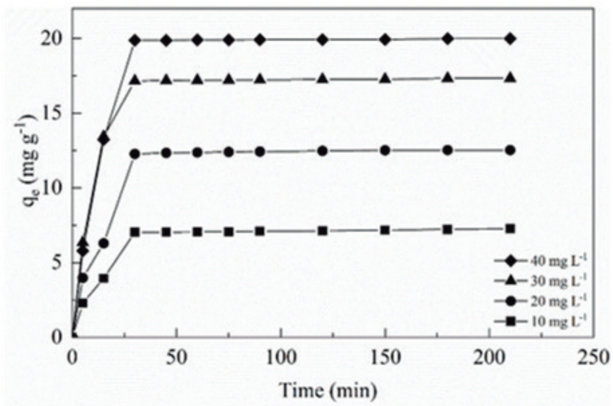
Table 1 presents the results of the removal percentage of the MB, R (%), as a function of the adsorbate concentration. It is possible to conclude that as lower the solution concentration, the higher is the adsorption capacity. Moreover, the maximum adsorption capacity (q_e), increases with the adsorbate concentration in the solution (Figure 1), in agreement with the literature (PATHANIA *et al.*, 2017). The concentration and the contact time between adsorbent and adsorbate are important information for the treatment of wastewater by adsorption. The quick remotion of the adsorbate and the achievement of equilibrium in low concentrations in a short period indicate that the adsorbent is efficient (MALL *et al.*, 2006). According to Figure 1, above 30 min, the value of the maximum adsorption capacity (q_e) is constant to all concentrations of MB evaluated, indicating its quality.

Table 1 - Removal percentage of the MB as a function of the adsorbate concentration.

Initial dye concentration (mg L^{-1})	Removal percentagem R (%)
10	50.8647 ± **0.05**
20	43.8701 ± **0.0**4
30	40.4497 ± **0.05**
40	34.9731 ± **0.0**3

Source: Author's construction

Figure 1 - Maximum adsorption capacity of MB ()using FS, to different concentrations of MB.



Source: Author's construction.

ADSORPTION EQUILIBRIUM

Table 2 presents the equilibrium constants to the Langmuir and Freundlich isotherms. Considering the Langmuir isotherm, the maximum adsorption capacity (Q_{max}) was found among

1.353 and 2.759 mg g^{-1}. For all concentrations of MB evaluated it is possible to admit that the adsorption is favorable, considering the value of equilibrium parameter (K_L), which must be between 0 and 1 (NASCIMENTO *et al.*, 2014).

It is important to highlight that the Langmuir isotherm is based on the movement of the molecules in the adsorbent surface, providing a uniform distribution (monolayer and homogeneous), where the adsorption occurs in specific sites and without particle interaction (CHEN; WANG, 2007).

The Freundlich isotherm is the best isotherm model, presenting the highest R^2, compared to the Langmuir isotherm, indicating multilayer adsorption (physical adsorption) in non-uniform sites, characteristic of heterogeneous systems (GUPTA *et al.*, 2009). Evaluating the parameter n, from Friedman isotherm, which determines the adsorption intensity, values among 0 and 10 can be found, suggesting favorable adsorption (NASCIMENTO *et al.*, 2014).

Table 2 - Parameters obtained considering Langmuir and Freundlich isotherms with FS (0,7 g L^{-1}).

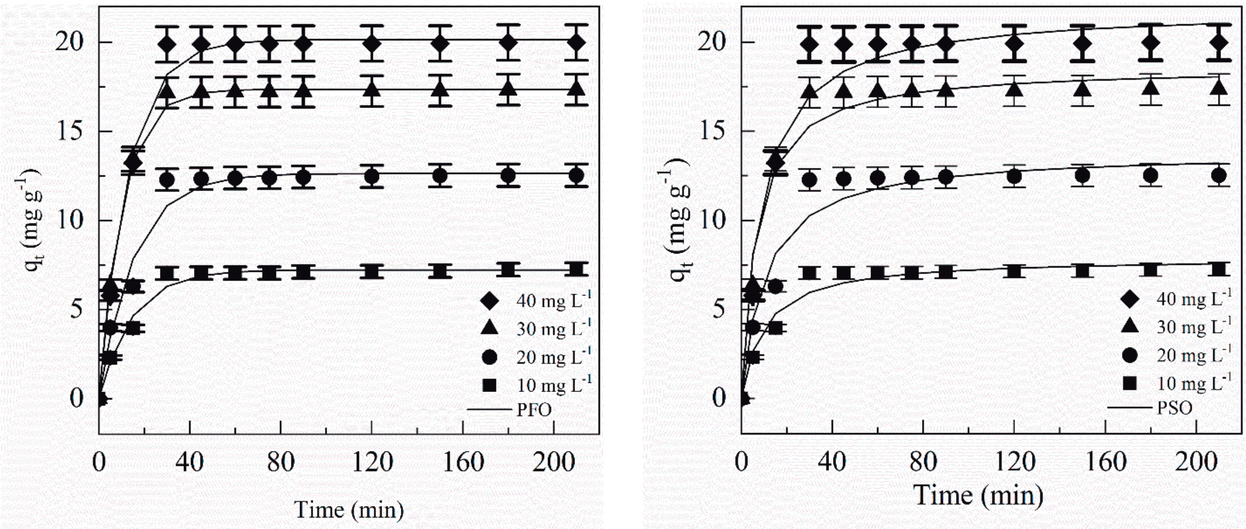
Isothermal model	**Parameter**	**MB (10 mg L^{-1})**	**MB (20 mg L^{-1})**	**MB (30 mg L^{-1})**	**MB (40 mg L^{-1})**
Langmuir	Q_{max} (mg g^{-1})	1.353 ± 0.05	2.018 ± 0.05	2.759 ± 0.05	2.296 ± 0.05
	K_L (L g^{-1})	0.245 ± 0.05	0.105 ± 0.05	0.066 ± 0.05	0.043 ± 0.05
	R_L	0.289 ± 0.05	0.322 ± 0.05	0.431 ± 0.05	0.536 ± 0.05
	R^2	0.903	0.929	0.935	0.901
Freundlich	n	0.499 ± 0.05	0.4402 ± 0.05	0.369 ± 0.05	0.278 ± 0.05
	R^2	0.976	0.984	0.982	0.973

Source: Author's construction

ADSORPTION KINETIC

Figure 2 presents the adsorption kinetic of the MB into different concentrations (10 mg L^{-1} to 40 mg L^{-1}) using the FS. The adsorption is quick and in 15 min the equilibrium is reached to all concentrations evaluated. Moreover, there is a proportionality between the adsorbate concentration and the adsorption capacity (q_t) what can be attributed to the reduction in the adsorption sites unoccupied and due to the diminution in the concentration gradient. The adsorptions capacities are almost constants after 30 min. *Pseudo first- order* (PFO) and *pseudo second-order* (PSO) models were evaluated and the results are presented in Figure 2 (a) and (b), respectively.

Figure 2 - Adsorption kinetic of methylene blue (MB) for
(a*) pseudo first-order* (PFO) and (b)*pseudo second-order* (PSO)



(a) (b)

Source: Author’s construction

Table 3 presents the kinetic parameters obtained considering PFO and PSO models. Considering the R^2, the PFO presents the best fit to experimental data, which indicates that this is the best model to represent the adsorption kinetic. Therefore, we can suggest that the adsorption process occurs through the electrostatic interaction, reversibly, suggesting physical adsorption. Besides, this model establishes that the adsorption velocity is proportional to the number of free sites, typical of a physisorption process (MIMURA *et al.*, 2010).

Table 3 - Kinetic parameters to MB adsorption using FS (60 mg L^{-1}).

Model	**Concentration (mg L^{-1})**	**q_1 (mg g^{-1})**	**k_1 (min^{-1})**	**R^2**
Pseudo first-order (PFO)	10	7.2235	0.0686	0.9829
	20	12.6355	0.0647	0.9752
	30	17.3556	0.0986	0.9979
	40	20.1446	0.0777	0.9919
	Concentration (mg L^{-1})	q_2 (g mg^{-1} g^{-1})	k_2 (g mg^{-1} min^{-1})	R^2
*Pseudo second-order (*PSO)	10	7.9136	0.0127	0.9601
	20	13.8783	0.0068	0.9489
	30	18.6200	0.0082	0.9737
	40	21.9237	0.0052	0.9614

Source: Author’s construction

CONCLUSION

In this work, the adsorption of methylene blue using fish scales as biosorbent. Considering the results evaluated, it is possible to conclude that the FS can be used as a biosorbent in nature to remove the methylene blue from wastewater to the concentrations evaluated. The best removal potential was reached at a concentration of MB of 10 mg L^{-1} with adsorption of 50.87%. The equilibrium data were adjusted to Langmuir and Freundlich isotherms which confirmed that the adsorption is heterogeneous and occurs considering the physical-chemical interactions. The Freundlich isotherm was the best to represent the experimental data, considering the R^2. The pseudo first-order is the best to represent the kinetics of the adsorption process. Therefore, the fish scales present potential as an alternative biosorbent to wastewater treatment to the methylene blue dye.

ACKNOWLEDGEMENT

We thank the Franciscan University (UFN) for the support and assistance to carry out the present work.

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