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Vermicompost for Indigo Blue and Congo Red Removal

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Abstract

The data obtained through this work revealed that the vermicompost is a natural adsorbent able to removal two textile dyes from an aqueous medium. The values of maximum adsorption capacity for congo red (23.25 mg/g) and indigo blue (40.39 mg/g) obtained from the Mathematical Langmuir Model reveal it. The conditions of adsorbent mass, stirring time between adsorbent and dyes were optimized. Additionally, the values of Gibbs free energy demonstrate the predominance of physical interaction between both dyes and vermicompost. Through Langmuir constant values, it was possible to identify similar affinities between both dyes and vermicompost. The value of dimensionless constant indicates favorable adsorptions process. Finally, through physicochemical analysis from scanning electron microscopy and Fourier Transform Infra-Red Spectroscopy, the characteristics of vermicompost were verified revealing essential aspects to efficient adsorbent.

Keywords

Vermicompost, Adsorption, Congo Red, Indigo Blue

1. Introduction

The adsorption of toxic dyes pollutants using natural adsorbents is widely studied [1]. For example, Kebaili *et al.* [2] studied orange industry residues as an adsorbent for methylene blue. Sun *et al.* [3] evaluated the use of *Angelicae dahuricae* residue for the adsorption of methylene blue. Ribeiro, A. *et al.* [4] evaluated the liquorice *Glycyrrhiza glabra* L. root powder for the removal of congo red and indigo blue in the water. Munagapati *et al.* [5] investigated the removal of reactive black 5 and congo red using banana peel. Raymundo *et al.* [6] evaluated sugar-cane bagasse as an adsorbent in the textile wastewater treatment contaminated with congo red dye. Wang *et al.* [7] utilized a green composite hydrogel based on cellulose and clay as an efficient absorbent of colored organic effluent. The optimized cellulose/montmorillonite hydrogels showed excellent methylene blue adsorption. Kubra *et al.* [8] showed that a polymeric natural carbohydrate was able to removal methylene blue dye from an aqueous medium. Jawad and Abduhameed [9] revealed the mesoporous Iraqi red kaolin clay as an efficient adsorbent for methylene blue dye. Pereira *et al.* [10] evaluated the vermicompost for organic cationic dyes retention. The results obtained by these authors were satisfactory for removal of crystal violet and methylene blue. Cygeroglu and Yildirir [11] evaluated the vermicompost to removal of methylene blue from aqueous medium obtaining excellent results. This same material was used in the bleaching of water containing other polluting dyes [12]. The vermicompost can be also used for the retention of other pollutants such as pesticides [13], metals ions [14], and pharmaceuticals [15]. The vermicompost has a complex physical and chemical structure and its production occurs in the digestive system of certain species of earthworms which feed on organic residues present in the soil. In the digestive system of these earthworms occurs the fragmentation of several biomolecules such as proteins and nucleic acids generating complex molecules rich in aromatic rings, carboxyl groups, and others which consequently returns to the soil and is denominated vermicompost [16]. In this work we studied the ability of vermicompost (VMC) to remove textile dyes present in aqueous medium. The dyes used were Congo Red (CR) and Indigo Blue (IB). Effluents of the industrial textile dye containing IB and its derivative anthranilic acid are hazardous to the aquatic environment [17] and CR is a toxic dye that has carcinogenic properties [18]. For this study, physicochemical tests were performed using Scanning Electron Microscopy (SEM) and Fourier Transform Infra-Red Spectroscopy (FTIR) to verify the vermicompost characteristics. Subsequently, the following parameters were evaluated: 1) mechanical agitation time between dye and adsorbent, 2) influence of adsorbent mass and dye concentration on the adsorptive process. Finally, the maximum adsorptive capacity (MAC) of vermicompost for Indigo Blue and Congo Red were calculated using the Langmuir mathematical model [10]. These experiments aimed to reveal the efficiency of vermicompost as a removing agent for CR and IB in aqueous media.

2. Material and Methods

2.1. Materials

Vermicompost was obtained from Suzan Humus (Suzano-SP, Brazil), congo red dye (CR) was purchased from Vetec Company (Duque de Caxias-RJ, Brazil), while Indigo Blue (IB) was purchased from Tupy Industry (São Paulo-SP, Brazil). Ultrapure water used to prepare all solutions was obtained from a water purification unit Quimis (Q241-22 model, Diadema-SP, Brazil). Several laboratory glasswares were used, including beckers, erlemeyers, kitassato, and volumetric flasks. Others equipments were used such as: laboratory oven (Quimis Q-317 B model, Brazil), particle size sieves (Granutest, Brazil), Analytical balance (Shimadzu AY 220 model, Japan), sputter coater (Shimadzu, IC-50 Ion Coater model, Japan), scanning electron microscope (Shimadzu, SSX 550 model, Japan), infrared spectrophotometer (Cary 630 model, Agilent Technologies, Santa Clara, California, USA), pHmeter (PHTEK, Labitec, Londrina-PR, Brazil), magnetic stirrer (Warmnest, 78HW-1 model, Brazil), UV/Vis spectrophotometer (Even, IL-562 model, Brazil). For data processing, a graphical/statistical program Origin version 6.1 was used (OriginLab Corporation, Northampton, MA 01060, United States).

2.2. Methods

2.2.1. Adsorbent Preparation

The vermicompost was sieved to obtain particle sizes between 2.38 and 4.76 mm subsequently, the material was dried at 50 Celsius for 15 hours. The Crushed vermicompost shape can be seen in **Figure 1**.

2.2.2. Scanning Electron Microscope Analysis

Vermicompost surface was analyzed using a Scanning Electron Microscope (SEM). Before analysis, a thin layer of gold covered vermicompost particles using the spray coating. Subsequently, VMC surface was visualized utilizing an electron beam that was accelerated at 20 kV.

2.2.3. Fourier Transform IR Spectroscopy Analysis

This analysis was performed to identify functional groups on the vermicompost particles using Fourier Transform IR (FTIR) Spectroscopy at a spectral range from 4000 to 600 cm^{-1}. Typically, a powder vermicompost sample was introduced into a diffuse reflectance accessory.

2.2.4. Mechanical Agitation Time

Aqueous solutions (50.00 mL) containing Congo Red (CR) at 40 $mg \cdot L^{-1}$ or Indigo

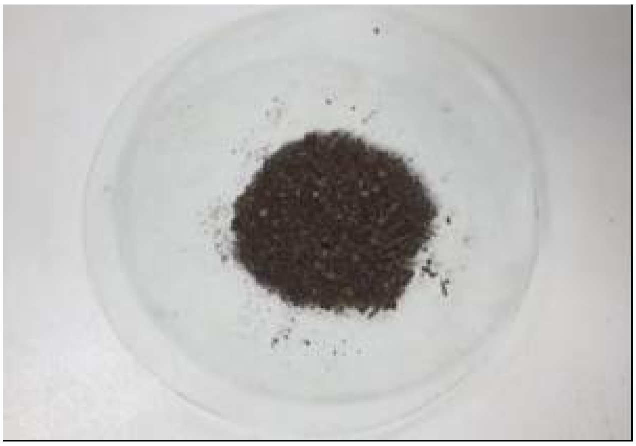

Figure 1. Crushed vermicompost shape.

Blue (IB) at 1000 mg·L^{-1} were prepared at pH 7.0. Subsequently, these solutions were stirred at 1000 rpm (298.15 K) at different mechanical agitation times (5, 10, 15, 20, 25, and 30 minutes) with 3.0 g of vermicompost. The solutions were filtered at vacuum. The absorbance of supernatants was measured at 573 and 500 nm for IB and CR, respectively. The aim of this test was to evaluate the more adequate mechanical agitation time. These experiments were performed as described by [4] who completed the experiments in triplicate. The percentage of dye retained on the adsorbent at different mechanical agitation times was calculated using Equations (1) and (2).

$$adc = idc - sdc \quad (1)$$

$$\begin{array}{l} idc \text{ ----------}100\% \\ adc \text{ --------- } x \end{array} \quad (2)$$

In Equation (1), *adc* is adsorbed dye concentration, *idc* is the initial dye concentration (40 mg·L^{-1} and 1000 mg·L^{-1} for CR and IB, respectively) before mechanical agitation time and filtration, while *sdc* is the dye concentration (mg·L^{-1}) in the supernatant after filtration. The *sdc* values were estimated after supernatant quantitative analyses in which dye calibration curves were daily built according to the following concentration ranges: 3 to 50 mg·L^{-1} (CR) and 10 to 1000 mg·L^{-1} (IB). In turn, Equation (2) was used to estimate the percentage of dyes (CR or IB) that were adsorbed on vermicompost particles after each mechanical agitation time. After these calculations, a graph relating dye adsorption percentage (%) and mechanical agitation time was built. These experiments were performed in triplicates and using analytical blanks for each mechanical agitation time. In this case, the analytical blanks consisted of 50.00 mL ultrapure water and 3.0 g of vermicompost.

2.2.5. Vermicompost Mass

In this experimental part, it was evaluated the influence of vermicompost mass on the adsorption of IB and CR. To perform the experiments, 50.00 mL of aqueous solutions (pH 7.0) containing IB (1000 mg·L^{-1}) or CR (40 mg·L^{-1}) were stirred (1000 rpm) (25˚C) during 10 and 25 minutes for IB and CR respectively (times selected in the previous step). The solutions containing CR or IB were stirred in the presence of different vermicompost masses (0.50, 1.0, 2.0, 3.0, 4.0, 5.0 and 6.0 g). Subsequently, the solutions were filtered at vacuum for retention of vermicompost particles containing IB or CR. The absorbance of supernatants was measured in triplicate using a spectrophotometer (573 and 500 nm for IB and CR respectively) to evaluate the percentage (%) of these dyes retained on different vermicompost masses. Equations (1) and (2) were also employed to calculate these adsorption percentages, which were plotted against the different vermicompost masses. The percentage of dye retained on different vermicompost masses was also calculated using Equations (1) and (2). These experiments were performed in triplicates and using analytical blanks for each vermicompost mass. In this case, the analytical blanks consisted of 50.00 mL ultrapure water and different masses of vermicompost.

2.2.6. Dye Concentration Influence, Adsorption Isotherms and Derivations of Thermodynamic Parameters

After determining the more adequate mechanical agitation times and vermicompost masses for both dyes, adsorption isotherms were built using different concentrations of CR and IB, whose pH values were adjusted to 7.0. For this purpose, vermicompost particles were mechanically stirred (1000 rpm), at 298.15 K, with 50.00 mL CR or IB solutions with concentrations ranging from 50 to 500 mg·L^{-1} and from 50 to 4000 mg·L^{-1}, respectively. For CR solutions, 1 g of vermicompost and 10 minutes of mechanical agitation time were adopted. In turn, for IB solutions, 3 g of vermicompost and a mechanical agitation time of 25 minutes were fixed to build adsorption isotherms. Subsequently, the solutions were filtered at vacuum for retention of vermicompost particles containing CR or IB. The supernatant absorbances were measured in a spectrophotometer (573 nm for IB and 500 nm for CR) to evaluate the amounts of CR and IB (mg) retained per gram of vermicompost. Once the adsorption isotherms were built for CR and IB, they were linearized according to the Langmuir Mathematical Model [10] to estimate the following thermodynamic parameters concerning the evaluated adsorptions: maximum adsorption capacity (*MAC*), Langmuir constant (K_L) that is related to the adsorption energy, variation of Gibbs free energy, and the dimensionless constant (R_L) associated with adsorption processes.

3. Results and Discussion

3.1. Scanning Electron Microscope Analysis

The Scanning Electron Microscope (SEM) analysis of the vermicompost resulted in a morphological aspect as shown in **Figure 2**. According to this figure, vermicompost particle surface had heterogeneous and irregular morphology. These morphological characteristics contribute to larger surface area and, consequently, to the adsorption processes [19]. Similar results using SEM for vermicompost were found by [20]. Pereira *et al.* [10] also verified that the vermicompost, which was efficiently used to adsorb crystal violet and methylene blue dyes, presented very irregular surface.

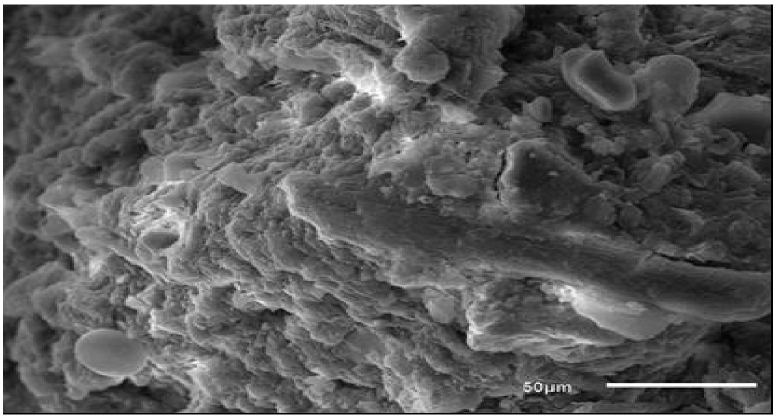

Figure 2. Scanning electron microscopy of the vermicompost with magnifications of 1000×.

3.2. Fourier Transform IR Spectroscopy Analysis

Figure 3 indicates the presence of chemical groups able to adsorb pollutants chemical species as those evaluated in this study [21]. As can be seen, infrared spectrum of vermicompost revealed characteristic bands, as those associated to N-H stretching (asymmetrical and symmetrical) at 3694 cm^{-1} and 3619 cm^{-1} [20] [22]. Vermicompost spectrum was also characterized by a strong and broad band from 3500 - 3000 cm^{-1} (3522 cm^{-1} and 3436 cm^{-1}) associated to the stretching υ (O-H) of adsorbed water molecules or H-bonded OH groups, including those presents in acid carboxylic, phenolic, and alcoholic groups [20] [23]. The weak absorption at 2900 cm^{-1} was related to aliphatic C-H stretching, indicating the absence of large amounts of CH_2 and CH_3 groups [20] [23]. Absorption between 1750 - 1500 cm^{-1} (1640 cm^{-1}) was associated to C-O and C=O stretching bands attributed to the COOH groups, or to C-H of alkene group (Silverstein *et al.*, 1991; Stevenson & Goh, 1971). Adsorption band at 1400 cm^{-1} was due to OH deformation and C-O stretching of phenolic OH groups, and to C-H deformation of CH_2 and CH_3 groups [22] [23]. The broad and weak band at 1200 cm^{-1} is assigned to C-O stretching and OH deformation of COOH. The strong bands at 1002 cm^{-1} and 909 cm^{-1} were associated to the C-H stretching of alkene group [22].

Figure 4 reveal that through these characteristics showed in **Figure 3** is possible to suppose some interactions between some vermicompost chemical groups and both dyes. The hydrogen bond appears as one of these possible chemical interactions. However, the hydrogen bonds are pH medium dependent. In this experiment we utilized aqueous solutions dyes with pH around 7.0.

3.3. Mechanical Agitation Time

As can be seen in **Figure 5**, mechanical agitation times of 10 and 30 minutes were associated with the higher adsorption percentages of Indigo Blue. It is

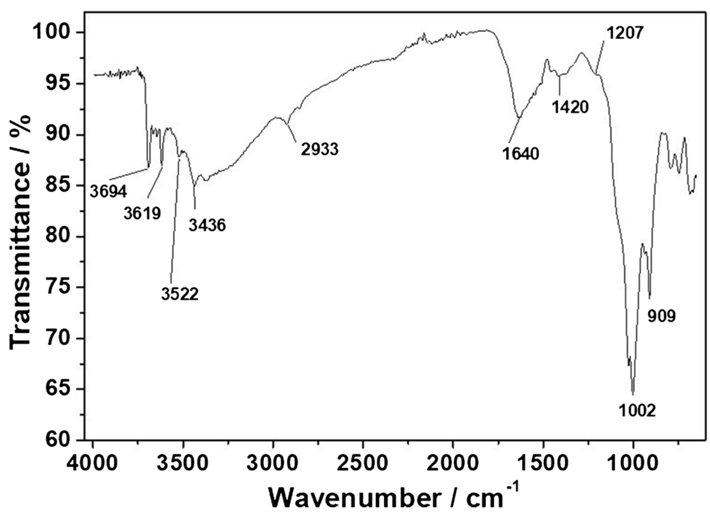

Figure 3. FTIR spectrum of vermicompost.

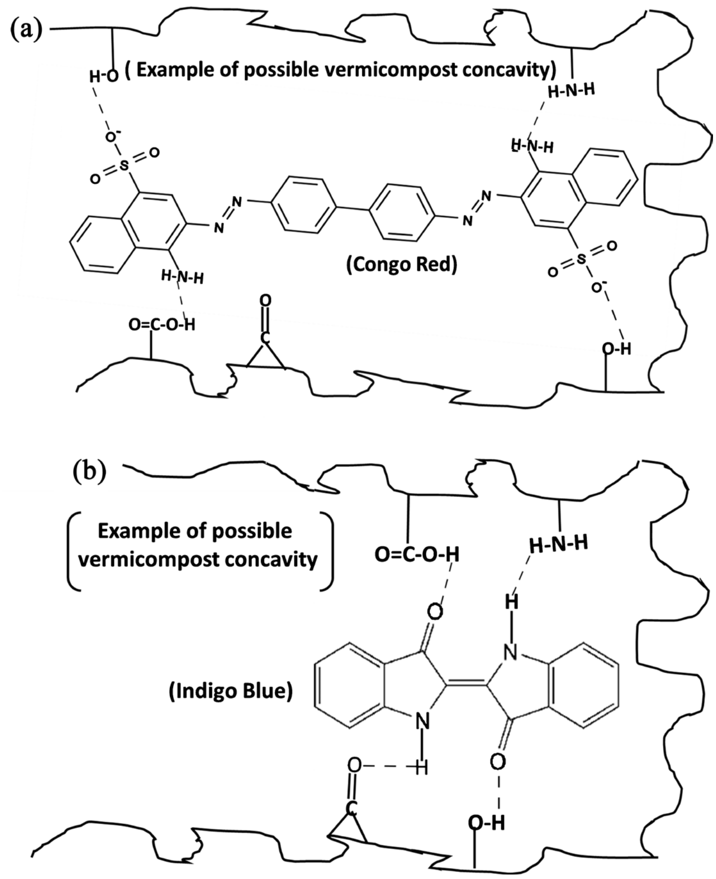

Figure 4. Example of some possible chemical interactions between some chemical groups of vermicompost and Congo Red (a) and Indigo Blue (b).

important to note that there was no significant difference between adsorptions achieved at 10 and 30 minutes. Thus, the mechanical agitation time of 10 minutes was adopted in all adsorption tests involving Indigo Blue. In turn, as indicated in **Figure 5**, there was a clear favoring of Congo Red adsorption at 25 minutes, thus justifying the use of this mechanical agitation time in all adsorption tests with Congo Red. Moreover, it was possible to notice desorption events along the time scale indicated in **Figure 5**. Raymundo *et al.* [6] also observed similar results, which were achieved when sugar cane bagasse was used to adsorb Congo Red. Ribeiro *et al.* [24] also noticed that adsorption of Indigo Blue by *Ziziphus joazeiro* peel showed desorption events as mechanical agitation time increased.

3.4. Vermicompost Mass

The influence of different vermicompost masses (g) on the adsorption of Congo Red (40 mg·L^{-1}) and Indigo Blue (1000 mg·L^{-1}) is plotted in **Figure 6**. It was shown that Congo Red adsorption percentage presented small increase from 0.5 to 1.0 g of vermicompost (approximately 96% to 100%). From 1.0 g, adsorption percentage of Congo Red remained practically constant. Therefore, 1.0 g of vermicompost was selected for determining the maximum adsorption capacity for

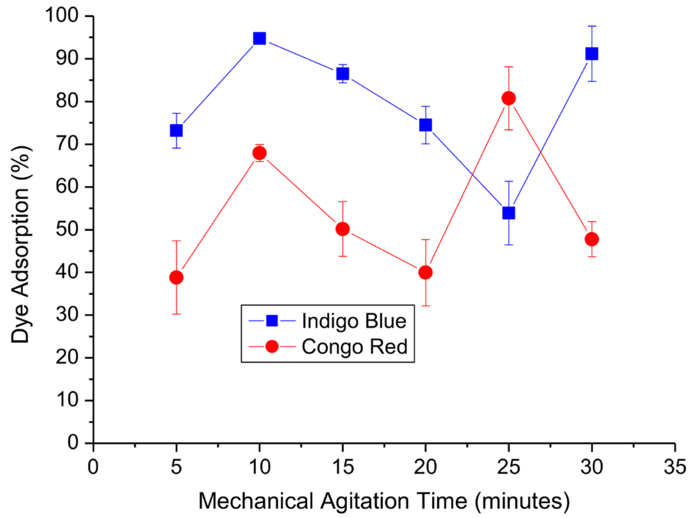

Figure 5. Influence of stirring time on Indigo Blue and Congo Red adsorption (%).

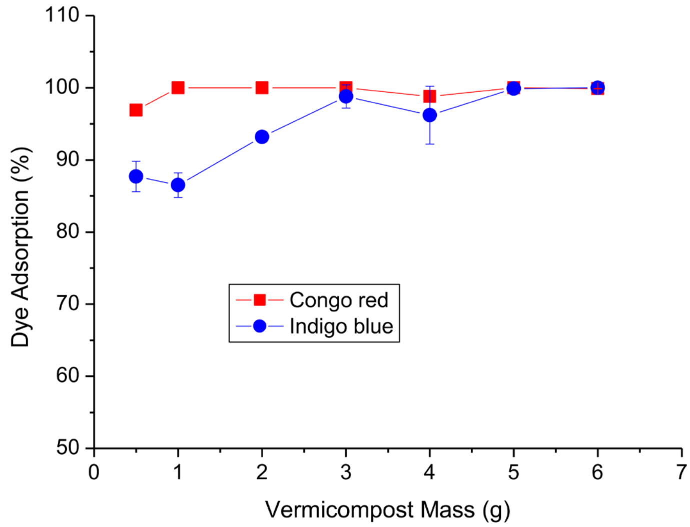

Figure 6. Influence of vermicompost mass on Indigo Blue and Congo Red adsorption (%).

Congo Red. Indigo Blue adsorption percentage increased when vermicompost mass varied from 0.5 and 3.0 g (approximately 87.7% to 99.9%), remaining unchanged with the addition of larger masses (**Figure 6**), thus justifying the use of 3.0 g of vermicompost along the tests to estimate the maximum adsorption capacity for Indigo Blue. Raymundo *et al.* [6] utilized sugar-cane bagasse as adsorbent for treating textile wastewater contaminated with Congo Red (100 μM) to remove approximately 88% of this dye using 10 g of adsorbent. This outcome was considerably different in relation to the vermicompost evaluated in this work (**Figure 6**). In a work developed by [25], it was determined that 1.5 g of wood sawdust powder from *Corymbia citriodora* was able to remove approximately 90% of Congo Red (25 $mg \cdot L^{-1}$) from aqueous medium. Ribeiro[A] *et al.* [4] ascertained the liquorice *Glycyrrhiza glabra* L. root powder as a natural adsorbent to remove textile dyes in water. They demonstrated that 2.0 g of this adsorbent were able to remove 90% and 89% of Congo Red (25 mg·L^{-1}) and Indigo Blue (1000 mg·L^{-1}), respectively. Ribeiro *et al.* [24] demonstrated that 1.5 g of *Zizyphus joazeiro* Mart peel removed approximately 84% of Indigo Blue (1000 mg·L^{-1}) from aqueous medium.

3.5. Dye Concentration Influence, Adsorption Isotherms and Derivations of Thermodynamic Parameters

The no linearized adsorption isotherms for both dyes (**Figure 7** and **Figure 8**) were built the ration q (mg·g^{-1}) against C_{eq} (mg·L^{-1}), where q (mg·g^{-1}) is the adsorbed amount of dye per gram of vermicompost in each dye concentration evaluated, while C_{eq} (mg·L^{-1}) is the equilibrium concentration of dyes in the supernatants (dye amount no adsorbed by vermicompost). The forms of this isotherms indicate relatively favorable conditions for adsorption [26]. Mathematically, an adsorption isotherm can be treated by different models, but we adopted

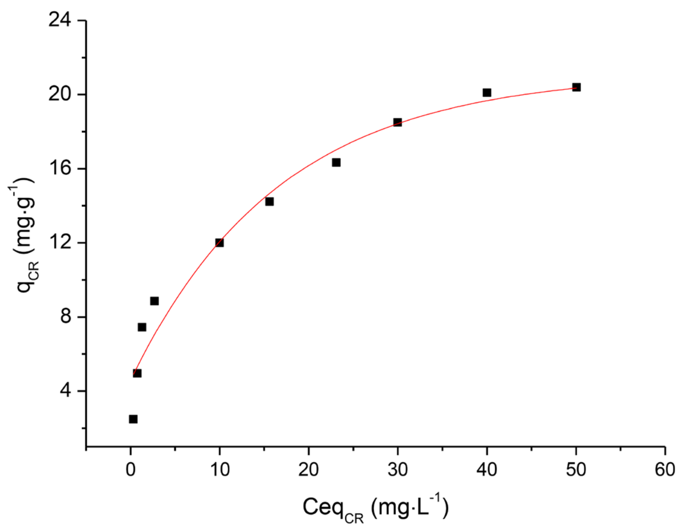

Figure 7. Adsorption isotherm for Congo Red in the presence of vermicompost.

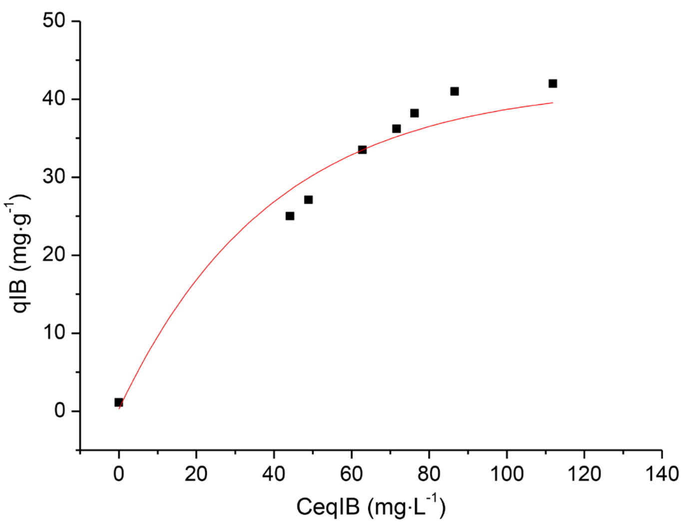

Figure 8. Adsorption isotherm for Indigo Blue in the presence of vermicompost.

the Langmuir Model [27], which is one the most used models to evaluate the adsorption of pollutants from aqueous media. Langmuir Model considers that adsorption occurs in monolayers, and all adsorption sites have the same energy. Equation (3) is the general equation in which the Langmuir model is based on:

$$q = K_L \cdot MAC \cdot C_{eq} \cdot \left(1 + K_L \cdot C_{eq}\right)^{-1} \quad (3)$$

where: q and C_{eq} have the definitions already given, K_L is the Langmuir constant ($L \cdot g^{-1}$), which is related to the adsorption energy, and MAC means maximum adsorption capacity of vermicompost in relation to Congo Red and Indigo Blue, as previously defined. The MAC unit is $mg \cdot g^{-1}$ (mg of adsorbed dye per gram of vermicompost).

As can be noted, both adsorption isotherms were not linear (**Figure 7** and **Figure 8**), thus presenting a similarity in relation to the results derived from other studies regarding the use of natural adsorbents and synthetic dyes [4] [6] [10] [24] [25].

Maximum adsorption capacity (MAC) and Langmuir constant (K_L) can be estimated after linearizing Equation (3) [plotting the ration C_{eq}/q ($g \cdot L^{-1}$) against C_{eq} ($mg \cdot L^{-1}$)], thus resulting in linearized isotherms (**Figure 9** and **Figure 10**) described by Equation (4). In this sense, after analyzing Equation (4), MAC could be defined as the inverse of the angular coefficient. In turn, K_L could be estimated as 1 divided by product between MAC and linear coefficient.

$$C_{eq}/q = \left(K_L \cdot MAC\right)^{-1} + \left(MAC\right)^{-1} \cdot C_{eq} \quad (4)$$

When Equation (4) was specifically applied to the data that generated adsorption isotherms showed in **Figure 7** and **Figure 8**, it was possible to obtain Equations (5) and (6), respectively. Equations (5) and (6) represent the lines indicated in **Figure 9** and **Figure 10**, respectively.

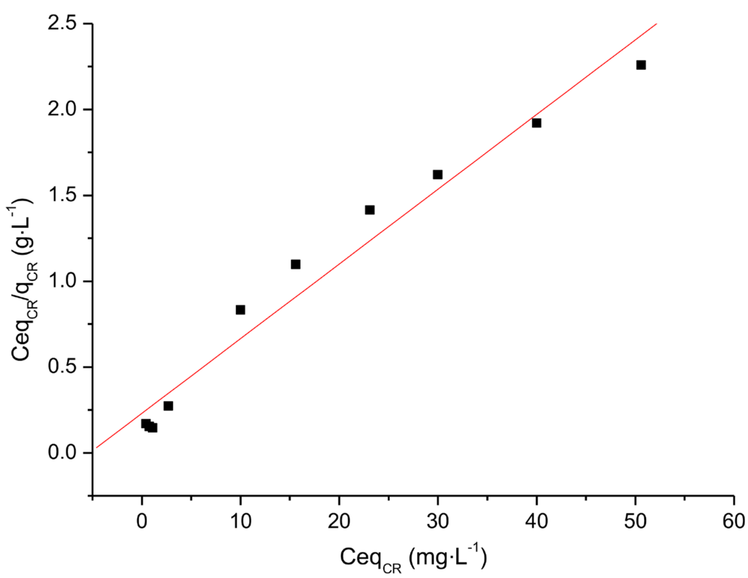

Figure 9. Adsorption isotherm linearization for Congo Red in the presence of vermicompost.

$$C_{eq}/q = 0.229 + 0.0430 \cdot C_{eq} \tag{5}$$

$$C_{eq}/q = 0.1610 + 0.0248 \cdot C_{eq} \tag{6}$$

From the K_L values, it was possible to obtain the ΔG_{ads} concerning the adsorption processes through Equation (7):

$$\Delta G_{ads} = -RT \ln K_L \tag{7}$$

where ΔG_{ads} is the variations of Gibbs free energy related to the adsorption processes, R is the universal gas constant (8.314472 J·mol^{-1}·K^{-1}), T is the temperature in Kelvin (298.15 K), and K_L is a constant related to the adsorption or binding energy (L·mol^{-1}), between vermicompost and dyes. The values of K_L were originally calculated in L·g^{-1}, but these values were subsequently converted to L·mol^{-1}. Table 1 lists all thermodynamic parameters concerning the adsorption of Congo Red and Indigo Blue on vermicompost. As can be seen in Table 1, the R^2 (Determination Coefficient) values for Equations (5) and (6) highlighted good adjustments of the Langmuir Model in relation to the evaluated adsorption results. The values of ΔG_{ads} indicate the nature of adsorption. Thus, $\Delta G_{ads} < -200$ kJ·mol^{-1} reveals chemical adsorption with low reversibility degree [28]. In this context, all adsorptions that were evaluated in this study could be classified as

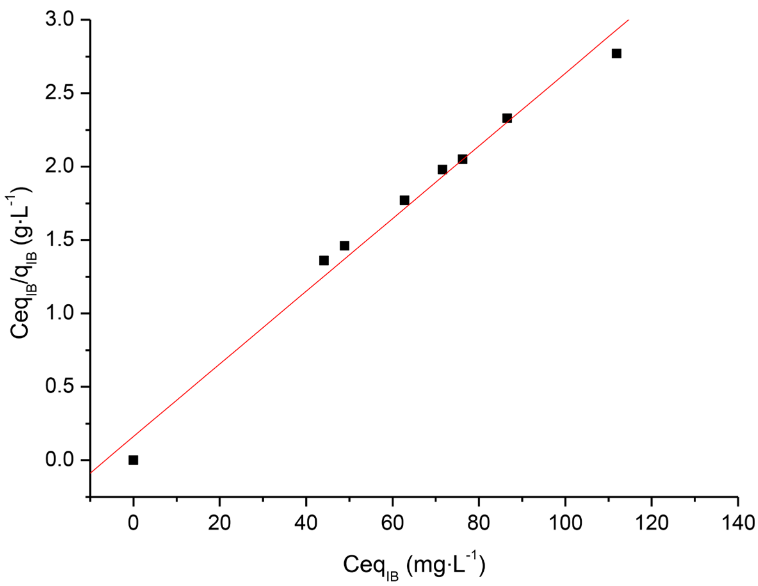

Figure 10. Adsorption isotherm linearization for IB in the presence of vermicompost.

Table 1. Physicochemical parameters concerning the adsorption of Congo Red and Indigo Blue on vermicompost.

Physicochemical parameters	Congo Red	Indigo Blue
R^2	0.9841	0.9919
MAC (mg·g^{-1})	23.25	40.39
K_L (L·mg^{-1})	0.19	0.15
ΔG (KJ·mol^{-1})	−29.24	−26.22

physical adsorption, in which it is observed weaker interactions between dyes and vermicompost. Therefore, it can be said that the chemical interactions shown in **Figure 4** are not dominant, occurring discreetly in a much lesser degree than before mentioned.

These discrete values of ΔG_{ads} are consistent with the structures of the evaluated dyes (Congo Red and Indigo Blue), which have bulky molecular structures with high electronic density. Both features are responsible for steric impediments and electrostatic repulsions, thus avoiding effective accommodation of the dyes on vermicompost particles. Based on the K_L values, which are related to the adsorption energy, it was possible to identify similar affinities of both dyes in relation to the adsorption sites. Ultimately, this outcome reveals that both molecular structures, although remarkably different, presented a set of common characteristics capable of decreasing adsorption strength. Despite low adsorption strength, which are related to the relatively small K_L values, reasonable values of *MAC* were achieved for both dyes, mainly for Indigo Blue. This finding means that despite the low adsorption energy, appreciable amounts of Congo Red and Indigo Blue could be retained per gram of vermicompost. The comparison between the *MAC* values obtained in this work with the values shown in **Table 2** reveals that the adsorption of CR and IB by natural adsorbents is many varied [4] [6] [17] [24] [25] [29]-[35]. For CR, values vary between 0.52 $mg \cdot g^{-1}$ [25] and 751 $mg \cdot g^{-1}$ [29]. While for IB all values, except one [4], have *MAC* around 50 $mg \cdot g^{-1}$ [17] [24] [30]. Making a comparative critical analysis, the values revealed in this work are satisfactory. In addition, the vermicompost used in this work is totally natural. No chemical modifications were made to it.

Table 2. *MAC* values for adsorption of CR and IB by different adsorbents.

Adsorbent	Pollutant	*MAC* values ($mg \cdot g^{-1}$)	References
Liquorice root powder	Congo Red	0.77	[4]
Liquorice root powder	Indigo Blue	1.70	[4]
Sugarcane bagasse	Congo Red	4.43	[6]
Sodium carboxymethyl cellulose and chitosan	Indigo Blue	50.00	[17]
Zizyphusjoazeiro peel	Indigo Blue	50.00	[24]
Wood sawdust powder	Congo Red	0.52	[25]
Corncob-to-xylose residue (CCXR)	Congo Red	751.00	[29]
Natural clay	Indigo Blue	57.00	[30]
Activated carbon	Indigo Blue	53.00	[30]
Algerian kaolin	Congo Red	6.53	[31]
Natural zeolites modified with N,N-dimethyl dehydroabietylamine oxide	Congo Red	69.94	[32]
Coconut residual fiber	Congo Red	128.94	[33]
Red mud	Congo Red	4.05	[34]
Polysaccharide of Carica papaya seeds	Congo Red	319.08	[35]

The dimensionless constant (R_L) is another thermodynamic parameter to favoring the adsorption process. In this case, $0 < R_L \leq 1$ indicates favorable adsorptions, while $R_L > 1$ can be attributed to unfavorable adsorptions (Esmaeili and Beni 2015). R_L is calculated by means of Equation (8), where MAC is the maximum adsorption capacity and C_0 is the highest dye concentration (500 and 4000 mg·L^{-1} for Congo Red and Indigo Blue, respectively) used for building adsorption isotherm.

$$R_L = 1/\left(1 + MAC \cdot C_0\right) \tag{8}$$

After substituting MAC and C_0 values in Equation (8), it was possible to achieve the following values of R_L: 0.000086 and 0.0000062 for Congo Red and Indigo Blue, respectively. In this sense, adsorptions of Congo Red and Indigo Blue were favorable.

4. Conclusion

The surface and chemical groups analysis, besides tests of adsorbent mass, stirring time, determination of physicochemical parameters, and MAC values reveal that the vermicompost is an efficient remover of CR and IB dyes from the aqueous medium. Studies using other dyes can transform this material into a filter component for cleaning industrial effluents.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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