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Vermicompost for Tinted Organic Cationic Dyes Retention

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Abstract The use of vermicompost was expanded as natural adsorbent for cationic dyes retention. The adsorption profiles in batch and flow modes for crystal violet and methylene blue on vermicompost material were evaluated. In batch mode, a retention index higher than 97% was obtained for both compounds, while in flow condition, 40 g of dried adsorbent material were enough to remove 100 mg of crystal violet or methylene blue at column flow rates of 5 and 20 mL min^{-1}. Adsorption isotherms showed adsorptive maximum capacities for vermicompost of 0.78 and 5.47 mg g^{-1}, respectively, which were compatible with the literature. Due to this good efficiency capacity, incineration steps can be considered as acceptable disposal procedures for enriched vermicompost. From these characteristics, economical and environmental advantages of the proposed material for the evaluated cationic dyes retention were evidenced.

Keywords Vermicompost · Cationic dyes · Adsorption · Natural adsorbent

1 Introduction

Nowadays, numerous chemicals are daily used in domestic, agricultural, laboratorial, and industrial activities, generating aqueous residues composed by toxic metallic ions, biodegradable molecules, agrochemicals, surfactants, pharmaceuticals, and tinted dye compounds among other substances (Holt 2000). For aquatic ecosystems, colored species are mainly dangerous because they lead to biochemical disorders (Pearce et al. 2003). Crystal violet and methylene blue (Fig. 1) are two tinted organic cationic dyes extensively used in microbiological laboratories and pharmaceutical industries (Dart 1996).

The literature reports some alternatives related to organic compounds degradation and/or removal, including electrochemical procedures (Chatzisymeon et al. 2006), chemical oxidation (Weber and LeBoeuf 1999), biological removal (Manahan 1994), advanced oxidative process (Mason 1990), and adsorption (Li et al. 2006). Special features have been attributed to the natural adsorbents for water treatment (Otero et al. 2003) due to their high efficiency, low cost, and easy acquisition. Additionally, adsorption systems are easily handled.

Numerous natural materials have good adsorption efficiency and they are used to remove ionic species dissolved in aqueous media. Bark/tannin-rich substrates, lignin, dead biomass, humified materials, zeolite, clay, and fly ash are natural materials successfully employed for ionic pollutant removal (Bailey et al. 1999).

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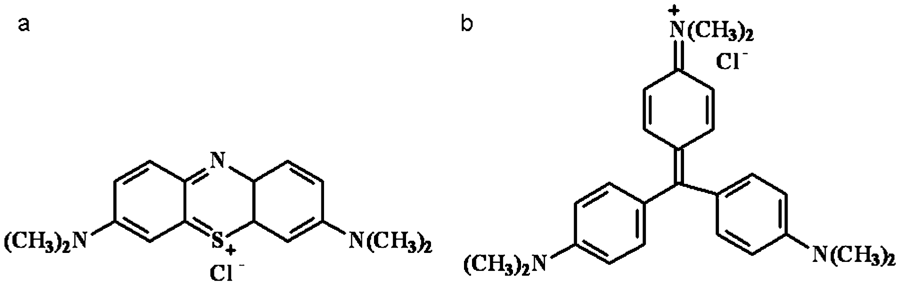

Fig. 1 Molecular structures of methylene blue (**a**) and crystal violet (**b**)

Fulvic and humic acids and humines are produced by the conversion of known biomolecules in very complex structures containing aromatic rings as well as hydrophilic portions composed by alcoholic, phenolic, carboxyl, and thiol groups. Humic materials are especially exciting due to their high surface area and porosity, great number of negative charges, and expressive cationic exchange capacity (CEC; Tuomela 2000). Vermicompost is one of the most important humified materials. It is obtained by earthworm excretion from metabolism of mixtures of soil and organic matter. In the digestive system of worms, microorganisms accelerate the humification leading to the production of a highly stable product named vermicompost. It has remarkable CEC, high water content, numerous mineral structures, and wide particle size distribution (Pereira and Arruda 2003).

As a result of their peculiar characteristics, vermicompost is extensively reported as an efficient adsorbent for metallic ions (Pereira and Arruda 2003, 2004; Jordão et al. 2007, 2002; Matos and Arruda 2003). Vermicompost has also been evaluated to diminish the availability of pesticide residues in soils, decreasing the potential risk of leaching to the groundwater (Fernández-Bayo et al. 2008). However, vermicompost is rarely used for retaining organic pollutants in aqueous medium as well as in liquid effluents. Thus, the present work aims to investigate the potentialities of the vermicompost to adsorb colored organic dyes.

2 Experimental

2.1 Equipment and Apparatus

The evaluation of adsorption vermicompost capacity in flow condition was carried out by inserting the natural material into a borosilicate column (φ=5 cm; h=20 cm) provided with Teflon tap and a Gilson Minipuls 3 peristaltic pump (France) was employed for fluid propulsion at a constant flow rate. Absorbance measurements were carried out in batch mode by using a Femto 700 Plus UV/vis spectrophotometer (Brazil) equipped with 10-mm optical path cuvette.

Physical and chemical information about vermicompost structure was obtained by employing a ThermoQuest Finnigan EA Flash 1112 elemental analyzer (Italy), a Shimadzu XRD-6000 X-ray diffractometer (Japan), a TA Instruments Universal V2.3C thermogravimetric analyzer (USA), a Hartmann & Braun-Michelson Bomem MB series infrared spectrometer (Germany), and a Jeol JMS T300 electron microscope (Japan).

The vermicompost enriched with dyes was incinerated by means of a Quimis D21 muffle furnace (Brazil) and all pH measurements were performed by a Digimed DM31 potentiometer (Brazil).

2.2 Reactants and Solutions

Solutions were prepared by using fresh highly pure water (18.2 MΩ cm^{-1}) obtained from Barnstead EASYpure-RF (USA). Methylene blue reference solutions were prepared by weighting from powdered chloride salt (Merck, Germany). Crystal violet stock solution (10,000 mg/L) was prepared by diluting a pharmaceutical formulation (Farmax, Brazil) and the working solutions were obtained by proper dilutions of the stock solution. Vermicompost was acquired in floricultures at Salvador City, Brazil.

2.3 Procedures

Except for the water content determinations, which were performed with fresh vermicompost samples, all analyses were carried out by employing vermicompost sieved at 2 mm and dried at 60°C for 24 h.

2.3.1 Characterization of the Vermicompost

The procedures described in this section were adopted or modified from the literature (Pace et al. 1982).

For pH determination, 10.0 g of the vermicompost were shaken with 25 mL of a 1.0-mol L^{-1} KCl solution for 1 min. The samples were then allowed to rest for 1.0 h. After that, samples were shaken for 1.0 min and the pH of the resulting suspension was potentiometrically determined.

The water content was gravimetrically determined after heating 10.0 g of fresh vermicompost at 60°C for 24 h. Total organic matter and ash contents were also quantified by gravimetric procedures after heating 1.0 g of the sample at 550°C and 800°C, respectively, in a muffle furnace for 3 h.

The organic functional groups present in the vermicompost were characterized by Fourier transform IR spectroscopy using KBr discs to prepare the samples. The spectral range varied from 4,000 to 400 cm^{-1}. The thermogravimetric analyses were performed by means of vermicompost heating (in an oxidant atmosphere) from 25°C to 1,000°C at 10°C/min.

For X-ray diffraction analysis, the powdered samples were exposed to X-rays (λ=1.54060 Å) with 2θ angle varying within 5° and 50°. The applied voltage and current were 40 kV and 30 mA, respectively. For electron microscopy analyses, vermicompost particles were covered with a thin layer of gold and an electron acceleration voltage of 20 kV was applied. After introducing small masses of vermicompost into tin capsules, elemental analyses were carried out according to the manufacturer's specifications. Organic compounds were used for calibrating the measurements.

2.3.2 Evaluation of the Crystal Violet and Methylene Blue Adsorptive Profiles at Batch Mode

In order to evaluate the vermicompost mass necessary to retain 1 mg of methylene blue or crystal violet, a set of experiments was carried out using the batch mode. Thus, adsorbent masses (from 0.3 to 9.6 g) were vigorously stirred with 50 mL of crystal violet or methylene blue solutions at 20 mg L^{-1} for 30 min and centrifuged for 10 min at 5,000 rpm.

Kinetic aspects related to dyes adsorption were investigated. For this purpose, a vermicompost mass was fixed and the agitation time varied from 1 to 30 min. Again, centrifugation was adopted after each agitation period. In all situations, supernatant absorbances were measured at 580 and 640 nm for crystal violet and methylene blue, respectively.

No pH adjustments were performed. The natural pH of the crystal violet and methylene blue solutions (at 20 mg L^{-1}) were 5.2 and 5.0, respectively.

2.3.3 Adsorption Isotherms

Adsorption isotherms were built for estimating the vermicompost maximum adsorptive capacities. For this purpose, 50 mL of crystal violet or methylene blue solutions (from 20 to 100 mg L^{-1}) were stirred with vermicompost in the conditions previously established for mass and agitation time. The experiments' temperature was 20±1°C. No pH adjustments were carried out.

2.3.4 Crystal Violet and Methylene Blue Removal at Flow Mode

After evaluating the adsorptive profiles of two tinted organic dyes and the maximum adsorptive capacities of the vermicompost, successive aliquots of 50 mL containing methylene blue or crystal violet at 20 mg L^{-1} were percolated through a column filled with 40 g of vermicompost (dried and sieved). A peristaltic pump was coupled to the column tap in order to maintain the flow rate at 5 or 20 mL min^{-1}.

2.3.5 Vermicompost Incineration

After the adsorption experiments of cationic dyes in the vermicompost, an enriched material was allowed and the vermicompost enriched with both dyes was incinerated at 550°C for 4 h by using muffle furnace and porcelain crucibles.

3 Results and Discussion

From the physical and analytical points of view, vermicompost materials are not homogeneous. The composition of a vermicompost depends on the worms, soil, and organic matter used in its formulation. Nevertheless, independent of the vermicompost source, critical properties for an effective adsorption are always expected for this natural material. In order

Table 1 Physical and chemical characteristics of diverse vermicompost samples

Sample origin	Parameters (%, m/m)						Reference
	C	N	H	Humidity	TOM	Ashes	
Salvador/BA	17.2±1.1	1.8±0.1	2.9±0.2	54.7±1.1	37.8±1.8	61.3±3.5	This work
Coimbra/MG	17.1±0.1	2.0±0.1	3.0±0.1	59.8±0.3	41.2	53.7	Pereira and Arruda (2003)
Coimbra/MG	33.2±1.0	2.3±0.1	5.0±0.2	56.2±0.1	53.5	41.1	Pereira and Arruda (2003)
Coimbra/MG	10.4±0.2	0.7±0.1	1.8±0.1	57.9±1.3	27.0	68.8	Pereira and Arruda (2003)
S. Carlos/SP	NI	NI	NI	NI	31.5	NI	Rocha et al. (1998)

BA Bahia state, *MG* Minas Gerais state, *SP* São Paulo state, *NI* not informed

to confirm this assumption, Table 1 shows a comparison between vermicompost collected in Salvador City and in other Brazilian regions.

As it can be observed, most of the available parameters concerning with the evaluated vermicompost sample are in agreement with the literature cited in the Table 1. The expressive ash contents can be associated with Brazilian soils, which presents large chemical wearing and high enrichments of tectosilicates (Lindsay 1979). On the other hand, high concentrations of total organic matter are related to the raw matter source, including bovine and pig manure. Additionally, the great number of hydrophilic groups contained in the humified material can be related to the expressive humidity levels observed in all vermicompost samples (Stevenson 1982) and carbon, hydrogen, and nitrogen contents also agree with the literature.

The infrared spectrum, thermogravimetric graph, and X-ray diffractogram of the vermicompost collected in Salvador are presented in Fig. 2. Spectroscopic information was similar to those of different vermicompost samples (Pereira and Arruda 2003; Jordão et al. 2002). The broad bands observed between 3,500 and 3,000 cm^{-1} can be ascribed to the stretching vibration of N–H bonds from amines and amides as well as alcoholic and phenolic hydroxyls and carboxylic acids. At the range of 3,000 to 2,800 cm^{-1}, the absorption is mainly assigned to the C–H bond from aliphatic groups. Peaks placed between 1,870 and 1,650 cm^{-1} can be ascribed to the C=O bond stretching of carbonyl compounds. Absorptions near 1,500 cm^{-1} correspond to the C=C bond of aromatic structures. The bands between 1,100 and 1,000 cm^{-1} are characteristic of the C–O bond stretching in polysaccharide or polysaccharide-like substances or even Si–O bonds of silicates present in vermicompost structure (Lambert et al. 1998).

According to thermogravimetric analysis, organic matter volatilization occurs from 100 to 550°C. The stabilization plateau observed in temperatures higher than 550°C was attributed to aluminosilicates with remarkable thermal stability. X-ray diffractogram has shown an intense peak at 26.8°, which was related to the distance among the atomic quartz planes, since it is usually present in Brazilian soils. The smaller peaks also correspond to this mineral.

Electron micrographs (Fig. 3) pointed out an irregular morphology as well as a higher porosity. These characteristics corroborate to elevated CEC, since it increases the particle surface areas (Landgraf et al. 1998). Notwithstanding, the morphology of the studied vermicompost was similar to those from Minas Gerais and São Paulo states, Brazil (Pereira and Arruda 2003).

Finally, as some of the physical–chemical characteristics of the studied vermicompost showed similar structural properties to those observed for other vermicomposts, similar adsorptive properties are expected for these materials, confirming the expectation considered at the beginning of this section.

As previously commented, all adsorption experiments were conducted without pH adjustments. The usual pH of the tinted dye solutions (5.2 for crystal violet and 5.0 for methylene blue) was maintained, since the vermicompost shows a very extensive number of negative sites at these pH values where it is able to retain cations (Jordão et al. 2002). Additionally, pH variations can provoke changes in the color solutions as well as in the color intensities. For a pH smaller than 5.2 and higher than this value, crystal violet molecules are discolored because the number of conjugated multiple bonds is decreased. A similar phenomenon also occurs with methylene blue (Solomons and Fryhle 1998).

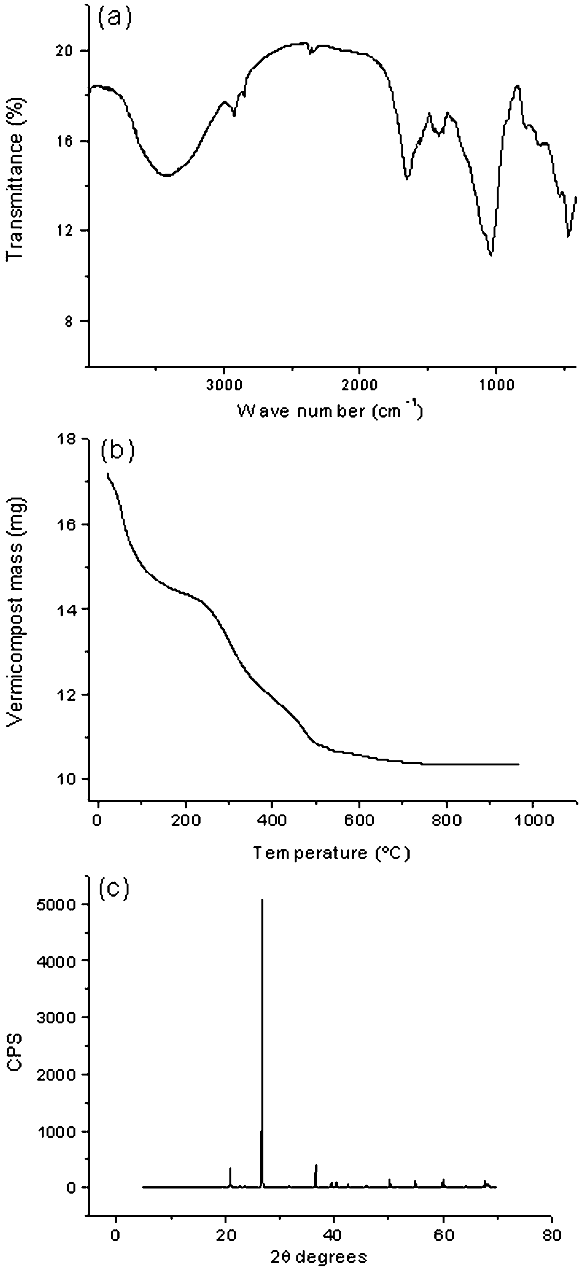

Fig. 2 Infrared spectrum (**a**), thermogravimetric graph (**b**), and X-ray diffractogram (**c**) of the vermicompost

Besides all concerns described above, the employment of dye alkaline solutions exhibit an additional inadequacy regarding the vermicompost stability. This problem occurs because humic acid, an important structural component of the vermicompost, is solubilized at pH values greater than 8 (Jordão et al. 2002).

Figure 4 illustrates the influence of the vermicompost mass and agitation time on dyes retention. Crystal violet was efficiently retained on vermicompost (99.9±0.07%) when 4.8 g of the referred adsorbent was employed. Moreover, a vermicompost mass of 0.6 g was necessary to retain 99.0±0.1% of methylene blue. This expressive difference in the vermicompost mass can be attributed to steric hindrance, since molecules of methylene blue are smaller than those of crystal violet.

In this study, the absorbance drop can be strongly related to the adsorption in vermicompost, since crystal violet and methylene blue molecules do not react in the aqueous phase. This consideration is true because the small vermicompost masses adopted in this work were not enough to enrich the supernatants with reactive chemical species, including those responsible for acidity increase of the medium.

A quantitative retention of crystal violet on vermicompost (100.0±0.3%) was attained after 10 min, thus indicating a fast equilibrium among solid and liquid

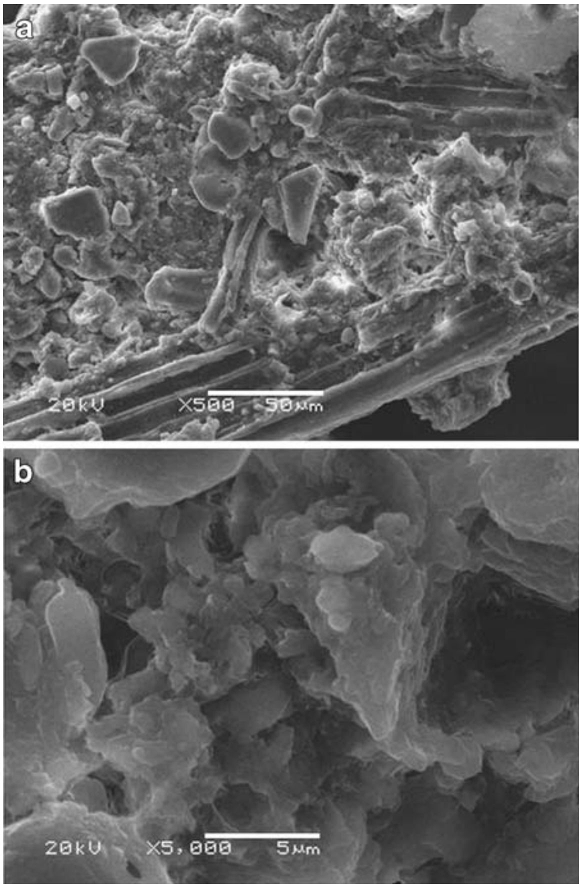

Fig. 3 Electronic micrographs of the vermicompost at magnifications of ×500 (**a**) and ×5,000 (**b**)

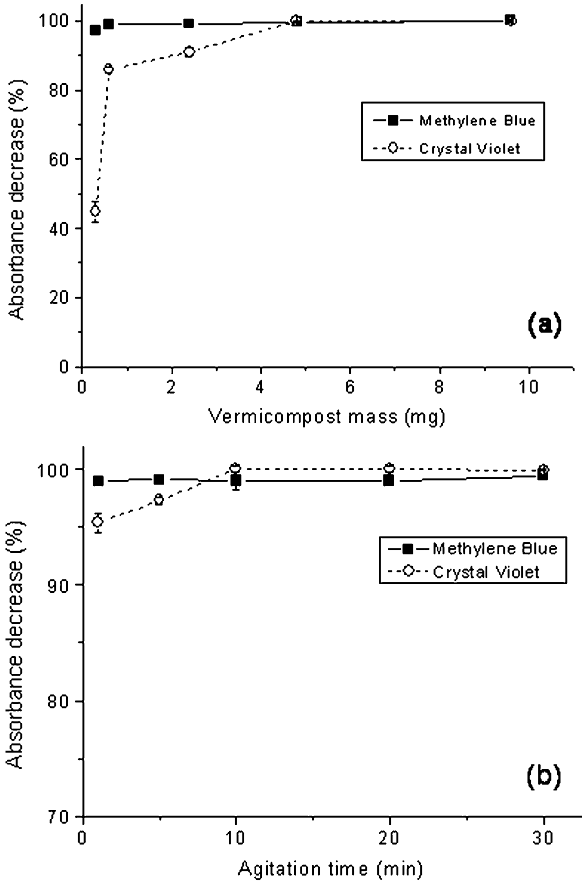

Fig. 4 Vermicompost mass (**a**) and agitation time (**b**) influences on the dyes retention (n=3)

phases. In the case of methylene blue, it was necessary for only 1 min to ensure a retention of 99.9±0.2%. These results reinforce the previous discussions about steric hindrance.

Adsorption isotherms (Fig. 5) were built by using vermicompost masses and agitation times of, respectively, 0.6 g and 1 min for methylene blue and 4.8 g and 10 min for crystal violet. As previously discussed, these conditions were enough to warrant absorbance reductions higher than 99%.

The adsorption isotherms have shown evident tendencies toward saturation. This kind of isotherm follows the mathematical model described by Eq. 1 (Sag and Kutsal 2000):

$$\mathrm{qM} = a \times b \times \mathrm{CeqM} \times (1 + a \times \mathrm{CeqM})^{-1} \qquad (1)$$

where qM is the quantity of adsorbed dye in vermicompost (mg g^{-1}), a is a constant related to adsorption energy (L mg^{-1}), b is the maximum dye adsorption capacity of the vermicompost (mg g^{-1}), and CeqM is the equilibrium dye concentration (mg L^{-1}).

Equation 2 is obtained by linearizing Eq. 1, which permits exact calculations of b values:

$$\mathrm{CeqM}\Big/\mathrm{qM} = (a \times b)^{-1} + (b)^{-1} \times \mathrm{CeqM}. \qquad (2)$$

The lines indicated in Fig. 6 are obtained by applying Eq. 2 on the adsorption isotherms. From these lines, it was possible to calculate the maximum adsorption capacities (b^{-1}) for crystal violet (0.78 mg g^{-1}) and methylene blue (5.47 mg g^{-1}).

Different natural adsorbents used for retaining crystal violet and methylene blue are listed in Table 2 and, as it can be seen, the maximum adsorptive capacities are similar to those observed for vermicompost. When compared with natural materials, some synthetic substrates exhibit better adsorption capacities for both tinted cationic dyes (Wang et al. 2006),

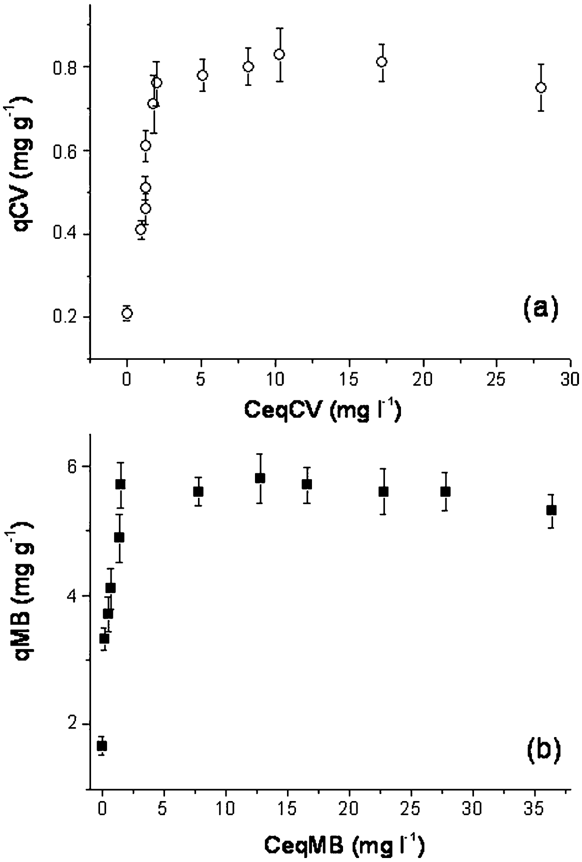

Fig. 5 Adsorption isotherm for crystal violet (**a**) and methylene blue (**b**) (n=3)

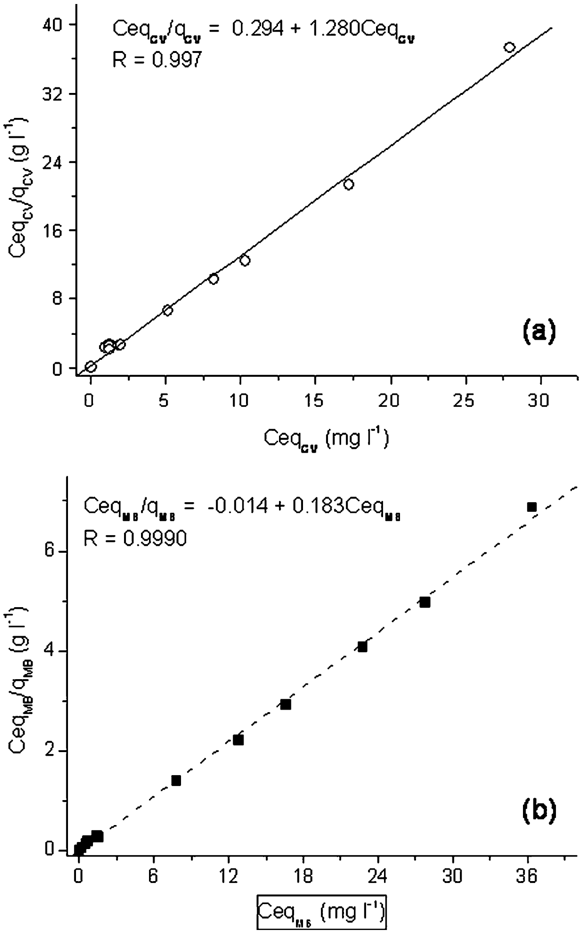

Fig. 6 Linearization of the adsorption isotherm for crystal violet (**a**) and methylene blue (**b**)

but they show limitations related to cost and/or acquisition difficulties.

After 100 aliquots or 5 L of both tinted cationic dyes went through vermicompost columns, all of them exhibited absorbance reductions of 100% for both flow rates (5 and 20 mL min^{-1}). The treated volume of 5.0 L was considered enough to demonstrate the vermicompost potentiality and, for this reason, the column operations were stopped in aliquot 100.

The vermicompost mass was fixed to 40 g because this mass permitted, according to the used column, a good particle accommodation and a large contact area among vermicompost and solutions. Obviously, this mass can be changed according to specific necessities, including the volume and concentration of the dye solutions as well as column geometry.

The flow rates of 5 and 20 mL min^{-1} were chosen to evaluate the adsorption efficiency at different contact times of vermicompost particles and dye molecules. Flow rates smaller than 5 mL min^{-1} were not tested because the column system operation would be very slow. On the other hand, column clogging was observed at flow rates higher than 20 mL min^{-1}.

Five liters of tinted solution were treated 16.7 and 4.2 h for flow rates of 5 and 20 mL min^{-1}, respectively. The period of 16.7 h is considered as reasonable for academic laboratories where small residual volumes of crystal violet and methylene blue solutions are generated. On the other hand, at 20 mL min^{-1}, the treatment time is appropriated for laboratories with higher generation of tinted wastewaters. In this category, those concerning microbiological activities can be mentioned.

New fillings of the vermicompost column would result in small differences related to particle accommodations and, thus, to adsorption percentages. This behavior was previously observed (Pereira and Arruda 2003) when vermicompost was employed in a column system for a prior concentration of cadmium

Table 2 Retention capacities of different natural adsorbents for crystal violet and blue methylene

Adsorbent	Maximum adsorption capacities (mg/g)		Reference
	Crystal violet	Methylene blue	
Vermicompost	0.78	5.47	This work
Pumice powder	0.3	0.4	Akbal (2005)
Pyrolyzed petrified sediment	NI	1.6	Arogus et al. (2008)
Fly ash	2.2	1.4	Li et al. (2006)
Yellow passion fruit peel	NI	2.2	Pavan et al. (2008)
Chrome sludge	NI	0.5	Lee et al. (1996)

NI not informed

ions from mineral water samples. In that manuscript, the authors observed discrete differences in cadmium adsorption (6.5%) when the same column was filled with three identical portions of vermicompost.

After new column assemblies, great differences in vermicompost performance are not expected because this natural adsorbent has numerous negative charges, which are able to compensate possible disparities in particle accommodation. This compensation effect is very pronounced for expressive vermicompost masses, as the one employed in this work.

Besides all desirable adsorptive properties of the vermicompost previously discussed, it was not necessary to adopt sophisticated preparation steps in order to use this adsorbent. This characteristic offers economic viability and high versatility for the proposed column systems.

Some disposal alternatives could be applied for enriched vermicompost such as leaching and incineration. The main goal of a leaching process is to recover the adsorbed species. However, the natural occurrence of a great diversity of organic and inorganic species leached from vermicompost can be considered as a drawback for dyes reuse. Therefore, since quartz has great chemical inertia, incineration is a safe procedure for disposing the vermicompost enriched with tinted dyes. In spite of CO_2 evolution, this inconvenience would not be frequent due to the high vermicompost adsorptive performance.

It is important to note that, all treatment procedures and not only the one developed in this work, exhibit some environmental liabilities after their operations. For example, advanced oxidative processes (AOPs) present excellent performance concerning organic pollutants mineralization, but AOPs also release CO_2 and nutrients (as NO_3^- and PO_4^{3-}) and, thus, provoke undesirable effects in atmosphere and aquatic ecosystems.

In this work, single aqueous solutions containing crystal violet or methylene blue were employed to evaluate vermicompost potentiality. This procedure must be considered as satisfactory, since many anthropogenic activities generate aqueous residues containing only one of the cited dyes. For example, microbiological laboratories use and discharge large quantities of single crystal violet solutions (Dart 1996). Likewise, single methylene blue solutions are prepared and used in diverse practices carried out in universities, including spectrophotometric measurements and chromatographic separations. In addition, methylene blue is employed in practices of biochemistry and in pharmaceutical production. Thus, single aqueous methylene blue solutions also comprise dangerous effluents for aquatic ecosystems.

4 Conclusions

The column systems proposed in the present work can be considered efficient, versatile, and economically feasible for small treatment plants. They can be operated at flow rates compatible (5 and 20 mL min^{-1}) with the necessities of academic, microbiological, and pharmaceutical laboratories.

The adsorptive performance of the vermicompost for crystal violet and methylene blue were comparable with other natural adsorbents reported in the literature. The column system operation generates small masses of residues, which can be safely disposed by incineration steps. Finally, the present work increased the number of natural adsorbents used for removing organic pollutants from aqueous media.

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