



Short communication

Physicochemical study of a bio-based adsorbent made from grape marc



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ABSTRACT

In the last few years bio-oxidized grape marc (formulated by encapsulation in calcium alginate beads) has been proposed as an eco-friendly adsorbent for wastewater treatment. However, the physicochemical properties of the bioadsorbent have not yet been reported. This study analyzed the stability of the bio-based adsorbent before and after adsorption of dye compounds from winery wastewater. Minor morphological changes in the adsorbent were observed after it was used to treat the wastewater. The physicochemical properties of the bioadsorbent at equilibrium were also evaluated. The process of adsorption onto the heterogeneous surface of the bio-based adsorbent was best described by the Freundlich isotherm, and the adsorption process was physically controlled ($E = 0.61$ kJ/mol).

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1. Introduction

Increasing importance is being given to minimizing the residues generated by all industrial sectors, in order to increase the sustainability of production processes and render them less harmful to the environment. The viticulture and winery industry, which generates large amounts of agricultural residues, is playing an important role in this respect (Vázquez-Rowe et al., 2012). Traditionally, woody vineyard residues were incinerated directly in the field or used as biomass for energy production, thus generating greenhouse gases that contribute to global warming. However, wineries must now comply with legislation for waste minimization and revaluation or pay the financial penalties for non compliance (Devesa-Rey et al., 2011).

The utilization of lignocellulosic residues that can be spontaneously oxidized by their own microbial population to produce eco-friendly adsorbents is a promising option that has been considered in previous studies (Perez-Ameneiro et al., 2014). After grape marc undergoes spontaneous biodegradation, the initial composition (56.7% lignin, 18.2% cellulose and 8.0% hemicellulose) is altered, yielding a polymeric matrix containing lower proportions of cellulose and hemicellulose (9.3% and 2.6%, respectively) and a higher proportion of lignin (66.3%, Moldes et al., 2007).

Most studies considering the formulation of bioadsorbents focus on the kinetics of contaminant adsorption and do not consider the morphological changes that occur during the bioadsorption process (Varshini et al., 2015; Ofomaja et al., 2015). However, the physicochemical surface properties of cellulosic and lignocellulosic materials are of great importance in the context of composite production, in papermaking and in the textile sector (Gamelas, 2013). Better knowledge of the morphology and structure would be helpful for understanding the behavior of bioadsorbent materials and any possible limitations to industrial application.

In this study, it was investigated the performance and morphological structure of a promising eco-friendly adsorbent for the treatment of effluents from the winery industry. The bioadsorbent was formulated by encapsulation of composted grape marc in calcium alginate beads. Physicochemical analysis of the bio-based adsorbent was also carried out by applying different equilibrium isotherm models.

2. Materials and methods

2.1. Grape marc

Grape marc was obtained from local winery industries and allowed to biodegrade spontaneously, following the procedure outlined in a previous study (Moldes et al., 2007).

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2.2. Formulation of the grape marc bioadsorbent

The bio-based adsorbent was formulated with 2% biodegraded grape marc, 2% sodium alginate (concentration in solution) and added to a crosslinking solution of 0.58 mol/L calcium chloride, as previously described (Perez-Ameneiro et al., 2014).

2.3. Adsorption experiments

Adsorption experiments were carried out with wastewater initially containing 19.83 mg/L of dye. The bioadsorbent and wastewater were added at a ratio of between 0.5:1 and 1.5:1 (v/v) to 250 mL Erlenmeyer flasks, which were then shaken at 112 rpm for 120 min at 25 °C. The amount of colored compounds in the wastewater at equilibrium was measured as equivalents of Amaranth dye, following a previously described method (Perez-Ameneiro et al., 2014).

2.4. Morphological analysis of grape marc-based bioadsorbent using high-resolution optical images

Quintuplicate measurements of size and morphology parameters of the cellulosic-based bioadsorbent were made with a stereoscopic microscopic (Nikon SMZ 1500) fitted with an objective lens HR Plan Apo 1×, eyepiece C-W 10×, F.N. 22, zoom range position 1×, a 12 V/100 W halogen illuminator and controlled by Image tool 3.00 software.

2.5. Isotherm study

The adsorption capacity of the bioadsorbent at equilibrium was calculated using Eq. (1).

$$q_e = \frac{(C_0 - C_e) \cdot V}{W} \quad (1)$$

where C_0 and C_e (mg/L) are the concentration of dye in the wastewater at the initial time and at equilibrium, respectively, V (L) is the volume of solution, and W (g) is the mass of adsorbent. Langmuir, Freundlich, Dubinin–Radushkevich and Temkin equations were used to study the adsorption process at equilibrium.

3. Results and discussion

3.1. Morphological characterization of grape marc bioadsorbent

Analysis of some physicochemical properties of the bioadsorbent under study is important to enable proper design of a filtering column and thus enable pilot or industrial scale application of the material. Wide variations in the size of the bio-based adsorbent or its decomposition after adsorption may result in the collapse of the fixed bed system. Morphological and surface measurements (including area, perimeter, major and minor axes, Feret diameter, elongation, roundness and compactness) of the bio-oxidized grape marc composite are shown in Table 1.

Elongation is the ratio of the length of the major axis to the length of the minor axis. The result is a value between 0 and 1 that can be calculated with Eq. (2). Roundness is a measure of the sharpness of a particle's corners, which can vary between 0 and 1, and is determined with Eq. (3). Compactness measures the circularity of an object based on the Feret diameter, which is the average diameter of a particle. These parameters are defined by Eqs. (4) and (5), respectively.

$$\text{Elongation} = \frac{\text{Major axis length}}{\text{Minor axis length}} \quad (2)$$

Table 1

Morphological parameters of the grape marc biopolymer before and after adsorption.

	Before adsorption	After adsorption
Area (mm ²)	7.66 ± 0.85	8.01 ± 0.96
Perimeter (mm)	11.02 ± 0.66	11.13 ± 0.60
Major axis length (mm)	3.33 ± 0.21	3.37 ± 0.23
Minor axis length (mm)	3.03 ± 0.17	3.13 ± 0.16
Elongation	1.10 ± 0.04	1.08 ± 0.03
Roundness	0.79 ± 0.03	0.81 ± 0.02
Feret diameter (mm)	3.12 ± 0.18	3.19 ± 0.19
Compactness	0.94 ± 0.01	0.95 ± 0.01

$$\text{Roundness} = \frac{4 \cdot \text{Area}}{\pi \cdot (\text{Major axis length})^2} \quad (3)$$

$$\text{Compactness} = \frac{\text{Feret diameter}}{\text{Major axis length}} \quad (4)$$

$$\text{Feret diameter} = \sqrt{\frac{4 \cdot \text{Area}}{\pi}} \quad (5)$$

The structure of the grape marc-based adsorbent was very stable after the adsorption process (Table 1). Compactness was close to 1, indicating that the adsorbent remains spherical. Among the parameters evaluated, area yielded the greatest differences between loaded and unloaded bioadsorbent (4% increase) and size increased slightly after adsorption. This was validated with the images shown in Fig. 1, which includes macro views of the bioadsorbent before and after the removal of dye compounds from winery wastewater.

3.2. Equilibrium study

The influence of the initial dose of adsorbent, W (g), on removal of colored compounds from winery wastewater was assessed. The results indicate that once equilibrium was reached, removal of colored compounds from wastewater increased with the initial dose of adsorbent. However, the adsorption capacity of the biopolymer decreased significantly as the initial dose increased, as expected from Eq. (1). A maximum adsorption capacity of 1.31 mg/g was reached. Moreover, the adsorption capacity was optimal, i.e. removal of dye compounds was maximal (86.20%), at a dose of 0.70 g of adsorbent. Similar findings were reported in a study analyzing the uptake of malachite green from aqueous solution by use of compost from kitchen waste as an adsorbent (Bhagavathi Pushpa et al., 2015).

Different isotherms were used to study the adsorption process at the equilibrium. The Langmuir isotherm is described by Eqs. (6) and (7) (Langmuir, 1918), the Freundlich isotherm by Eq. (8) (Freundlich, 1906), the Dubinin–Radushkevich isotherm by Eqs. (9) and (10) (Dubinin et al., 1947) and the Temkin isotherm by Eqs. (11) and (12) (Temkin and Pyzhev, 1940).

$$\frac{1}{q_e} = \frac{1}{q_m K_L C_e} + \frac{1}{q_m} \quad (6)$$

$$R_L = \frac{1}{1 + K_L C_0} \quad (7)$$

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (8)$$

$$\ln q_e = \ln q_m - \beta \left[RT \ln \left(\frac{1}{C_e} \right) \right]^2 \quad (9)$$

$$E = \sqrt{\frac{1}{2\beta}} \quad (10)$$

$$q_e = B_1 \ln K_T + B_1 \ln C_e \quad (11)$$

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