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Grapefruit peelings as a promising biosorbent for the removal of leather dyes and hexavalent chromium

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ABSTRACT

The objective of this study was to find a suitable and versatile agroforestry waste as alternative low-cost biosorbents for the removal of inorganic and organic compounds present in the wastewater of tannery industry [leather dyes and Cr(VI)]. Grapefruit peelings, fern, eucalyptus barks, oak leaves and cane pruning of grapevines were evaluated as biosorbents. Among them, grapefruit peelings showed the best results reaching 45 and 55% for the mixture of dyes and Cr(VI), respectively. A significant improvement in the performance of this biosorbent was obtained when it was pretreated with H_2O_2 (1 M) attaining the highest removal capacity of 80% and 100%, respectively. This biosorbent was characterised showing an adsorption capacity of 1.1003 meq/g and pH_{ZPC} 3.48. The adsorption working parameters, kinetics and isotherms were deeply studied in order to scale up the process to a continuous treatment system. Adsorption isotherms data fitted well to the Langmuir model with a maximum uptake of 37.427 mg/g for dyes mixture and 39.0628 mg/g for Cr(VI). Finally, the dynamic behaviour of the system, operating at different flow rates, was evaluated using a Homogeneous Surface Diffusion Model. Pretreated grapefruit peelings demonstrated to be a suitable low-cost biosorbent in the treatment of wastewater of tannery industry.

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Abbreviations: HSDM, Homogeneous Surface Diffusion Model; CP, cane pruning wine grapes; EL, eucalyptus barks; FR, fern; GP, grapefruit peelings; OL, oak leaves; DM, leather dyes mixture; C_i, initial pollutant concentration of solution (mg/L); C_e, concentration of pollutant at adsorption equilibrium (mg/L); C_t, concentration of pollutant at time t (mg/L); D, removal of DM or Cr(VI) (%); V, volume (mL); m_{ads}, mass of biosorbent (g); q_e, equilibrium adsorption capacity of biosorbent (mg/g); q_t, amount of pollutant adsorbed per unit of biosorbent at time t (mg/g); t, time (min); q_{max}, maximum pollutant uptake (mg/g); b_L, Langmuir model affinity between the adsorbate and biosorbent (L/mg); r^2, linear correlation coefficient; K_F, Freundlich model binding capacity ($mg^{1-(1/n_F)}\,L^{1/n_F}/g$); n_F, Freundlich model affinity between the biosorbent and adsorbate; K_{RP}, Redlich–Peterson model isotherm constant (L/g); a_{RP}, Redlich–Peterson model constant $(L/mg)^{\beta_{RP}}$; β_{RP}, Redlich–Peterson model exponent; K_S, Sips model isotherm constant ($L^{\beta_S}\,mg^{1-\beta_S}/g$); a_S, Sips model constant (L^{β_S}/mg^{β_S}); β_S, Sips model exponent; k_1, pseudo-first order rate constant (1/min); k_2, pseudo-second order rate constant [g/(mg min)]; k_{id}, intra-particle diffusion rate constant [$mg/(g\,min^{1/2})$]; I, intra-particle diffusion rate parameter; ε, porosity; ρ_p, particle density (g/mL); d_p, particle diameter (mm); Q, flow rate (L/min); h, column height; D_C, column diameter (m); V_C, column volume (L); K_L, film diffusion coefficient (L/mg); D_s, surface diffusion coefficient (m^2/s).

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1. Introduction

Industrial processes generate a wide range of wastewaters containing different pollutants that may involve either organic or inorganic reagents once used. Large amount of these pollutants arrive daily to both groundwater and surface water.

Tannery industries wastes present an important environmental impact on waters (Mwinyihija, 2010). Dyes and heavy metals such as chromium have been widely used in these industries (Rosales et al., 2012). Cr(VI) is a recognised carcinogenic and mutagenic agent and the presence at concentrations exceeding 0.05 mg/L for Cr(VI) (Acar and Malkoc, 2004) and 5.0 mg/L for Cr(III) (Acar and Malkoc, 2004; Pérez Marín et al., 2009) may be toxic for aquatic life. Dyes presence in water is undesirable due to the lasting colour, the increase in the chemical oxygen demand and the effect in the microorganisms present in the water streams.

Adsorption is one of the most commonly used techniques due to the easiness of design, operational simplicity and good efficiency in the removal of pollutants (Rosales et al., 2015; Crini, 2006). In the last years, the utilisation of wastes derived from agriculture or forestry has attracted the attention of the researchers due to their availability, biodegradability and inexpensive cost. Moreover, to improve the adsorption of organic and inorganic compounds, several pretreatments of the biosorbents have been proposed. Among the different pretreatments, several compounds such as NaOH, $CaCl_2$, organic acids (citric acid, oxalic acid), inorganic acids (HCl, H_2SO_4 and HNO_3) or H_2O_2 (Daffalla et al., 2012; Cobas et al., 2014; Shen et al., 2011) have been used. However, these biosorbents have been developed for the treatment of a specific target compound (organic or inorganic). According to the literature, different biosorbents have been proposed as alternative to more expensive and widely applied activated carbon. Thus, sawdust, husk rice, orange peelings, chestnut, hazelnut, peanut, cabbage, maize, *Colocasia esculenta* or grapefruit peelings have been proposed for metal removal (Rosales et al., 2015; Torab-Mostaedi et al., 2013; Hossain et al., 2014; Xu et al., 2014; Guyo et al., 2015; Pakshirajan et al., 2013) and peat, straw, sugarcane bagasse, orange or lemon peelings (Crini, 2006; Cobas et al., 2014; Bhatnagar et al., 2009; Gupta et al., 2009) for dye adsorption. However, there is a lack of biosorbents that can be efficiently applied for both inorganic and organic pollutants. Few studies have been developed in this field. Recently, a study developed in our laboratory demonstrated the feasibility of the biosorbent *Fucus vesiculosus* for the removal of both types of contaminants (Cobas et al., 2014).

The aim of this study is to find a versatile low-cost biosorbent for removing the typical pollutants present in the tannery effluents, Cr(VI) and mixture of commercial leather dyes. For that, several agroforestry wastes (grapefruit peelings, fern, eucalyptus barks, oak leaves and cane pruning of grapevines) were evaluated. The best biosorbent was studied in depth (kinetics and isotherms) in order to model and scale up its behaviour operating in a pilot scale column at different flow rates.

2. Material and methods

2.1. Reagents

Four leather dyes (Sella Solid Blue, Special Violet, Derma Burdeaux and Sella Solid Orange) were provided by a local tannery industry, Padronesa Industrial de Curtidos S.A. (Spain). The leather dyes mixture (DM), employed in all the experiments, was composed of a mixture of selected dyes in equal proportion. Cr(VI) as potassium dichromate was purchased from Panreac, and H_2O_2 (37%) was purchased from Merck. All the used chemicals were reagent grade.

2.2. Wastes

2.2.1. Biosorbents

The selected wastes, grapefruit peelings (GP), fern (FR), eucalyptus barks (EL), oak leaves (OL) and cane pruning of grapevines (CP), were provided by a local supplier. The lignocellulosic composition of the selected wastes is shown in Table 1. The wastes were washed with distilled water three times and then dried in an oven at 60 °C for 24 h. Afterwards, the dried wastes were grinded using a cutting mill Retsh SM100 until a size minor than 0.5 mm.

2.2.2. Pretreatment of biosorbents

The grapefruit peelings were pretreated using H_2O_2 according to Shen et al. (2011). Briefly, 1.5 g of biosorbent and 150 mL of H_2O_2 (1 M) were added to an 250 mL Erlenmeyer flask and stirred at 110 rpm for 24 h. These wastes were dried and grinded following the procedure described in the previous section.

2.2.3. Biosorbent characterisation

2.2.3.1. Scanning electron microscopy and energy dispersive spectrometry. Scanning electron microscopy and energy dispersive spectrometry (SEM/EDS) were performed on a JEOL JSM-6700F SEM equipped with an EDS Oxford Inca Energy 300 SEM using an accelerating voltage of 15 kV (Electron Microscopy Service, C.A.C.T.I., University of Vigo).

2.2.3.2. Fourier-transform infrared spectra. Fourier-transform infrared (FT-IR) spectra of the biosorbents were recorded on an FT-IR spectrometer (model FT-IR/4100, Jasco). Previously, the GP samples before and after adsorption processes were dried in an oven at 60 °C for 40 min.

2.2.3.3. Functional group evaluation. The acidic and basic oxygen surface functional groups distribution was measured via Boehm titration procedure (Boehm, 1966). The experiments were carried out in 250 mL Erlenmeyer flasks containing 1 g of pretreated biosorbent and 50 mL of 0.1 N solutions ($NaHCO_3$, Na_2CO_3, NaOH or HCl). The mixture was shaken in an incubator (Thermo scientific MaxQ800) for 24 h at room temperature, and then the supernatant was filtered to remove particles and back-titrated using an acid–base indicator. The number of acidic sites were calculated under the assumption that NaOH neutralises carboxyl, phenolic and lactonic groups, Na_2CO_3, carboxyl and lactonic and $NaHCO_3$ only carboxyl groups. The number of basic sites was calculated under the assumption that HCl neutralises the basic groups.

2.2.3.4. Point of zero charge. The point of zero charge (pH_{PZC}) was determined by mass titration (Noh and Schwarz, 1989). Briefly, 1 g of biosorbent was contacted for 24 h with 50 mL of $NaNO_3$ as background electrolyte at several different pH values within the range of 2 to 8. The solution pH was adjusted with diluted HNO_3 and NaOH up to achieve desired values. The pH supernatant was measured with a Jenway 3520 pH Meter after three-point calibration.

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