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Research article

A method for dye extraction using an aqueous two-phase system: Effect of co-occurrence of contaminants in textile industry wastewater

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ABSTRACT

This paper reports a green and efficient procedure for extraction of the dyes Malachite Green (MG), Methylene Blue (MB), and Reactive Red 195 (RR) using an aqueous two-phase system (ATPS). An ATPS consists mainly of water, together with polymer and salt, and does not employ any organic solvent. The extraction efficiency was evaluated by means of the partition coefficients (K) and residual percentages (%R) of the dyes, under different experimental conditions, varying the tie-line length (TLL) of the system, the pH, the type of ATPS-forming electrolyte, and the type of ATPS-forming polymer. For MG, the best removal ($K = 4.10 \times 10^4$, %R = 0.0069%) was obtained with the ATPS: PEO 1500 + $Na_2C_4H_4O_6$ (TLL = 50.21% (w/w), pH = 6.00). For MB, the maximum extraction (K = 559.9, %R = 0.258%) was achieved with the ATPS: PEO 400 + Na_2SO_4 (TLL = 50.31% (w/w), pH = 1.00). Finally for RR, the method that presented the best results ($K = 3.75 \times 10^4$, %R = 0.237%) was the ATPS: PEO 400 + Na_2SO_4 (TLL = 50.31% (w/w), pH = 6.00). The method was applied to the recovery of these dyes from a textile effluent sample, resulting in values of K of 1.17×10^4, 724.1, and 3.98×10^4 for MG, MB, and RR, respectively, while the corresponding %R values were 0.0038, 0.154, and 0.023%, respectively. In addition, the ATPS methodology provided a high degree of color removal (96.5–97.95%) from the textile effluent.
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1. Introduction

There is increasing global concern related to the use of water resources, due to the limited availability of unpolluted freshwaters and the high costs of obtaining potable water. As a result, environmental legislation has become more rigorous in several countries (Vajnhandl and Valh, 2014; Hessel et al., 2007) and there is the need for research aiming at the development of new technological procedures to prevent or minimize the discharge of industrial wastes to the environment (Chauhan et al., 2015; Fischer et al., 2011).

The textile industry has attracted much attention, not least due to its importance to the economies of many countries (Hasanbeigi and Price, 2015). This industrial sector generates large volumes of liquid effluents containing considerable quantities of dye compounds, mainly due to incomplete fixation of these substances during the step of dyeing the fibers and their subsequent loss during the removal of excess dye in the final washing steps and finishing of the fabric (Hessel et al., 2007).

The presence of dye compounds in aquatic environments reduces light penetration, which affects photosynthetic activity (Gürses et al., 2002), and the dyes may be absorbed by microorganisms and react with biologically important molecules (Banat et al., 1996). Moreover, one of the main characteristics of these pollutants is their durability and capacity to bioaccumulate, which means that they can remain in terrestrial and aquatic ecosystems for periods of up to around 50 years. Therefore, effluents from dye manufacturing and textile dyeing processes represent emerging ecological concerns, especially if they reach reservoirs or water

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treatment plants (Kabra et al., 2013).

Due to the environmental and economic concerns, several procedures have been developed for the treatment of wastes containing dyes, including oxidative processes (Nguyen and Juang, 2013; Constapel et al., 2009; Mondal, 2008), coagulation (Liang et al., 2014), use of surfactants (Montoneri et al., 2009), electrochemical treatment (Haque et al., 2015; Mondal, 2008; Gürses et al., 2002), biological methods (Zeng et al., 2015; Malachova et al., 2013), adsorption (Souza et al., 2013; Errais et al., 2012; Haider et al., 2011; Malik et al., 2007), and separation using membranes (Zuriaga-Agustí et al., 2014).

Despite the advantages inherent to each procedure, the techniques mentioned above have various limitations. The derivatives formed in oxidation processes can be even more toxic than the dye molecules; removal by coagulation generates significant amounts of other wastes; there are high costs associated with treatments involving adsorption (Hubbe et al., 2012), electrochemical procedures, and membrane separation; and biological plants require high commitments in terms of time and space (Pearce et al., 2003).

Hence, there is still a strategic demand for the development of new techniques and procedures for the removal of dyes from aqueous wastes. Furthermore, it is essential that these methodologies should be environmentally safe and follow the principles of Green Chemistry (Salgueiro-Gonzalez et al., 2012; Anastas and Eghbali, 2010; Rodrigues et al., 2010).

Shao et al. (2016) recently showed that liquid-liquid extraction can be used for wastewater remediation, achieving 98% extraction efficiency for naphthalene-2-ol using organophosphorus solvents. The liquid-liquid extraction process is versatile and the system can be formed by different combinations of components. In the last decades, aqueous two-phase systems (ATPSs) have been introduced as a promising alternative for the liquid-liquid extraction of dye compounds from aqueous matrices. The advantages of these systems are that they are mostly composed of water and other constituents that are not toxic or flammable, and that in some cases are biodegradable and recyclable (Rodrigues et al., 2013). Moreover, ATPSs present other features that are attractive from the applicability point of view, such as possibility of linear scale-up, ease of use, low cost, and short times for phase splitting without stable emulsion formation (Rodrigues et al., 2012). Under specific thermodynamic conditions, ATPSs are formed by mixing aqueous solutions of two chemically distinct polymers (Errais et al., 2012), solutions of a polymer and an electrolyte (Souza et al., 2013), or solutions of two electrolytes (de Alvarenga et al., 2015; Bridges et al., 2007), causing the formation of two phases in equilibrium. One of these phases, denoted the top phase, is rich in polymer (or electrolyte), while the other phase, denoted the bottom phase, is rich in the electrolyte (or the other polymer) (Rodrigues et al., 2009).

The applicability of ATPS as an extraction technique has been proved for dyes including Remazol Yellow Gold (de Alvarenga et al., 2015), Carmine (Mageste et al., 2009), Norbixin (Mageste et al., 2012), chloranilic acid and Rhodamine 6G (de Souza et al., 2014), and chloranilic acid, Indigo Blue, and Sudan III (Ferreira et al., 2014). However, textile processing produces a wastewater rich in dyes, salts, surfactants, softener, suspended solids, and organic matter. The co-occurrence of compounds can lead to changes in the dye extraction behavior in ATPS. Nonetheless, there is great potential for the use of ATPSs to remove dyes from textile effluent on an industrial scale.

The aim of this work was to develop a greener methodology for extraction of the dyes Malachite Green (MG), Methylene Blue (MB), and Reactive Red 195 (RR) (Fig. s1, Supplementary Material), employing an aqueous two-phase system. The influence of different parameters on the dye partition coefficient (K) was examined, considering the pH of the system, the nature of the ATPS electrolyte and polymer, and the tie-line length (TLL). Determination was made of the residual percentage (%R) of dye in the bottom phase after application of the procedure. The extraction method was then applied for the recovery of the dyes from a textile effluent sample, which was also submitted to a discoloration study using the ATPS.

2. Experimental

2.1. Apparatus

Stock solutions were prepared by weighing appropriate amounts of the compounds on an analytical balance (AUY 200, Shimadzu; uncertainty of ±0.0001 g). Spectrophotometric measurements were performed with a UV/Vis spectrophotometer (Varian Cary 100, Agilent Technologies), using quartz microcells with 1.00 cm optical path length. The pH measurements were made with a digital pH meter (HI2221, Hanna Instruments; uncertainty of ±0.02). A thermostatic water bath (SL 152, Solab; uncertainty of ±0.1 K) was used to control the temperature, and a centrifuge was used to accelerate the separation process.

2.2. Chemicals

All reagents were analytical grade and were used as received, without further purification. Distilled water was used in all the experiments. The chemicals employed in the proposed method were Li_2SO_4, Na_2SO_4, $Na_2C_4H_4O_6$, and polyethylene oxide with an average molar mass (M_m) of 1500 g mol^{-1} (denoted PEO 1500), all obtained from Vetec (Duque de Caxias, Rio de Janeiro, Brazil). In addition, other polymers of polyethylene oxide used in ATPS preparation were PEO 400 (Aldrich, St. Louis, MO, USA) and PEO 4000 and PEO 6000 (Synth, São Paulo, Brazil). The Malachite Green and Methylene Blue dyes were purchased from Vetec (Duque de Caxias, Rio de Janeiro, Brazil). The Reactive Red 195 dye was kindly provided by Quimisa S/A. The pH of the solutions was adjusted using sulfuric acid (H_2SO_4) and sodium hydroxide (NaOH), acquired from Vetec (Rio de Janeiro, Brazil) and Merck (New Jersey, USA), respectively.

2.3. ATPS composition, solutions, and standards

Aqueous two-phase systems were prepared by mixing aqueous polymer solutions with aqueous salt solutions to obtain a total system composition in accordance with the phase diagrams provided in Table s1 (Supplementary Material). Most of the phase diagrams for the ATPSs used in this work have been reported previously, except the equilibrium data for the PEO 4000 + $Na_2C_4H_4O_6$ + H_2O and PEO 6000 + $Na_2C_4H_4O_6$ + H_2O systems, which were obtained experimentally in this work using a method available in the literature (de Lemos et al., 2011).

Five or four different compositions were chosen from each phase diagram. The total weights of the ATPSs prepared were 40.0 g. After being shaken vigorously for about 1 min, they were allowed to equilibrate at 25.0 °C for a minimum of 12 h in a thermostatic water bath. Once thermodynamic equilibrium had been reached, the top and bottom phases were collected separately using syringes. The stock dye solution was then prepared using the top phase as solvent. The concentration was previously established according to the solubility of the dye in the polymer phase, simulating an effluent with a high content of the analyte. The concentrations used for each dye are provided in Table s2 (Supplementary Material).

Amounts of 3.0 g of each phase (salt-rich bottom phase and

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