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Removal of Reactive Blue 19 using nonionic surfactant in cloud point extraction

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ABSTRACT

Textile wastewater represents a serious environmental issue due to the presence of toxic dyes. The cloud point extraction, which involves the application of nonionic surfactants at temperatures above the cloud point, was applied in this research to remove Reactive Blue 19 dye from a synthetic wastewater. Nonylphenol with 9.5 ethoxylation degree was used as nonionic surfactant. Dye removal was evaluated considering the influence of surfactant concentration, temperature, and initial dye concentration. Equilibrium data followed the Langmuir isotherm model. Negative Gibbs energy, enthalpy, and entropy showed that the process was spontaneous and exothermal. Dye removal reached 91% when using 7.5 wt% surfactant, at 65.0 °C. Cloud point extraction can be used as an efficient alternative for treating textile wastewater containing Reactive Blue 19.

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1. Introduction

Because of the changing characteristics of the wastewater, studies of wastewater treatability are increasing, especially with reference to the treatment of specific constituents [1]. The textile industry is one of the major sources of water pollution. It uses a huge quantity of water and produces substantial volumes of colored wastewater. The removal of color from wastewater, due to the presence of unused synthetic dyes, is one of the most difficult tasks faced by the textile industry [2,3].

A dye is a natural or synthetic coloring substance with affinity to bind to a substrate, e.g. fabric. It is usually applied in an aqueous solution, and may require a mordant to improve the fastness of the dye on the fibers [4]. Dye classes include basic, acid, cationic, direct, reactive, sulfur, azoic, and disperse, with reactive being the most widely used in the textile industry.

Around 1 million tons of synthetic dyes, including reactive dyes, are produced every year worldwide and 5–15% of this amount is discharged as textile wastewater [5,6]. The main problem associated with the use of reactive dyes is their low affinity for textile material, with up to 50% of them present in the discarded bath [7,8]. Reactive dyes, in their original or hydrolyzed forms, are difficult to degrade biologically and are highly toxic to animals and humans [9]. In the case of Reactive Blue 19 (RB19), fixation efficiency in cotton ranges between 75% and 80%, due to the competition between the formation of vinyl sulfone and that of 2-hydroxyethylsulfone [10], Fig. 1 shows the molecular structure of Reactive Blue 19, explaining the formation of vinyl sulfone and 2-hydroxyethylsulfone [11]. According to Weber and Stickney [11], the hydrolysis of the vinyl sulfone moiety before the formation of a covalent bond between dye and fiber is the major problem associated with dye technology, because, after hydrolysis, this dye looses its affinity for the textile fiber.

It is difficult to remove dye from wastewater using conventional treatment systems [12]. A number of techniques have been developed in search of efficient alternatives to treat dye-bearing wastewater, including electro oxidation [13], adsorption [4,14], ionic-liquid-based aqueous two-phase systems [6], sonoelectrochemical degradation [7], and ozone application [15]. Oxidative processes can be complicated for Reactive Blue 19, since its anthraquinone aromatic structure, highly stabilized by resonance, makes it very resistant to chemical oxidation [16].

Surfactants are applied in many industrial separation processes. A solution of nonionic surfactant becomes cloudy at a certain temperature, known as cloud point temperature. Above this temperature, the solution will separate into two phases: an aqueous phase containing surfactant at a concentration slightly above its critical micelle concentration (cmc) and the other, a surfactant-rich one, called coacervate phase. When organic solutes are present in the solution, they separate along with the surfactant to the coacervate phase, in a process known as Cloud Point Extraction (CPE). This

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interaction occurs due to adsorption of the solute on the surface of the micelles or some others sites within micelles. This phenomenon can be suggested by the monolayer coverage of the solute on the surface of the micelles [17]. Cloud point extraction, as observed by several authors, is an effective technique for removing dissolved organic contaminants from wastewater and groundwater [17–23].

In the present research, experiments were developed to remove Reactive Blue 19 dye by CPE using a nonionic surfactant widely used in Brazil, despite its known toxicity [24]. Its residual concentration in the process after phase separation is very low, in the order of the cmc, which can still be resolved by recovery of solvent [20,21]. The removal efficiency was evaluated considering the influence of temperature, dye initial concentration, and surfactant concentration. The thermodynamic parameters were evaluated (enthalpy, entropy and Gibbs energy) and the Langmuir isotherm model was used to fit the equilibrium data.

2. Materials and methods

2.1. Material

Nonylphenol polyethoxylate with an average number of 9.5 ethylene oxide units per molecule (NP9.5EO) was used as nonionic surfactant (weight = 617 g mol^{-1}, complete solubility in water, viscosity 230–270 mPa s at 25 °C, density 1060 kg/m^3, cloud point 56 °C in aqueous solution at 1% m/m). Reactive Blue 19 (RB19) was used to obtain the synthetic dye wastewater (Dystar; molar weight = 626.54 g mol^{-1}; λ_{max} = 592 nm, blue). All reagents were acquired commercially and used without further purification.

2.2. Methods

2.2.1. Cloud Point Experiments (CPE)

In CPE, the NP9.5EO concentration was varied in 3.0, 3.5, 4.0, 4.5, 5.0, 5.5, 6.0, 6.5, 7.0, and 7.5% (m/m). The initial RB19 concentration was varied in 10, 25, 50, 75, 100, 125, 150, and 200 ppm. Five temperatures were chosen (65.0, 67.5, 70.0, 72.5, and 75.0 °C). First, in 100 mL – graduated test tubes (±1 mL), 100 mL solutions of NP9.5EO and RB19 were prepared by dissolving them in distilled water at different concentrations, according to each experimental run. The tests were performed in a thermostatic bath (Koehler Instrument Company, Inc, USA) at a constant temperature and stirring rate (500 rpm), for five minutes to assure homogenization. After, the samples were left at rest for two hours to allow phase separation. The volumes of the dilute and coacervate phases were visually measured in the graduated test tubes. Dye concentration in the dilute phase was determined with a UV–Vis spectrophotometer (Varian Analytical Instruments, Cary 50 Conc, USA). Dye removal efficiency was calculated by using Eq. (1):

$$\%Efficiency = \frac{C_{RB19,\,initial} - C_{RB19,\,dilute}}{C_{RB19,\,initial}} \times 100 \quad (1)$$

where $C_{RB19,initial}$ is the initial dye concentration and $C_{RB19,dilute}$ is the dye concentration in the dilute phase.

2.2.2. Adsorption isotherm

To describe equilibrium data, dye concentrations between aqueous and coacervate phases were fit to the Langmuir isotherm model. The linear expression for the Langmuir model is given by Eq. (2):

$$\frac{1}{q_e} = \frac{1}{q_m} + \frac{1}{q_m k_L C_e} \quad (2)$$

where q_e is the equilibrium adsorption capacity (g of RB19/g of NP9.5EO), C_e is the equilibrium concentration in the dilute phase (g/L), q_m the maximum adsorption capacity (g of RB19/g of NP9.5EO) and k_L is the Langmuir parameter (L/g of RB19).

3. Results and discussions

3.1. Dye removal efficiency

In order to maximize dye removal efficiency by CPE, the influences of surfactant concentration and temperature were first evaluated, maintaining constant the initial dye concentration (100 ppm). Surfactant concentration was fixed between 3.0 and 7.5% (m/m), since preliminary tests showed that dye removal was very low at concentrations below 3% and concentrations above 7.5% were not considered economically viable. Two factors were considered to determine temperature range, as follows: temperatures above the cloud point (56.0 °C), in order to guarantee phase separation, and work in a temperature range close to that found in the textile effluent which can reach 70 °C [25]. Fig. 2 shows the extraction efficiency for different temperatures and initial surfactant concentrations.

In Fig. 2 one can observe that removal efficiency decreases with increasing temperature and increases with increasing surfactant concentration, especially in the surfactant concentration range between 3.0 and 6.0% (m/m). An increase in surfactant concentration outside this range will not cause a significant increase in dye removal efficiency. This behavior was expected because the number of micelles rises with increasing surfactant concentration,

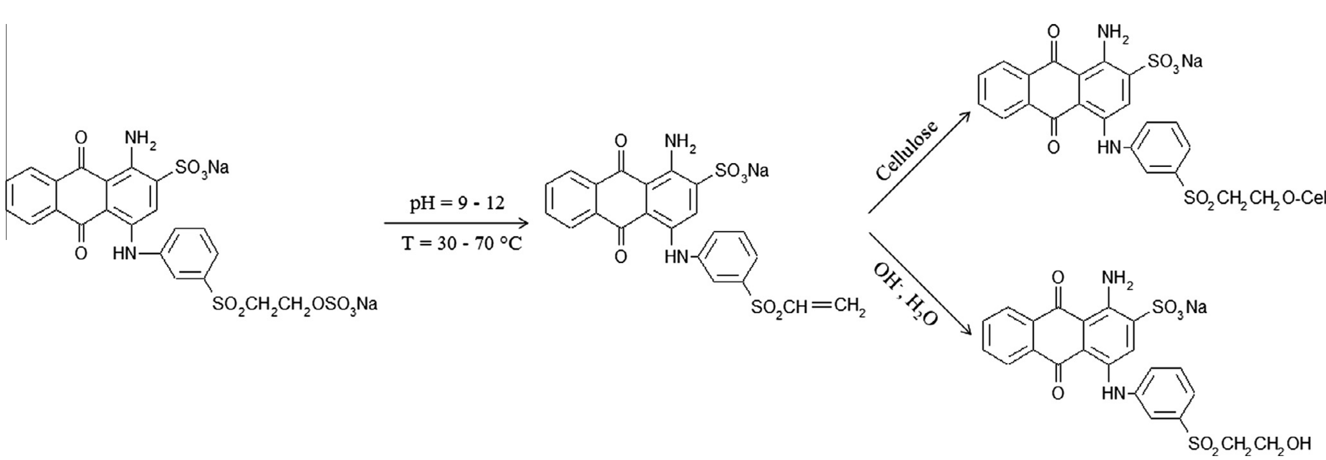

Fig. 1. The molecular structure of Reactive Blue 19, explaining the formation of vinyl sulfone and 2-hydroxyethylsulfone.

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