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ORIGINAL ARTICLE

Liquid–liquid extraction of methylene blue dye from aqueous solutions using sodium dodecylbenzenesulfonate as an extractant

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Stripping;
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Abstract The extraction of methylene blue dye from aqueous solutions has been investigated using sodium dodecylbenzenesulfonate as an extractant. The various diluents used were benzene, toluene, xylene and dichloroethane. Dichloroethane was found to be the most effective diluent for the extraction of the dye. The investigated parameters, governing the extraction of the dye, were equilibrium time, pH of the dye solution, extractant concentration, dye concentration, temperature and aqueous to organic phase ratio. The dye was totally extracted at the optimum conditions. The dye loaded in the organic phase was stripped using various types of acids as stripping agent; complete recovery of the dye was achieved using 0.5 M thiourea in 1 M HCl.
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1. Introduction

Dyes are widely used in various industries such as textile, paper, leather and plastic. Textile industries produce large amounts of liquid wastes that contain organic and inorganic compounds [1]. The major operations performed in a typical textile processing industry are desizing, scouring, mercerizing, bleaching, neutralizing, dyeing, printing and finishing. The discharge of polluted effluents and use of various raw materials may cause contamination of soil, ground water and surface water which may have adverse consequences on environment in general and local population in particular. The effluents generated from the textile industry are of utmost concern because of their high volume and pollution potential. The release of colored wastewater from this industry may present eco-toxic hazard and may eventually affect human life through food chain if accumulated [2]. Color interferes with the transmission of sunlight into the stream and therefore reduces photosynthetic action so it is essential to treat the textile wastewater prior to discharge [3].

Methylene blue (MB) is one of the most commonly used substances for dyeing cotton, wood and silk. Though MB is not strongly hazardous, it can cause some harmful effects where acute exposure to MB will cause increased heart rate, vomiting, shock, cyanosis, jaundice, and quadriplegia and tissue necrosis in humans [4]. It is, therefore, essential to remove the dye from wastewater or treat it in such a way so as to

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minimize the damage to the environment and also to decolorize the water [5]. Various methods have been used to remove dyes from aqueous solutions. The widely used methods include micellar enhanced ultrafiltration [6], several oxidation processes [7], electrochemical degradation [8], ozone based processes [9], photo catalytic degradation [10–16], electrocoagulation [17], nanofiltration (NF) [18–21], adsorption onto agricultural solid waste [22], different bentonites [23], various types of activated carbon [24,25], biological treatments [26–28]. The physical methods are non-destructive and merely transfer the pollutants from one medium to another, thus giving secondary treatment [29]; chemical methods are not economically viable due to high dosage and production of a large quantity of sludge [30–32]. Ultrafiltration (UF) and nanofiltration (NF) can be used for complete removal of all classes of dye, but care is needed to avoid membrane fouling which decreases the flux. Due to low biodegradability of dyes, the conventional biological wastewater treatment process is not very efficient in treating dye containing wastewater [33].

In recent years, much attention has been focused on a separation technique such as solvent extraction or liquid–liquid extraction (LLE) [34–39]. LLE is based on the principle that a solute can distribute itself in a certain ratio between immiscible solvents, and the extraction process depends on its mass transfer rate [40]. Advantages of LLE include high throughput, ease of automatic operation and of scale up and high purification [41].

In the present work the influence of the main operating parameters, such as effect of equilibrium time, dye concentration, initial pH, effect of diluents, sodium dodecylbenzenesulfonate, temperature and aqueous to organic phase ratio on extraction of dye from aqueous solution has been investigated. Also, the stripping of dye from organic phase and the reusability of solvent have been studied.

2. Experimental technique

Commercially available methylene blue dye was used for the preparation of synthetic dye solution. The dye was obtained from El-Nasr Company, Alexandria, Egypt, and its molecular structure is shown in Fig. 1. Sodium dodecylbenzenesulfonate (MW = 348.5) was used as an ionic surfactant. The solvent used for the removal of dye from water was dichloroethane. UV–visible spectrophotometer (Labomed, USA) with the calibration method at maximum wavelength of 640 nm was used to measure the absorbance of the dye. pH of an aqueous solution was measured by a pH meter and was adjusted by sodium hydroxide and nitric acid solutions. The dye solution was prepared in distilled water. Hydrochloric acid mixed with thiourea was used as stripping agent. All chemicals used in this study were of AR grade.

The organic solvent used for extraction was added to the prepared aqueous dye solution in a glass-stoppered bottle.

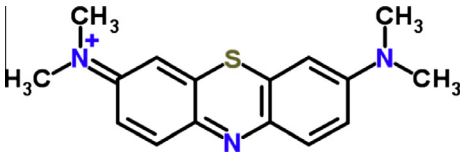

Figure 1 Structure of methylene blue.

The pH was adjusted and the bottles were shaken in a thermostatted shaker at 250 rpm for a known time. The two-phase dispersions were allowed to settle for 48 h to form two clear liquid phases: the solvent phase containing the dye and the clear aqueous phase. Samples were drawn from aqueous phase using 10 ml pipette for absorbance measurement of the dye to determine dye concentration. The percentage of extraction (E) was calculated as follows:

$$E = \frac{C_{aq0} - C_{aq}}{C_{aq0}} \times 100 \quad (1)$$

where C_{aq0} is the initial dye concentration of aqueous phase (mg/L), C_{aq} is the dye concentration of aqueous phase after extraction (mg/L).

In stripping, the loaded extractant and the aqueous acid solution were added together into a glass stoppered bottle and shaken at 250 rpm for a known time. The content was then allowed to settle for 20 min. The aqueous strippant was taken for dye concentration measurements.

3. Results and discussions

In order to evaluate the effect of pH of the solution on the percentage of dye extracted, a series of experiments were performed and the dye solution was adjusted to the desired pH by adding NaOH or HNO_3 solutions. As shown from Fig. 2, the percentage dye extracted increases from 20.2% to 100% as the pH increases from 2 to 12. The changes in the behavior of the dye could be attributed to the hydrolysis or aggregation at these pH conditions [35]. The results reveal that the maximum extraction of dye occurred in the pH range of 9–12. For further studies, it was decided to maintain the extraction at pH 11.

The effect of equilibrium time on the percentage of dye extracted was studied at 10, 15, 20, 25 and 30 min. Fig. 3 shows that the percentage of extraction increased significantly at the initial stage (93.65%). The extraction efficiency of dye increased with increase in equilibration time. The whole dye was transported to the organic phase after 25 min (100% dye extracted), and hence an equilibrium period of 25 min. is recommended.

The effect of sodium dodecylbenzenesulfonate on the extraction of dye from aqueous solution was investigated in the concentration range 0.02–0.07 M. Fig. 4 shows that the percentage of extraction increased significantly in all concentration ranges with maximum extraction occurring at 0.06 and 0.07 M; therefore, all subsequent experiments were performed at 0.06 M.

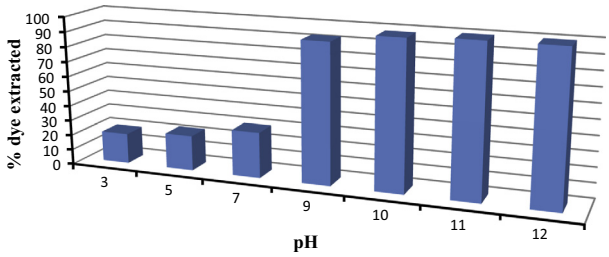

Figure 2 Effect of pH. (A/O phase ratio = 1:1, dye concentration = 30 mg/l, extractant concentration = 0.06 M, equilibrium time = 25 min., temperature = 20 °C).

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