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Extraction of methyl red from industrial wastewater using xylene as an extractant

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Abstract

A laboratory study on the liquid–liquid extraction system has been carried out for the removal and recovery of methyl red from aqueous solutions using xylene as an extractant. The concentration of methyl red has been studied in the range of 1.72×10^{-4} to 1.72×10^{-3} mol L^{-1}. The efficiency of dye extraction increased with increasing time of extraction. Distribution ratio is reasonably high ($D = 49$) even in the presence of inorganic salts. Extracted dye in the organic phase has been stripped into sodium hydroxide solution. The organic solvent is reused for reextraction of the dye from aqueous solution. The efficiency of reused organic solvent was maintained up to 10 runs. Loading capacity of dye has been found to be 14.32 mg. Under optimized conditions, real textile wastewater has been studied and the results are satisfactory.

Keywords: Liquid–liquid extraction; Distribution ratio; Extractant; Strip; Methyl red; Reextraction

1. Introduction

Dyes are widely used in various industries, such as textiles, leather, plastic, paper and cosmetics, for coloring their final products. The release of colored wastewater from industry may produce an eco-toxic hazard and introduce potential danger of bioaccumulation, which may eventually affect humans through the food chain [1]. Azo dyes are the largest group of dyes used in industry [2]. The term azo dye is applied to synthetic organic colorants that are characterized by a nitrogen-to-nitrogen double bond: $-N=N-$ [3]. Durability of azo dyes causes pollution to the environment. Besides, some azo dyes are toxic and mutagenic [4]. Methyl red is an anionic azo dye [5]. It is well known that methyl red dye has been used in paper printing and textile dyeing [6] and it causes irritation of the eye, skin and digestive tract if inhaled/swallowed [7]. It is, therefore, essential to remove the dye from wastewater or treat it in such a way so as to minimize the damage to the environment and also to decolorize the water [8]. Textile wastewater contains residual dyes that are difficult to remove in conventional treatment plants [9].

Various methods have been used to remove dyes from aqueous solutions. The widely used methods include adsorption, coagulation, photocatalytic degradation, ozone treatment, electro Fenton's and hypochlorite treatment [10–19]. The physical methods are non-destructive and merely transfer the pollutants from one medium to another, thus giving secondary treatment [20]. Chemical methods are not economically viable due to high dosage and production of a large quantity of sludge [21–23].

In recent years, much attention has been focused on a separation technique such as solvent extraction or liquid–liquid extraction (LLE) and liquid membrane. 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 [24]. The

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advantages of LLE include high throughput, ease of automatic operation and of scale up and high purification [25].

Lee et al. [24] reported the removal of dye from water using predispersed solvent extraction. In this study, kerosene was chosen as the organic solvent and solvent yellow 1 was used as the dye. The removal of anionic methyl orange dye from water was carried out in the presence of cationic hexadecyltrimethylammonium bromide surfactant. And the removal of cationic methylene blue dye was carried out in the presence of anionic sodium dodecylbenzene surfactant solution where amyl alcohol was used as the solvent [26]. Hu et al. [27] reported the recovery of strong acid dye from wastewater by solvent extraction, and multistage counter current extraction and stripping experiments were carried out. The reusability of the organic phase regenerated was tested. Removal of azo dyes from aqueous solutions using Calix [4] arene and β-cyclodextrin was investigated by LLE [28] and dichloromethane has been used as the extractant. The recoveries of methylene blue by LLE with propylene and 1,2-butylenes carbonates were significantly better compared with the cloud point technique. Cloud point technique yielded 10–49% recoveries in the concentration range of 0.01–0.25 mol L^{-1}, whereas LLE yielded almost 100% recoveries [29].

Muthuraman et al. [30] reported the extraction and recovery of methylene blue from industrial wastewater by LLE, using benzoic acid as the extractant. Under optimized conditions, 99% of the dye was extracted from aqueous solutions and the extracted dye in the organic phase was back extracted into sulphuric acid solutions. Similarly, golden yellow low salt anionic-type dye was extracted and recovered from aqueous solutions by the solvent extraction method using tri-*n*-butyl phosphate as the carrier. The extracted dye was stripped into 0.01 mol L^{-1} sodium hydroxide solutions. The solvent used was recycled [31]. The selective extraction and separation of textile anionic dyes from aqueous solution were also reported [32]. Tetra butyl ammonium bromide was used as a carrier and methylene chloride was used as a diluent. The extracted dye was back extracted into salicylic acid and sodium carbonate solution. The stripped solution upon acidification precipitated salicylic acid and the dye was recovered.

In the present study, the recovery of an anionic dye, methyl red, from an aqueous solution has been studied by LLE using xylene as an extractant. Parameters such as, pH in feed, NaOH concentration in strip, effect of time, individual dye concentration, aqueous to organic phase ratio, addition of inorganic salts and organic to aqueous phase ratio have also been studied.

2. Experimental

2.1. Materials

Xylene, methyl red, sulphuric acid, sodium hydroxide, sodium chloride, nitric acid and hydrochloric acid were obtained from Merck. All chemicals used in this study were of AR grade.

A UV visible spectrophotometer (Spekol 1200, Analytical Jena, Germany) was used to measure the absorbance of the dye and to establish its λ_{max} and its concentration. The pH of an aqueous solution was measured by a pH meter (WTW, Germany). A mechanical stirrer (IKD-KS 50, Germany) was used for agitation of solutions.

Xylene was used as an extractant. The dye solution was prepared in distilled water. Sodium hydroxide was used as the stripping agent and sulphuric acid was used to adjust pH.

2.2. Procedure

2.2.1. LLE of dye

The organic solvent [(xylene) (V_o mL)] used for extraction was added to the prepared aqueous dye solution (V_a mL) in a glass-stoppered bottle and the glass-stoppered bottle was shaken for a known time in a shaker at 100 rpm. The mixture was then transferred into a separating funnel. A sample of the aqueous solution at the bottom of the separating funnel was withdrawn for absorbance measurement of dye. The experimental set up was the same as that in an earlier work [30]. The wavelength of maximum absorption (λ_{max}) for methylene red was 412 nm. The structure of the dye is shown in Fig. 1. The distribution ratio (D) and percentage of extraction (E) were calculated by the following equations

$$D = [\mathrm{dye}]_{org}/[\mathrm{dye}]_{aq} \quad (1)$$

$$E = 100 \times ([\mathrm{dye}]_{aq0} - [\mathrm{dye}]_{aq})/[\mathrm{dye}]_{aq0} \quad (2)$$

where $[\mathrm{dye}]_{org}$ is the dye concentration in organic phase (mg L^{-1}), $[\mathrm{dye}]_{aq0}$ the initial dye concentration of aqueous phase (mg L^{-1}), and $[\mathrm{dye}]_{aq}$ the dye concentration of aqueous phase after extraction (mg L^{-1}).

In stripping, the loaded extractant (V_o mL) and the aqueous strippant (sodium hydroxide solution) were added together into a glass-stoppered bottle and shaken at 100 rpm. The content was then transferred into a separating funnel. The aqueous strippant was taken for dye concentration measurements. All the experiments were run in duplicate and the analytical parameters were performed in triplicate for each run. A confidence limit of 95% was taken for reliable results.

The extraction and stripping processes were repeated on aqueous dye solutions in which inorganic salts were added to study the effect of the presence of these salts.

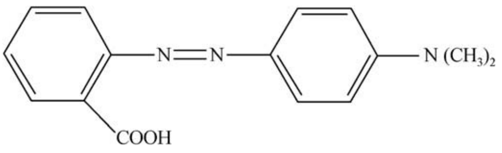

Fig. 1. Structure of methyl red.