

Separation of aromatic alcohols using micellar-enhanced ultrafiltration and recovery of surfactant

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Abstract

The removals of single aromatic alcohols, including *para* nitro phenol (PNP), *meta* nitro phenol (MNP), phenol (P), catechol (CC), beta naphthol (BN) and *ortho* chloro phenol (OCP) from aqueous solution have been studied using micellar-enhanced ultrafiltration (MEUF). Cetyl (hexadecyl) pyridinium chloride (CPC) has been taken as the cationic surfactant. An organic polyamide membrane of molecular weight cut-off 1000 is used in the MEUF experiments. Experiments are conducted using unstirred batch cell and a continuous cross flow cell. The effects of surfactant-to-solute concentration ratio in the feed, transmembrane pressure drop and cross flow rate on the permeate flux and observed retention of each solute have been studied in detail. The retention of solutes without using surfactant varies from 3 to 15% only at a typical feed solute concentration of 0.09 kg/m³. However, under the same operating pressure (345 kPa), retention increases to about 66–98% depending on the nature of solute at the end of 30 min of experiment in the batch cell using surfactant micelles (10 kg/m³). The maximum retention of solute is obtained at surfactant-to-solute concentration ratio of 110. Free surfactant molecules present in the permeate and retentate are then recovered by a two-step chemical treatment process. In the first step, the surfactant is precipitated by potassium iodide and in the second step, the surfactant is recovered from the precipitate by the addition of cupric chloride. Optimum consumptions of potassium iodide and cupric chloride are also obtained experimentally.

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1. Introduction

Phenol and its derivatives like *para* and *meta* substituted nitro phenols, catechol, beta naphthol and *ortho* chloro phenol are introduced into the aquatic environment through various sources, such as waste disposal from different chemical and petrochemical industries. The presence of these compounds in waste water emerges as the focus of environmental remediation efforts because of their toxicity and threat directly to aquatic and indirectly to non aquatic life.

Traditional methods for the separations, purification and concentration of these toxic chemicals often involve a phase change as in distillation or extraction followed by distilla-

tion. These techniques are energy intensive. Separations of these toxic chemicals are often unattainable to a satisfactory level through conventional separation based on adsorption. Therefore, it is desirable to develop a low energy separation process.

Rate governed separation process, like reverse osmosis (RO) is more useful and already recognized as the best available technique for the separation of several inorganic and organic compounds. Compared to nanofiltration (NF), ultrafiltration (UF) and microfiltration (MF), relatively 'dense' membranes are used in RO process. Permeability of RO membranes is very low, and thus, to get desired throughput (permeate flux), high operating pressure is required. Therefore, a modified membrane separation process can prove to be a competitive alternative where operating pressure requirement is low compared to RO and a membrane of higher permeability can be used.

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Micellar-enhanced ultrafiltration (MEUF) is one such promising technology that employs surfactant micelles to solubilize inorganic and organic contaminants from the aqueous stream [1]. In MEUF, the solute-containing stream needs to be treated with a surfactant stream with a concentration well above the critical micellar concentration (CMC). Most of the solute molecules are solubilized in the micelles. Micelles being larger in size can be removed along with the solubilized organic contaminants using a relatively porous membrane at lower operating pressure. The permeate stream contains unsolubilized solutes and free surfactants. Therefore, MEUF may be an alternative to overcome the limitations of the RO process.

A review of literature related to MEUF reveals that a number of studies have been carried out to understand the mechanism of separation and solubilization of the solute in the micelles to optimize the operating conditions. Dunn et al. [2,3] have studied the performance of MEUF to remove dissolved 4-tert-butylphenol (TBP) from aqueous phase using hexadecyl (cetyl) pyridinium chloride (CPC) as a cationic surfactant. Tucker and Christian [4] studied the effect of added sodium chloride on solubilization of benzene during MEUF using sodium octylsulfate as an anionic surfactant. Solubilization studies of phenol during MEUF have also been studied [5,6].

Bhat et al. [7] carried out both solubilization as well as MEUF studies on cresol and CPC micelles in aqueous medium under a wide range of composition and concluded that highly polar solutes tend to solubilize in the vicinity of the micellar surface due to a strong ionic or polar interaction between the organic solutes and the micelles. On the other hand, hydrophobic solutes are solubilized to a significant extent within the micellar interior. Apart from the experiments on MEUF of organics, MEUF can successfully be used to separate toxic dye [8,9] and different metal ions [10–12]. In MEUF, factors, those determine the extent of solubilization of organic and inorganic compounds in the micelles are the structure and ionic character of both the solute and surfactant molecule and the medium of the solute–surfactant system [13].

In the present work, separation of different aromatic alcohols from aqueous stream based on MEUF using unstirred batch UF cell and rectangular cross flow cell is studied. The effects of important factors like ionic character and structure of the solute, surfactant-to-solute ratio and trans membrane pressure drop on the solubilizing capacity of CPC micelles and the concentration polarization over the membrane surface in terms of retention and flux have been studied in detail. To make the MEUF system more economical, it is necessary to recycle the surfactant molecules present both in the retentate and permeate stream before final disposal. Several authors have studied the precipitation method for the recycling of sodium dodecyl sulphate (SDS) using multivalent counter ions [1]. Air stripping method for the separation of surfactant contaminant and surfactant reuse has also been reported by Lipe et al. [14]. Many researches performed MEUF experi-

ments using CPC as the cationic surfactant [15] but experiments for the recovery of CPC are still scant. The present work describes the recovery of CPC by the precipitation method [8]. A two-step chemical treatment process has been adopted to recover the surfactant from the permeate stream. In the first step, monovalent iodide (I^-) is used to precipitate the cetyl pyridinium ion. In the second step, concentrated CPC is regenerated using cupric chloride solution and is recycled to the feed stream. The optimum consumptions of potassium iodide and cupric chloride are also obtained experimentally. The same procedure is used to recover the surfactant from the retentate stream. A schematic of the MEUF and surfactant recovery is shown in Fig. 1.

2. Experimental

2.1. Materials

(i) The surfactant CPC (MW 358.01) is procured from SISCO Research Laboratories, Mumbai, India, (ii) all phenolic compounds (phenol (MW 94.11), catechol (MW 110), *ortho* chloro phenol (MW 128.56), *para* nitro phenol (MW 139), *meta* nitro phenol (MW 139) and beta naphthol (MW 144.16) are obtained from Merck Ltd., Mumbai, India and (iii) potassium iodide (KI, MW 166.01) and cupric chloride ($CuCl_2 \cdot 2H_2O$, MW 170.48) are also obtained from Merck, Mumbai, India. The osmotic pressure can be estimated using Vant Hoff's equation. For the maximum concentration of 0.5 kg/m^3 used in these experiments, the osmotic pressure will be about 2 kPa for phenol.

2.2. Membranes

Organic polyamide membrane of molecular weight cut-off (MWCO) 1000 obtained from Genesis Sepratech Pvt. Ltd, Mumbai, India, is used for all the MEUF experiments. The membrane material is hydrophilic in nature and used without any further treatment. Permeability of the membrane is measured using standard technique and is found to be $3.35 \times 10^{-11} \text{ m/Pa s}$.

2.3. Experimental set up

2.3.1. Batch cell

The unstirred batch experiments are conducted in a $50 \times 10^{-6} \text{ m}^3$ capacity filtration cell (MILLIPORE, model 8050, USA). Inside the cell, a circular membrane is placed over a base support. The effective membrane area is $13.4 \times 10^{-4} \text{ m}^2$. The maximum allowable pressure is 518 kPa. The permeate is collected from the bottom outlet of the cell. The cell is pressurized using a nitrogen cylinder.

2.3.2. Cross flow cell

A rectangular cross flow cell, made of stainless steel, is designed and fabricated. A schematic of the experimental set up is shown in Fig. 2. The cell consists of two matching

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