The concentrating of alizarin using a reverse osmosis process

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(Received 11 December 2003, revised 7 June 2004)

Abstract: Membrane technologies in general and reverse osmosis in particular have been employed for the concentrating of solutions. In this study, the concentrating of a heat sensitive alizarin extracted from madder root was realized using an FT30 reverse osmosis membrane. The effects of cross flow velocity, transmembrane pressure and pH on the flux and rejection were studied. Increasing the transmembrane pressure increased the flux while the rejection was constant. At pH 7–8, the highest flux was achieved. This study showed that reverse osmosis is the process of choice for the concentrating of alizarin solutions. The optimum operating conditions were 1.0 m/s crossflow velocity, 16 bars transmembrane pressure and pH 7. The system was tested for 12 h without severe fouling problems.

Keywords: reverse osmosis, alizarin, FT30 membrane.

INTRODUCTION

Alizarin is one of the dyes based on the anthracene structure that can be used in the textile and food industry. Alizarin may be prepared synthetically or from natural sources, such as madder root. If the dye is to be used in the food industry, the source must be natural and for a saleable product, the dye must be in powder form.

The production of a powder form dye is possible using membrane technology followed by spray drying. This process eliminates thermal damage to a heat sensitive substance, such as alizarin. The operation is automatic and therefore the cost of labor may be drastically reduced.1

Membranes in general and reverse osmosis in particular have been employed for processing of dye effluents mostly for treatment purposes. A wide range of membranes, from home-made cellulose triacetate2 to commercial cellulose acetate,3 have been used for textile dye waste treatment.

Ultrafiltration was successfully employed for recycling high molecular weight and insoluble dyes.4,5 Although ultrafiltration is not capable of removing low molecular weight and soluble dyes6 efficient color removal may be achieved by

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nanofiltration and reverse osmosis, even for special dyes. The retention depends on the feed pressure and concentration. The mechanisms involved in retention and flux decline are not clearly understood. The influences of the presence of salt on dye processing have been studied for nanofiltration and ultrafiltration.

Multistage operations consisting of pre-filtration, neutralization, nanofiltration and reverse osmosis or screening, coagulation, microfiltration and reverse osmosis have been conducted for the treatment of dye effluents. The recovery of water was around 85–95 %. The energy consumption was moderate. Noël et al. applied an electric field to eliminate fouling problems.

Gholami et al. evaluated the effectiveness of biodegradation versus a membrane process for the removal of basic, reactive, disperse and acidic dyestuffs from a textile waste stream. The results demonstrated a higher removal efficiency for reverse osmosis treatment compare to the biodegradation process.

A comprehensive literature survey revealed that membrane technology has not been applied for the concentration of alizarin. In this study the concentration of an alizarin solution extracted from a natural source (madder root) using the reverse osmosis process has been elucidated.

**MATERIALS AND METHODS**

**Apparatus**

All experiments were carried out in a reverse osmosis rig at 28 °C. The rig (Fig. 1) consisted of a feed tank, a high-pressure pump, valves, pressure gages and a stainless steel cross flow cell with a membrane area of 0.002 m². The operational mode was cross flow batch concentration, i.e., the concentrate was recycled to the feed tank.

**Membrane**

An FT30 polyamide composite membrane was used for all experiments. This is a reverse osmosis membrane produced by the FILMTEC Company (United States).
Feed
The feed was a colored solution of alizarin. In all experiments, the feed solution was prepared by heating a mixture of madder root and distilled water up to the boiling point. After filtering the mixture through a filter paper, the solution was poured into the feed tank for concentrating purposes. The initial concentration of the feed was 4%.

Flux and rejection

The purpose of this work was to determine the best operating conditions for the concentrating of alizarin solutions. In order to evaluate the success of the concentrating process, the flux and rejection were measured versus time for different conditions, i.e., transmembrane pressures and crossflow velocities.

The permeate flux was calculated by measuring the permeate weight over a specified time and the rejection was determined using Eq. (1) by measuring the absorbance at $\lambda_{\text{max}} = 500$ nm.

$$R \% = \frac{A_f - A_p}{A_f} \times 100 \quad (1)$$

where $A_f$ and $A_p$ are the absorbance of the feed and the permeate solution, respectively. $\lambda_{\text{max}}$ is the maximum absorption wavelength.

RESULTS AND DISCUSSIONS

Effect of cross flow velocity

The behavior of the flux and rejection for different crossflow velocities during 180 min operating time are presented in Figs. 2 and 3, respectively. The crossflow velocity is one of the hydrodynamic factors that enable fouling to be minimized. Usually, a higher crossflow velocity causes a turbulence and results in an increment in mass transfer and flux. However for the conditions of this study, a moderate crossflow velocity (0.5 m/s) provided a slightly higher flux. A higher crossflow velocity (0.8 and 1.0 m/s) may initially produce a greater flux but the deposi-
tion of the alizarin molecules in the membrane matrix is higher due to the higher diffusion of the molecules into the membrane. This results in a lower flux.

For all membrane processes including this work, the flux decreases with time due to fouling or irreversible deposition of materials on the membrane surface or in the membrane matrix.

Rejection is the second factor that may be employed for the evaluation of the efficiency of a concentrating process. A higher rejection means a lower passage of alizarin molecules through the membrane, i.e., a lower loss of alizarin. As indicated in Fig. 3, the rejection varied between 96 to 100%. The rejection data show a “sag” profile. The rejection is initially high due to the adsorption of molecules into the membrane matrix. After accommodating the adsorption sites, the alizarin molecules pass through the membrane more easily, i.e., the rejection is lower. The rejection gradually increased due to the deposition of alizarin molecules and the formation of a secondary barrier which acts as a membrane and prevents the passage of other molecules through the membrane.

These numbers, i.e., a flux around 20 kg/m² h and a rejection higher than 99 %, indicate the suitability of the reverse osmosis process for the concentrating of alizarin solutions.

**Effect of transmembrane pressures**

One of the factors that affects the flux and rejection is the transmembrane pressure. Usually increasing the transmembrane pressure causes an increase in flux (Fig. 4). Rejection varies from 98 to 100% (Fig. 5). However, a higher transmembrane pressure provides better conditions for fouling.
Effect of pH on the flux and rejection

The final fluxes and rejections (after 3 h of operation) at different pH values are given in Figure 6. The flux in basic and acidic media is higher. This behavior may be attributed to the charges of alizarin particles. In basic solution, alizarin is negatively charged.\textsuperscript{18} Repulsion between the charged dyes is responsible for the higher flux with high pH values. Probably at pH 6 alizarin is neutral so there is no repulsion between the dye molecules, therefore, the condition for fouling is provided which results in a lower flux. At pH 4 and pH 5, the flux is higher compared to that at pH 6,
which is probably due to the repulsion between the dye molecules that carry a positive charge. The rejection is approximately constant. This may be related to the sieving mechanism of the separation process. In this case, pH has no effect.\textsuperscript{18}

**Flux and rejection for long term operation**

In order to evaluate the efficiency of the membrane for long term concentrating of alizarin, an experiment was carried out for 12 h. Figure 6 indicates that the flux decline during time is negligible. However, the rejection (Fig. 7) increased with time from 95 % to 99 %. This increase in the rejection is probably due to the deposition of material on the membrane surface which acts as a secondary barrier.
Concentration of alizarin

The investigation of parameters affecting the flux and rejection in the previous sections revealed that the optimum conditions for the concentrating of alizarin solutions using FT30 reverse osmosis membranes are 1.0 m/s crossflow velocity, 16 bars transmembrane pressure and pH 7. The long term experiments indicated that a flux of 15 kg/m² h and a rejection of 99 % are achievable without severe fouling during the concentrating process.

For the concentrating of alizarin solutions, a membrane system may be employed with the above mentioned operating parameters. The required membrane area depends on the volume of the alizarin solution. The operating time can be easily calculated from the flux data for any batch of alizarin.

CONCLUSIONS

This study shows that the concentrating of alizarin solutions with a reverse osmosis membrane (cold concentrating) is possible without the problems associated with other concentrating processes.

Working with a low crossflow velocity may causes membrane fouling which decreases the efficiency of the reverse osmosis process. Experiments of long-term operation indicate that rejection versus time increased due to the deposition of feed materials on the membrane surface. Operation at different pH values revealed that the pH affects the efficiency of the membrane process. With basic and neutral pH values, the flux is higher compared to operation under acidic conditions.

The optimum operating conditions for the concentrating of alizarin solutions using FT30 reverse osmosis membranes are 1.0 m/s crossflow velocity, 16 bars transmembrane pressure and pH 7. The system was tested for 12 h without severe fouling problems.

ИЗВОД

КОНЦЕНТРОВАЊЕ АЛИЗАРИНА КОРИШЋЕЊЕМ ПРОЦЕСА РЕВЕРЗНЕ ОСМОЗЕ

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Мембранске технологије, а посебно реверзна осмоза користе се за концентровање раствора. У овом раду остварено је концентровање на топлоту осетљивог алizarина из корена броћа коришћењем FT30 реверзне осмотске мембране. Проучавани су утицаји унакрсних брзина протока, разлике притисака на мембране и pH на флукс и сепарацију. Повећање трансмембранско притиска повећава флукс, али сепарација остаје константна. Највећи флукс добијен је при pH 7 – 8. Ова студија је показала да је коришћење процеса реверзне осмозе добар избор за концентровање алizarина. Опти-
malni uslovi su 1,0 m/s unakrsne brzine protoka, 16 bara transmembranskog pritiska i pH 7. Sistem je proban tokom 12 časova bez ozižnijih problema.

(Primljeno 11. decembra 2003, revidirano 7. juna 2004)

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