

ABSTRACT

Rapid growth in industrialization and urbanization has resulted in release of huge quantities of waste streams containing valuable chemical entities. Traditional methods employed for recovery of these value added products generate large quantities of solid wastes and require huge capital and operating costs. The present research focuses on application of membrane based strategies such as membrane contactors (MC), electrodialysis (ED), reverse osmosis (RO), membrane distillation (MD) and forward osmosis (FO) for recovery of acids, solvents, catalysts and sugars from aqueous streams. Hydrophobic membranes such as polyvinylidene fluoride (PVDF), polystyrene (PS) and polyvinyl chloride (PVC) were selected on the basis of contact angle, chemical and thermal stability besides ability to undergo modification. Hydrophobic membranes were prepared by phase inversion technique and their properties were altered using additives such as polyurethane (PU), inorganic salts, high boiling solvents and zeolites. Hydrophilic FO membrane was prepared by interfacial polymerization technique on an ultraporous polyethersulfone (PES) layer. Membranes were extensively characterized using FTIR for intermolecular interactions, SEM for surface and cross-sectional morphology, X-ray diffraction for degree of crystallinity and intersegmental spacing, TGA for thermal stability and sorption studies to understand polymer-liquid affinity.

Recovery of carboxylic acids such as lactic acid, acetic acid and tartaric acid from aqueous solutions besides levulinic acid from an industrial effluent was carried out by reactive extraction through membrane contactors using tri-*n*-octylamine (TOA) as extracting agent. Performance of indigenously synthesized pristine, blend and mixed matrix membranes on the rate of acid extraction was evaluated by varying concentrations of acid in water and TOA in the diluent 1-octanol, respectively. H-beta zeolite incorporated PVDF mixed matrix membrane exhibited maximum extraction efficiency of 34.36% during isolation of lactic acid whereas a maximum efficiency of 29% was obtained for acetic acid extraction using commercial polytetrafluoroethylene (PTFE) membrane. Performance of ultraporous PVDF/PU blend and microporous PVC membranes containing different additives such as polyethylene glycol, LiCl₂, ZnCl₂ and glycerol was investigated for extraction efficiency of tartaric acid and levulinic acid. Glycerol additive facilitated maximum acid extraction efficiency due to larger pore size created by its dissolution in water during membrane preparation. ED was studied for separation of tartaric acid from crude tamarind pulp mixture

and recovery of levulinic acid from industrial effluent under varying voltage. Results indicated a considerable reduction in diluate conductivity and decrease in concentrate pH ensuring transfer of acid into the concentrate chambers. Further, the tartaric acid enriched stream from ED system was concentrated in an economical way using RO.

Dehydration of glycerol, diethylene glycol (DEG), acetamide catalyst, lactic acid, separation of hydrochloric acid (HCl) from chloroalkali industrial effluent and recovery of hexane solvent from sunflower oil miscella were performed using MD technique. Effect of feed composition, permeate pressure and feed temperature on MD performance was evaluated. Ultraporous PS membrane exhibited a water flux in the range of 0.56–0.02 kg/m²h with infinite selectivity for feed glycerol concentration of 10 to 90% (v/v). Dehydration of DEG and lactic acid was carried out using ultraporous polyvinyl chloride (PVC) and ZSM-5/PVC mixed matrix membranes, respectively. An increment in feed DEG concentration from 61% to 90% (v/v) reduced the flux from 0.35 to 0.09 kg/m²h with $\leq 0.7\%$ DEG loss in permeate. On the other hand, ZSM-5/PVC mixed matrix membrane exhibited higher flux of 0.61 kg/m²h with $< 1.3\%$ lactic acid loss. PVDF membrane provided a high flux of 0.32 kg/m²h with $< 1.2\%$ acetamide loss during concentration of this catalyst from 30% to 70% (w/v). Microporous PTFE membrane exhibited commercial potential for recovery of impurity free HCl (33 wt %) at a low operating cost of Rs. 2.28/- per L. PVC membrane enabled separation of hexane solvent of $> 95\%$ purity. A mathematical model was developed for MD process to optimize operating parameters.

Fructose sugar solution was concentrated using indigenously synthesized hydrophilized thin film composite (TFC) polyamide membrane by FO process. Effect of operating parameters such as draw solution (NaCl) concentration, cross flow velocity and temperature on FO performance was evaluated. Increase in NaCl concentration and cross-flow velocity resulted in enhanced water flux. A combined model based on molecular dynamics (MD) and computational fluid dynamics (CFD) was developed to predict FO system performance for different parametric conditions.

The proposed research study infers that the indigenously developed membranes exhibit considerable potential for recovery and recycle of valuable chemical entities by offering safe, eco-friendly and economical routes as against conventional evaporation, liquid-liquid extraction or distillation.