



Study of the supported liquid membrane for the estimation of the synergistic effects of influential parameters on its stability



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Abbreviations:

LM, liquid membrane
SLM, supported liquid membrane
TBP, tributyl phosphate
PVDF, polyvinylidene fluoride
HLB, hydrophilic–lipophilic balance
W/O, water in oil
O/W, oil in water

ABSTRACT

Supported liquid membrane (hereafter referred as SLM) is considered as an impressive alternative and the improved version of the liquid–liquid extraction (hereafter referred as LLE) process owing to some its inherent advantages over the traditional LLE processes. The most crucial problem associated with the SLM technique is its stability *i.e.* the ability to operate for prolonged time without compromising the quality of the separation. The prevalent reasons for the instability are investigated experimentally to reach to the solutions. In this case study, the extent of stability has been quantified by measuring the performance of SLM in terms of percentage of extraction and recovery of phenol while it is transported across the membrane in a series of runs. The percentage of transportation of phenol at various time scales during the transportation is measured in order to quantify the stability. Initially, the components of the liquid membrane (hereafter referred as LM) *e.g.* solvent–carrier combination are selected based on their physical properties that yield best transportation and stability as well. Further, the components and concentrations of feed phase and LM are optimized for the more stable SLM system. The addition of electrolyte in the aqueous phases in order to enhance the interfacial tension between LM and either of the aqueous phases increases the stability up to 120 h. The addition of surfactant in LM reduces the loss of LM from pores of the membrane support and provides the SLM stability up to 144 h. Polymeric gel formation on the outer surface of SLM (LM into the micro-pores of membrane support) increases the stability of SLM up to 168 h with the reduction of recovery of phenol after 120 h only up to 10.3%.

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1. Introduction

Liquid membrane techniques are used for separation of trace quantity of solute from a solution, *e.g.*, the removal of metal ions from the wastewater [1–4]. The other applications of LM technique include but are not limited to removal of organics from wastewater [5–8], recovery of bio-actives compounds from plants [9–11], separation of biologically important products such as various antibiotics from the fermentation broth, and bioactive catechin from tea leaves [12–14]. Among the various configurations of the LM, the SLM is the most widely applicable technique in commercial scale owing to certain advantages including requirement of very less amount of solvent, requirement of low footprint, low capital investment and higher flux of the solute [10,11]. Recently several researchers [15–17] have exploited the advantage of hollow fiber

supported liquid membrane (hereafter referred as HF-SLM) that provides more interfacial area to mass transfer per unit volume of the membrane module for the separation of trace amount of hazardous heavy metals from wastewater. The LM is immobilized into the pores of the polymeric solid support in order to provide the mechanical strength [10,11]. However, the instability of the SLM *i.e.* the ability to work for substantially longer time is most serious problem that restricts the said technique to be viable in the industrial applications.

Neplenbroek et al. [18] identified emulsification as the cause of instability that occurs as a consequence of application of shear forces originated from the stirring and/or flow of aqueous phases. The flow of aqueous feed and the stripping phases with lower salt concentration along the membrane surface causes loss of both solvent and carrier from the pores of the support of SLM. A significant correlation between the instability of SLM and the stability of emulsion is observed. Hence, a stable SLM requires a favorable condition in which formation of relatively stable emulsion is prevented. A gel network of polymer coating was provided on the outside of the pores of the membrane in such a

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way that the permeability was not reduced much while the stability increased substantially [19]. Neplenbroek et al. [20] also reported the behavior of instability of several SLMs for a system in which nitrate ions were transported. The stability of membrane depends largely upon the type of solvent and the molecular structure of the carrier [21]. Zha et al. [22] investigated certain mechanisms of instability of SLMs too. It was observed that any factor facilitating the formation of stable oil-in-water (O/W) emulsion, such as addition of surfactant of higher HLB (hydrophilic-lipophilic balance) value in the LM, low electrolyte concentration in the aqueous phases and high stirring speed of the aqueous phases, etc. are favorable to the loss of LM from the pores of the support. It can be shown by thermodynamic analyses, that emulsification can be realized with the supply of a suitable energy. The energy in the SLM processes is applied by forced flow of aqueous solutions i.e. the hydrodynamic instability of the interfaces. Yang and Fane [23] investigated the stability and performance of SLM based on Celgard and Accurel membranes as supports and a commercial extractant LIX 984N as a carrier for transport of copper. They also reported that the surface shear forces due to stirring of aqueous phases are main reason for loss of LM leading to the instability of SLMs.

Zha et al. [22,24] reported another observation on the stability of SLMs. They prepared their membranes with dry outer surfaces, i.e. a surface which is not wet with organic phase (LM). These membranes were more stable than conventional SLM prepared with wet external surfaces. The SLM with a 'dry' surface yielded a similar initial flux as observed for the SLM with 'wet' surface, however during the prolonged operation (for 100 h) the flux of solute through SLM with 'dry' surface was found to be 2–4 times higher than the flux through SLM with 'wet' surface. Over a 50 h period the loss of LM was found ~10% for 'dry' SLM as compared to about 50% for 'wet' SLM. The difference is ascribed to the loss of membrane by emulsion formation at each of the aqueous-organic interfaces which would be greater for the 'wet' SLM with a continuous liquid film over the surface of the support.

Kemperman et al. [25] verified method of stabilizing SLM by the application of polymeric top layer to the surface of SLM which was investigated by Neplenbroek et al. [19]. The modified micro-porous membrane was tested on selective nitrate transportation. They concluded that most applied top layers did not hinder the transport of nitrate ions. Best results were obtained when piperazine (PIPA) and trimesoyl chloride (TMCI) were used as monomers of polymeric gel. They got constant nitrate flux at $18 \times 10^{-10} \text{ mol cm}^{-2} \text{ s}^{-1}$ for 350 h of simulated operation for Accurel polypropylene supports with PIPA/TMCI top layer. For uncoated supported liquid membranes (SLMs), the flux decreased within one day from $18 \times 10^{-10} \text{ mol cm}^{-2} \text{ s}^{-1}$ to negligible amount.

Zheng et al. [26] studied the instability mechanisms of SLM for phenol transport. Celgard 2500 and tributyl phosphate (TBP) were used as support and carrier, respectively. The instability was measured by electrochemical impedance spectroscopy. Various researchers [22–24] reported the experimental findings to improve the stability. Zidi et al. [7] studied complexing properties of a carrier, TBP for liquid-liquid extraction (LLE) as well as SLM based facilitated transportation of phenol. The best performance of transportation process involved with feed phase pH of 2 and stripping solution of 0.2 M NaOH. The efficiency of transport decreased with time during five days of continuous operation. The optimum transportation of phenol was obtained using 20% TBP (v/v) in kerosene as LM. The LM was stable for 3 days of continuous running with 17% reduction in the flux of phenol. Fortunato et al. [27] studied the use of room temperature ionic liquids (RTILs) in SLMs. RTILs have very low vapor pressure and are able to solvate a variety of organic and inorganic species. However, ionic liquids are very expensive. Moreover, due to its higher viscosity, the flux of

solute through ionic liquid is very less [28]. Hence, the employment of conventional solvent-carrier combination in SLM demands the detail investigation.

No single instability mechanism dominates during long term permeation. The instability of long term operation involves a complex and varied interaction of a number of factors, including surface shear forces, Marangoni effects, changes to membrane morphology, Bernard instabilities and membrane preparation protocols. In this experimental work, we study the synergistic effects of parameters for greater stability of SLMs. Initially the solvent was selected for the LM. Thereafter, various conditions of aqueous feed phase and LM are re-optimized for the greater stability. Finally, the SLM was coated with a polymeric layer to minimize loss of LM and reduction of emulsification. The composition and the thickness of the polymeric layer are optimized for greater stability with lesser reduction of permeability of phenol.

2. Materials and methods

2.1. Chemical and reagents

All reagents used in this experiment are of GR (Guaranteed Reagent) grade. Aqueous solutions are prepared by using Milli-Q de-ionized water. Chemicals such as dichloroethane, sodium hydroxide, tributyl phosphate (TBP), kerosene, 1-decanol, phenol and poly methyl methacrylate alcohol (PMMA) are procured from Merck (India). The surfactants such as Span 60, Span 65 and Tween 80 are obtained from Sigma Aldrich. Support membrane, PVDF (thickness = 127 μm , pore diameter = 0.2 μm , porosity = 0.45 and tortuosity = 3.4) is procured from Pall Life Science. The value of the tortuosity (τ) was calculated as:

$$\tau = \frac{1 + V_p}{1 - V_p} \quad (1)$$

where $V_p = (1 - \varepsilon)$ is the volume fraction of the polymeric support and ε is porosity of the membrane [29].

2.2. SLM setup

The SLM set up (permeation cell) used in this experiment has also been used and described elsewhere [10]. Two separated compartments are filled with aqueous feed and strip phases, each 140 ml. The organic phase (*a.k.a.* the LM) was impregnated and immobilized inside the pores of the support to form the desired SLM. The immobilization of organic phase inside the pores of support membrane was accomplished by dipping the support membrane into the organic phase at atmospheric pressure for 24 h. The support was then taken out of the organic phase and the excess liquid was removed from the surface of the support by good quality tissue paper. It was thereby placed between two flanges of pipes that connect the two compartments. LM immobilized in the micro-pores of the porous support acts as a medium of solute transport from feed phase to stripping phase. The effective membrane surface area for transportation of solute is 11.2 cm^2 . The solutions are continuously stirred by motor driven stirrers to minimize the concentration polarization of solute at interfaces.

2.3. Experimental procedure

Stock solution of phenol is prepared by dissolving 1 g of phenol ($\text{C}_6\text{H}_5\text{OH}$) in 1000 mL of Milli-Q de-ionized water. The feed phase is prepared from the stock solution by dilution with deionized water up to the desired concentration. Hydrochloric acid (HCl) and NaOH are used in order to maintain the desired pH of feed phase. LM is

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