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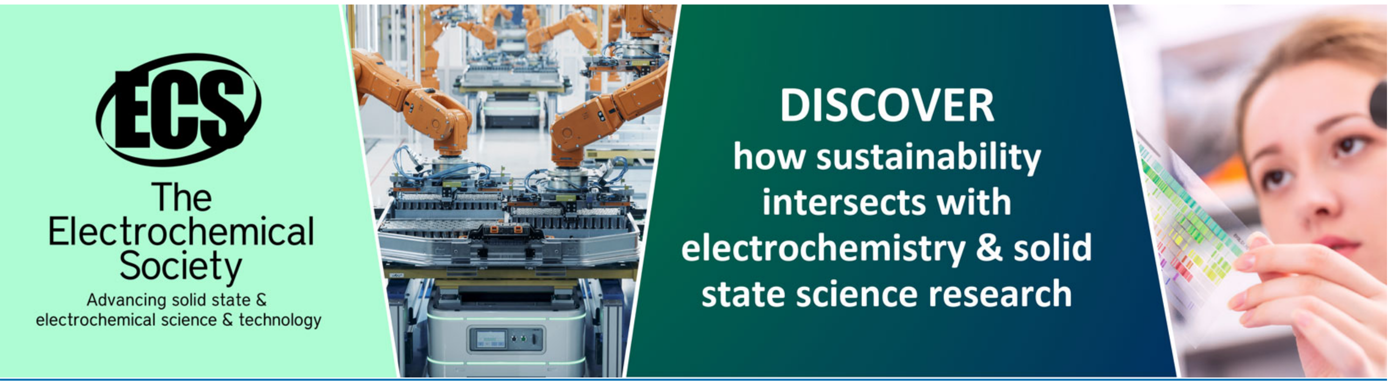

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# Membrane separation for non-aqueous solution

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**Abstract**. Membrane technology has been widely used in a number of applications competing with conventional technologies in various ways. Despite the enormous applications, they are mainly used for the aqueous system. The use of membrane-based processes in a non-aqueous system is an emerging area. This is because developed membranes are still limited in separations involving aqueous solution which show several drawbacks when implemented in a non-aqueous system. The purpose of this paper is to provide a review of the current application of membrane processes in non-aqueous solutions, such as mineral oil treatment, vegetable oil processing, and organic solvent recovery. Developments of advanced membrane materials for the non-aqueous solutions such as super-hydrophobic and organic solvent resistant membranes are reviewed. In addition, challenges and future outlook of membrane separation for the non-aqueous solution are discussed.

## 1. Introduction

The rapid growth of membrane applications in water treatment [1-8], wastewater treatment [9-12], gas separation [13, 14], food processing and biotechnology [15-20], medical and chemical processing [11, 21-26], energy conversion [27], etc., is due to their attractive features. The membrane processes require a relatively lower energy consumption, are typically lower operating cost, have a higher selectivity, and can be operated at moderate conditions which make membrane an interesting alternative to conventional technologies in various processes [28-32]. Most of the commercial membranes are applied in the aqueous solution processing [1, 2, 30, 33]. Membrane is also potential to be applied in non-aqueous system, such as polystyrene treatment [34], aromatic solvents [35], production of an alkali-alkoxide from alcohol and salt [36], micro emulsion processing for drug delivery [37], purification of fuel [38, 39], purification of CdSe-CdS-ZnS, organic solvent regeneration and valuable component [40-43], ethanol-alkanes solutions [44-47], used oil processing [48-52], and vegetable oils processing [53-69]. However, the commercial applications are still limited. Most of the membranes were developed for aqueous system. For non-aqueous system they show several drawbacks such as low stability and low flux. Therefore, the development of membrane materials is still an emerging subject for a non-aqueous solution [52].

The purpose of this paper is to provide a comprehensive review of the membrane technology for non-aqueous solution processing. Recent developments of membrane application in non-aqueous system including mineral oil treatment, vegetable oil processing, and separation of solvent from the organic mixture are reviewed. The developments of new membrane material, such as super-hydrophobic and organic solvent resistant membranes are overviewed. Finally, the challenges and future outlook of membrane separation for the non-aqueous solution are discussed.

## 2. Application of membrane in non-aqueous solution

In the following sections, membrane processes for the non-aqueous solutions will be presented and discussed including the prospect and challenges of application in non-aqueous solutions.

### *2.1 Vegetable oil processing*
In vegetable oil processing, a membrane could be applied in the stages of refining (Table 1) namely degumming, de-acidification, de-colorization, solvent recovery, metal removal, deodorizing, hydrolysis of fats and oils, synthesis of structured lipids, the concentration of certain compounds, and the separation of emulsions [70]. Several steps could be simplified into a single process using a membrane. For example, removal of undesirable compounds from the vegetable oil uses a single step without the addition of filter aids [71]. The removal of the damaging substance from vegetable oil caused an insignificant change of oil properties, minimized effluents, reduced the energy consumption, and preserved important nutritional components [70].

**Table 1**. Membrane processes in vegetable oil processing

| Processes | Membrane processes | Remark | Ref. |
|---|---|---|---|
| Degumming | MF | Reduction of phosphorus, FFA and water | [66, 68] |
| | UF (ceramics, PVDF, PES, PSF); MF | Separate oil from the oil-hexane mixture. Permeate contents: hexane, triglycerides, FFAs and other small molecules; almost all the phospholipids are retained. | [58, 59, 62, 63, 65, 67, 69, 72-75] |
| | UF ceramics | Degumming & production of soy lecithin | [60] |
| De-acidification | UF, polymer (PAN, PES, PS), ceramics zirconia | Addition of an alkali followed by membrane filtration or by following an indirect route of selective solvent extraction of FFA followed by membrane separation. | [59, 61, 63, 76, 77] |
| De-colorization | Polymeric composite NTGS- 2100 & NTGS-1100 (active layer) PI, PSF (support layers) Nitto Denko Corp. Jpn. | Removal of chlorophyll up to 96% and 72% from the undiluted and oil-hexane (50wt%) solutions, | [78] |
| | MF, NF, RO | A ‘membrane bleaching’ process could save cost | [59, 65] |
| Solvent recovery | RO, NF, UF | pre-concentrate the miscella before evaporation; reduce the energy consumption | [59, 65] |
| | NF, polymeric (NF-99-HF, Alfa Laval) | Separation of FFA retention, 70% | [56, 79] |
| | NF, PVDF as a support & PDMS/CA as coating layer | Removal of hexane and FFA from crude soybean oil–hexane mixture. The PVDF-12% siloxane composite NF membrane reached the best results | [80] |

The applications of membrane for degumming of the oil-solvent mixture were reported, i.e. nonporous polymeric membrane [81], inorganic solvent NF [82], ultrafiltration [72-75, 83, 84], and microfiltration [85]. Degumming of crude vegetable oil can be conducted directly using a membrane without the addition of solvent [70]. De Moura et al. [74] used UF for degumming of soybean oil, and the characteristics of the oil, i.e. phospholipids content, viscosity, color, FFAs and tocopherols were analyzed. The study found that the higher oil viscosity resulted in a lower flux. The corn oil processing in alumina multichannel ceramic MF showed that phosphorus rejection was significantly affected by trans-membrane pressure (TMP) [73]. The increasing TMP increase the phosphorus rejection, significantly reduce color and wax, and also remove 93.5% of phospholipids. The minor component e.g. tocopherols and tocotrienols were successfully preserved [74]. More than 99.7% of the phospholipids were rejected by a ceramic membrane [72], while almost complete rejection of oil was obtained using a cellulosic UF membrane in a dead-end filtration [86].

Fatty acid removal is an important step in vegetable oil processing, but the application of membrane still remains a great challenge. Bhosle et al. [79] reported that in the undiluted system, the solubility of triacylglycerols in oleic-acid determines the selectivity. The NF membrane exhibited good selectivity for oleic-acid and excellent solvent stability for acetone. However, the significant improvements are still required. De-acidification using NF composite membrane showed that PVDF materials were physically-resistant to solvent as well as a higher flux and oil recovery [80]. Removal of acid by forming a dispersion of saponified fatty acids/water/isopropanol in oil through a PAN UF membrane was investigated [77]. The study concluded that the flux reached the highest value of 95 $L.m^{-2}.h^{-1}.bar^{-1}$ at the fatty acid/water/isopropanol ratio of 1:6.5:3 (v/v). Purwasamita et al. [28] used hollow fiber poly-sulfone UF membrane contactor to reduce the acidity of crude palm oil (CPO). They found that membrane contactor was highly potential for de-acidification of CPO because of the higher surface contact provided by the membrane. Fractionation of fatty acid using UF membrane could decrease the acid chain while it increased the mass transfer coefficient and distribution coefficient [76].

Decolorization of vegetable oil using membrane processes has been conducted by several works. Reddy et al. [78] used a batch membrane cell using two polymeric composite membranes (NTGS-2100 and NTGS-1100) and one polyethylene MF (PE-30) to remove chlorophyll from sunflower oil and crude soybean oil. They found that the removal of chlorophyll from the undiluted and oil-hexane (50%-w) solutions using NTGS-2100 membrane achieved 96% and 72%, respectively. NTGS-1100 membrane exhibited higher flux rather than NTGS-2100 but resulted in a lower rejection [78]. Subramanian et al. [87] reported that the color compounds and oxidation products could be effectively reduced by using hydrophobic nonporous membranes while the beneficial compounds could be retained.

In the vegetable oil processing, the recovery of solvent from oil extraction (micelle) is the most critical steps regarding the economic, environmental, and safety reasons [70]. The controlling parameters of the permeation rate through membranes were solvent solubility parameter, dielectric constant (polarity), surface tension, and solvent viscosity [88]. The membranes processes like UF and RO have been used to recover the solvent from the micelle [89]. The important characteristics of membrane for solvent recovery are their solvents-resistance and the rejection performance [90]. The flux of permeate depends on the applied TMP, temperature, feed concentration, and cross-flow velocity. Higher pressures, temperatures, and cross-flow velocities yielded better permeate flux.

### *2.2 Waste oil processing.*

Several types of membrane materials have been used to remove contaminants and impurities in waste oil, e.g. poly-ethersulphone, poly-vinylidenefluoride, poly-acrylonitrile [91], polyimide [92], and ceramic membranes [31, 32, 49-51]. Inorganic membranes have a higher rejection of contaminants and a better ash removal [93]. The properties of waste oil significantly affect the filtration performance [94], e.g. a higher flux of permeate obtained at a lower oil viscosity [95]. The usage of pressurized supercritical $CO_2$ as viscosity reduction will improve the permeability of membrane [32, 96, 97]. The performance of membrane process is also influenced by the filtration mode. For example, the dead-end filtration mode causes a cake formation onto the membrane surface, drastically flux reduction, and a low separation performance. The larger pore size results in a higher flux but has a lower rejection. Therefore, the flux to quality ratio was used as the indicator of filtration performance [49]. The light hydrocarbon in waste oil could be recovered using polyimide membrane from the lube filtrates at purity better than 99%. Combining a membrane system with a conventional process increases the energy efficiency and the capacity of solvent recovery [52, 98]. In used frying oil treatments, the removal of degradation materials using membrane processes will improve the life of usage [99, 100]. Lai and Smith [101] used ceramic MF membrane to remove asphaltene from heavy oil. They found that the pore-restriction mechanism initiates fouling, following by gel layer formation on the membrane surface. Consequently, the pore size has no significant impact on flux.

### *2.3 Other chemical processing*

Membrane have been used in chemical processing like crude biodiesel treatment for catalyst removal [102] and glycerol reduction [38]. The ceramic MF produced a high-quality biodiesel by removing the residual catalyst. The retention of the catalyst was 93.64% and potassium was reduced from 8.3 mg/L to 0.3 mg/L [102]. Torres et al. [38] evaluated two UF membranes (PVDF and PSF) to reduce glycerol content from crude biodiesel. This study revealed that the membrane material is an important factor determining the separation performance. The PVDF membrane exhibited a higher stability and lower tendency of fouling than PSF membrane. Application of UF in the clarification of glycerin-rich solutions in the oleo-chemical industry demonstrated a declining flux because of fouling onto the membrane surfaces [83]. Similar to aqueous solutions, membrane rejection increased with applied pressure [83]. In solvent exchange, Livingston et al. [82] reported that NF exhibited a more effective process compared to distillation in swapping a high boiling point solvent to a lower one. By means of NF, the two distinct types of homogeneous catalysts, namely phase transfer catalysts and organometallic catalysts, can be separated from the main reaction products. Tsuru et al. [41] used porous silica-zirconia NF membrane for non-aqueous solution of ethylene glycol, diethylene glycol, triethylene glycol, and polyethylene glycol (PEG) in ethanol and methanol as solvent. They also concluded that the rejection strongly depended on the type of solvent used [43].

## 3. The development of membrane materials for non-aqueous solution

During the filtration of non-aqueous solution, the solvent significantly influences the retention of solute in the organic solvents. Yang et al. [43] reported that the lower retention value might be because of another transport mechanism in an organic solvent, not due to membrane deterioration. The flow of organic solvents through ceramic-oxide membranes was influenced by interfacial interactions [40]. The flux was affected by surface tension and viscosity of the solvent, furthermore the flux of solvents in the mixture was found to be highly non-linear [103]. In the case of oil filtration, flux is the main challenge due to the high viscosity of the oil. Improving membrane-oil interaction by modification or fabrication of hydrophobic or even super-hydrophobic membrane may be used to solve the problems. In the following sections, preparation of special membrane for non-aqueous systems including solvent-resistant and super-hydrophobic membrane will be presented.

### *3.1 Solvent-resistant membrane*

Membranes can be classified into six categories base on their resistance to the solvent and the stability under extreme operation, i.e. based on phosphazene, imide, methacrylate, inorganic, liquid crystalline and cross-linked polymers [33]. The presence of certain groups like imide in the backbone required for the solvent-resistant properties of polymers [52]. A polyimide membrane manufactured from a co-polymer of diaminophenylindane with benzophenone tetra-carboxylic di-anhydride has an excellent chemical resistance and economically viable flux and rejection characteristics for the separation of light hydrocarbon solvents from lube oil filtrates. Ochoa et al. [75] used three polymeric materials (PVDF, PES, and PSF) and concluded that PVDF has the higher stability to hexane among others. A significant decrease of flux at the beginning of filtration was due to the concentration polarization and internal fouling. Fabrication of silica-zirconia porous membranes by a sol-gel process were conducted to produce organic-inorganic hybrid membranes in the nanometer scale [41, 46]. Alicieo et al. [84] used ceramic tubular membrane (0.01μm) and a polysulfone hollow fiber membrane (100kDa) and found that the polysulfone membrane gives a higher flux than the ceramic membrane. The clarification of glycerin-rich solutions in the oleo-chemical industry using PVDF and PES membrane tend to be seriously fouled and PES exhibited a higher fouling than PVDF [83].

Tsuru et al. [47] modified the membrane with trimethylchlorosilane (TMS) to increase the performance as showed in a relatively constant permeability. The filtration of ethanol solutions with alkanes (hexane, decane, tetradecane) and alcohols (hexanol, octanol, decanol) as solutes showed that the rejection of alcohols decreased while alkanes rejection were nearly unchanged regardless of the temperatures. Cliff [39] used composite membranes consisted of a PDMS (polydimethylsiloxane) active layer bonded to a commercially available support layer of PAN. Silva [104] used integrally asymmetric polyimide and polyetherimide materials to develop organic solvent NF (OSN) membranes. The degradation of polyimide membrane in polystyrene-ethyl acetate solution shows that flow-induced deformation of the polystyrene chains highly affects the membrane retention [34]. Cliff [39] reported that membrane composition and the extent of polymer swelling were found to be the two key factors which influenced the solvent flux and the solute rejection. Polymer swelling was the most affected parameter when using a solvent with a solubility parameter close to the polymers. Therefore, development of membrane materials that suitable for those applications is required.

### *3.2 Super-hydrophobic membrane*

The performances of membranes filtration are varied as a function of the feed, the contaminations, the temperature, working pressure, and other factors [31]. The previous studies reported that the polymer swelling was the most affected when using solvents with a solubility parameter close to the polymers [39], the formation of micro-cracks at the bend result in the loss of functionality [34], and several membranes tend to be seriously fouled [83]. The separation performance could be improved by developing the membrane materials for non-aqueous system, such as PES, sulfonated poly-sulfone (SPS), PAN, PP, PE, PVDF or PTFE [105, 106].

Generally, the hydrophobic membranes have higher tendency to be fouled in aqueous systems and tend to be wetted in non-aqueous applications. The increasing of hydrophobicity of the membranes materials improves permeability and increases the wetting resistance. In the non-aqueous processing such as vegetable oil filtration, solvent recovery, and waste oil treatments, increasing the hydrophobicity of the membrane is one of promising strategy to improve membrane performance. A super-hydrophobic membrane has a value of water contact angle higher than $150^o$, while contact angle hysteresis and sliding angle are smaller than $10^o$ [107-109]. The development of super-hydrophobic membrane is an attractive strategy for the non-aqueous solution processing. The super-hydrophobic membrane has anti-wetting properties which resist the aqueous solution to flow through the membrane pores. The super-hydrophobic membranes also have antifouling properties which are result in a high permeability and high separation ability to the non-aqueous solution. In some cases, super-hydrophobic membranes also exhibit super-oleophilic characteristic, performing a high efficiency in non-aqueous separation. These advantages will spread out the applications of super-hydrophobic membrane especially for the non-aqueous processing [110, 111].

## 4. Summary and Future Outlook

Even though membrane processes have been used in almost industrial sectors, most of the commercial membranes are applied in the aqueous solution treatment. Meanwhile, in the non-aqueous solution, their applications are still limited. For non-aqueous processing, the stable material is needed, because membrane deterioration may occur during the operation which leads to a high operating cost. In addition, in case of oil filtration, membrane-oil interaction can be improved by fabricating a more hydrophobic or oleophilic membrane. Numerous studies on preparation of solvent resistant and super-hydrophobic membranes have been reported. Solvent resistant membranes have been prepared generally based on phosphazene, imide, methacrylate, inorganic, liquid crystalline, and crosslinked polymers as the backbone to support the solvent-resistant properties of polymers. Meanwhile, membranes with high hydrophobicity or super-hydrophobic are generally produced from polymers which have the low energy surfaces such as PP, PE, PVDF, or PTFE. In addition, the super-hydrophobic membrane can be obtained by modifying membrane surface roughness and coating hydrophobic material on the membrane surface. With those advanced membranes, membrane applications can be spread out for non-aqueous processing in the future.

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