ABSTRACT

One of the most important challenges for modern-day chemical engineers is separation and purification of products. Separation of one or more components from a complex mixture in order to achieve the goals of enrichment/concentration/purification/refining/isolation of any desired product is the requirement for many operations even in food and vegetable oil industries. Membrane separation technique is one of new area of separation science and has become common engineering tool. Indian food and vegetable oil industries are looking forward in meeting the standards of global competitors. On one hand, they are striving to get the better quality products, to reduce the production costs and to utilize the resources completely. To achieve these goals, use of better separation techniques like membrane separation technique plays a vital role. On the other hand, as environmental protection agencies are becoming more and more stringent, industries are looking forward for process that can treat their waste streams. In this study, attempts were made to apply membrane separation techniques in handling different wastewater samples.

The first two chapters of the thesis deals with general introduction and literature survey. The work done is described is in next four major chapters.

In Chapter 3, the details of the concentration of gallic acid from green and black tea leaves extracts using membrane separation techniques are described.

Gallic acid is widely used in food and pharmaceutical industries such as in synthesis of propyl gallate, trimethoprim etc. This is reported to have no effect on healthy cells. This is also used for treating psoriasis and external haemorrhoids. This compound is found either in free form or as a part of the tannins in various natural products. Gallic acids can be extracted from gall nuts, green or black tea leaves, tara pods, grape seeds, sumac, oak bark etc. using hot water and alcoholic solvents like ethanol and methanol. These are obtained in ppm level in the extract and generally concentrated by evaporation/lyophilization and obviously require huge energy. This makes the process commercially less competitive. In this present investigation, a membrane based approach was followed to concentrate the gallic acid extract up to a certain extent and thus to reduce the volume and energy required to get gallic acid concentrate. The reverse osmosis membranes were first tested for their resistivity to alcohols and then for suitability for concentrating gallic acid solution. Gallic acid was concentrated from ppm level to percentage level using alcohol resistant BW-30 and SW-30HR membranes. This technique may help in designing a process for commercial production of gallic acid from various natural sources.

In Chapter 4, the details of separation of free fatty acids (FFA) from their alcoholic solutions by using membrane separation technique are described. Liquid Liquid Extraction (LLE) using different alcohols is used for selective extraction of free fatty acids from oils. Since the latent heat of vaporization is

higher in case of alcohols, the energy required to separate free fatty acids from alcohols by evaporation is high and hence not preferred by the industry. Membrane separation may help in solving this problem, as membrane separation techniques do not require any phase change and hence require less energy for separation. The major hurdle for developing a membrane based process for separation of fatty acids from alcoholic solution is to get alcohol-resistant membranes for first instance, and then get the suitable MWCO of the membranes that can separate fatty acids from alcohol. In this study, attempts were made to check the suitability of four different commercially available polymeric membranes for separation of fatty acids from the alcohol solution. Initially compatibilities of different membranes in different alcohols were checked. Later, solutions of oleic acids in methanol and ethanol were taken as model mixtures. The best separation characteristics were shown by NF-90, a nanofiltration membrane. More than 90% rejection of fatty acids was achieved with methanol as solvent. In some cases, effects of addition of water on separation/flux characteristics were also evaluated. The addition of 5 wt% water in ethanol significantly improved the flux and the rejection characteristics. The results may help in selecting a suitable membrane for separation of fatty acids from its solution in alcohol.

The chapter 5 and 6 describe the work carried out wastewater treatment for vegetable oil based industries.

Biodiesel, the most attractive alternate to diesel fuel, is made from renewable biological sources such as vegetable oils and animal fats. It has gained much importance during the last few decades for the uncertainty in crude price and its supply. The process of preparation of biodiesel generates reasonably high quantities of wastewater contaminated with primarily salts and fats, oils and greases (FOG). The contaminants in biodiesel wastewater generated from non-edible oils like in India are more compared to edible oil based biodiesel industries such as in U.S.A and Europe. This requires elaborate treatment before discharge. In the present study described in Chapter 5, the wastewater discharged from a non-edible oil based biodiesel fuel production plant was chosen. In pretreatment studies, alum, poly aluminum chloride, ferric chloride and chitosan were used as coagulants. The results showed that best coagulation could be achieved by using alum as coagulant. The coagulant dosages, stirring time and settling time were studied. The pretreated water was first passed through microfiltration membrane and then nanofiltration (NF-500) and reverse osmosis membranes (TW-30-1812). All the major contaminants could be separated significantly by the combinations of these membrane separation techniques. The quality of processed water after membrane separation was found to be suitable for reuse to process stream or for various other purposes.

Wastewaters generated in typical perfumery chemicals were also treated using membrane separation techniques. The results are described in Chapter 6.

Heptaldehyde is a very important castor oil based perfumery chemical. This is produced by pyrolyzing castor methyl esters (CME). In this process large volumes of water are required and the wastewater produced is contaminated with heptaldehyde and other byproducts. The presence of heptaldehyde — a malodorous compound, causes serious problems to the nearby locality if the contaminated water is discharged without proper treatment. In the present investigation, a process was developed for the treatment of aldehyde contaminated perfumery chemicals wastewater. As the common coagulants such as alum, ferric chloride, alginic acid and chitosan either alone or in combination with powdered activated charcoal (PAC) as adsorbent did not have any desired effect, a novel pretreatment process was developed using sodium borohydride as reducing agent. This was followed by membrane processing. The process parameters for using both the reducing agent and PAC as adsorbent were studied keeping in mind the removal of mal-odor of the wastewater samples. The pretreated water was first filtered using a micron filter and then processed through a reverse osmosis membrane. The final treated water was clear, odorless and the quality of the water was found to be suitable for reuse.

Thus, in this study, feasibility of using different membrane separation techniques for processing and wastewater treatment of various food and vegetable oil based industries were studied and evaluated.

- A process was developed to concentrate antioxidants like gallic acid from their crude natural extracts using RO membranes.
- Various membranes were screened for solvent resistivities and a process was developed to separate fatty acids from alcoholic solutions.
- Feasibility studies were conducted to treat biodiesel wastewater using different MF/NF/RO membranes and the treated water was found to have quality to be reused.
- A novel process was developed to treat wastewater generated in castor oil based perfumery chemicals industries. This will help in solving a major problem pertaining to this particular industry.

A general conclusion is given in Chapter 7. The thesis was appended with the references cited in Chapter 8.