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Separation of Sunflower Oil-Water Emulsions Using Polyether Sulfone Cross Linked Polyether Sulfone Polyacrylonitrile Polysulphone Membranes

V. Nagamani,*
Department of Chemical Engineering
University College of Technology
Osmania University, Hyderabad
500007, T. S. India

R. Shyamsunder ,
Department of Chemical Engineering
University College of Technology
Osmania University, Hyderabad
500007, T. S. India

V. Lakshman
Department of Chemical Engineering
University College of Technology
Osmania University, Hyderabad
500007, T.S.India

Abstract: **The crude oil refinery units and the vegetable oil industries produces large volumes of aqueous effluents in the form of o/w and w/o emulsions. Aqueous effluents will contain high value added products which are required to be recovered. This research is aimed to treat the oily waste water by using ultra filtration polymeric membranes consisting of polyether sulfone (PES), cross linked polyether sulfone (CPES), polyacrylonitrile (PAN), polysulphone (PS) membranes. The oily waste water samples were taken from sunflower oil-water emulsions and characterized in terms of Total Dissolved solids (TDS), Total suspended solids (TSS), Conductivity, pH, Temparature and Chemical oxygen demand (COD).Meanwhile membrane samples were characterized by permeating flux and solute rejection at different pressures (3, 5, 7 bar) and feed concentrations (1000, 5000, 20,000ppm).The experimental results obtained in the present work shows flux 32 L/m^{2}h,the permeate water obtain was having almost 99% total dissolved solids and total suspended solids and 99.9% of chemical oxygen demand.**

Keywords: Aqueous effluents, Chemical oxygen demand, Flux, polymeric membranes, Rejection, Total dissolved solids.

1. INTRODUCTION

The hasty industrial growth, such as petroleum, Food. Metalurgical, Pharma and textile industries, has led to the large production of oily wastewater. These oily wastewater should not be discharge without prefer treatment in order to prevent negative impacts on the environment. Oily waste water pollution is mainly manifested in the following aspects: Affecting drinking water, endangering human health, atmospheric pollution, affecting crop production, soil degradation.

The main kinds of oily wastewater are floating oil, unstable oil/water (O/W) emulsion and stable O/W emulsion. Floating oil or unstable o/w emulsions can be easily removed by using conventional separation process such as chemical, mechanical and thermal methods. The chemical methods involve chemical pretreatment of emulsified oil to destabilize the emulsion followed by gravity separation, the mechanical and thermal methods primarily based on the phenomenon of gravitational and thermal emulsion breaking. However, the conventional methods for treatment of oil emulsions have several disadvantages, such as a low efficiency, high maintenance and operation cost, large space requirement and recontamination problems. Furthermore, these conventional processes are not effective enough for removing stable O/W emulsion.

Membrane technology that consists of Microfiltration, Ultra filtration, Nanofiltration and Reverse osmosis offers many advantages in several aspects such as high efficiency, low maintenance cost, flexible configuration and materials, can be used in various industries, compact size and environmental friendly. Membrane separation is a useful method for the treatment of micron sized oily wastewater. UF is considered to be a versatile separation method. Oily water with emulsion droplets of size higher than 50μm is unstable state, those less than 10 μm are considered to be highly stable. So it is very difficult to separate particularly when oil concentration is small. However little reports on treating oily emulsion with droplets below 4 μm have been reported.

While UF is a suitable technique to treat oily wastewater, membranes suffer serve fouling as a result of an adsorption and accumulation of rejected oil and other components on the membrane surface. To reduce fouling, the cross flow and batch type, where the feed flows parallel to the membrane surface, has been extensively applied to reducing the formation of cake layer. Several studies have reported that cross flow membrane filtration is an effective process in concentrating O/W emulsions.

In general inorganic membranes have good selectivity but poor mechanical strength, while polymeric membranes have good thermal and chemical stability, and immunity to microorganism. Among the various choice of polymeric material, polyether sulfone has been proved to be a robust membrane in terms of thermal, mechanical, electrical, chemical and hydrolytic stability, besides, polyacrylonitrile also posses good resistant in acid and base solutions make it easy to carry out membrane cleaning. Crosslinked polyether sulfone hydrophilic nature limits its potential to be an excellent membrane candidate. To our best comprehension, there have been fewer reports on combined by hydrophilic and hydrophobic membrane used in ultra filtration of oil emulsion.

In this study polyether sulfone cross linked polyether sulfone polyacrylonitrile polysulphone membranes, hydrophilic and hydrophobic membranes was used to treat stable O/W emulsion by cross flow and batch type configurations. The effect of pressure, feed concentration, flow velocity and filtration time, etc...on permeate flux and oil rejection were investigated. In addition, the cleaning of the fouled membrane was also studied.

2. EXPERIMENTAL DETAILS

2.1 Preparation of oil-water emulsion sample solutions

The oil-in –water emulsions were prepared vigorous mixing of oil and water with adding 2ml of sunflower oil and 2L of distilled water respect to o.1 ml of surfactant Tween 80 polysorbitone using a homogenizer at 8000rpm for 5min.

By changing the concentrations of oil, distilled water and surfactant we were prepared 1000, 5000ppm and 20,000

Table 1.Characteristics of polymeric membranes

Designation	Membrane material	Filtration Spectrum	Characteristics
PES20 kD	Polyethersulfone	UF	Partially hydrophilic
AN 50kD	Cross linked polyethersulphon e	UF	Good degree of hydrophilicity
HUF 5kD	Polyacrylonitrile	UF	Partially hydrophilic
UJ01	Polysulphone	UF	Hydrophobic

2.2 Experimental design

The UF experiments were carried out in batch stirred cell with a capacity of 70cm^3 to carry out feasibility studies, Cross flow system, the entire set of experiment were run in two modes of operation

a) Stirred cell to test the separation capabilities of membranes

b) Cross flow system to compare the performance

The UF experiments were carried out in batch stirred cell with a capacity of 70cm^3 to carry out feasibility studies. The UF cell was made of two pieces of Teflon. These parts were sealed by o-rings and rectangular membranes (0.0012m^2) were placed between them. It must be mentioned that for each experiment a new piece of membrane was employed. During the experiments supervision was carefully done to control pressure, feed concentration, temperature, pH.The permeate was collected for 30min and the pure water permeation flux (L/m^{2}h) was calculated using the following equation J=V/At, where A=membrane area, V is the collected permeate volume and t is the time. All of the adjustments for the UF experiments were the same.

Table 1.Characteristics of polymeric membranes

2.3 Analysis of waste water before treatment & after treatment

COD was determined according to the method described by APHA standard methods. For the estimation of the COD the digestion of the sample was done at 150ºc for 2h in a COD reactor(Model no 45600 supplied by Hach,coloradio USA).The digestion was done with potassium dichromate solution in presence of concentrated sulfuric acid 1:3 ratio. The digested sample was place at spectrophotometer.

2.4 Membrane Regeneration:

Regeneration of membranes depends on various factors such as membrane material, types of components in the feed system and cleaning methods used. The membrane was systematically cleaned and regenerated between runs. The cleaning cycle consisting of circulating 0.2% sodium hydroxide for 30mins, rinsing with permeate water; washing with 0.5% of SDS (sodium do decyl sulphate) and finally rinsing with permeate water.

3. RESULTS AND DISCUSSIONS

Oily wastewater characterization

Table 1 show the basic quantity of oily wastewater consists of conductivity, TDS, TSS, pH and COD. Those measurements are the water quality before and after the treatment process. According to the table, the water classified as fresh water. This is due to TDS reading that exceeds 1500mg/l as a requirement of fresh water and 500mg/l as WHO standards. Besides the reading emphasize the significant of TDS measurement cannot tell the exact percentage of amount for different solids in the feed.

Table 2: Characteristics of the sunflower oil-emulsion (Feed and permeate) PES, CPES

parameters	M_1		M_2	
	F	P	F	P
TDS (mg/L)	95	44	68	28
TSS (mg/L)	90	48	88	32
COD (mg/L)	640	165	640	46
Turbidity(NTU)	66	22	50	20

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Table 2: Characteristics of the sunflower oil-emulsion (Feed and permeate) PAN, PSF

parameters	M_3		M_4	
	F	P	F	P
TDS (mg/L)	95	32	88	36
TSS (mg/L)	96	40	96	40
COD (mg/L)	640	88	640	96
Turbidity(NTU)	66	18	78	32

F-Feed of the oil sample (before treatment)
P-permeate of the sample (after treatment)

TDS-Total dissolved solids
TSS-Total suspended solids
COD-Chemical oxygen demand
NTU- Nephelometric Turbidity Unit

M_1-Polyether sulfone
M_2-Crosslinked polyether sulfone
M_3-Polyacrylonitrile
M_4-Polysulphone

Effect of pressure on water flux, Membrane Type: PES20KD, Feed: Sunflower oil, batch type: stirred cell, Membrane area: 0.0064m^2 (Pressure at 30, 50, 70)

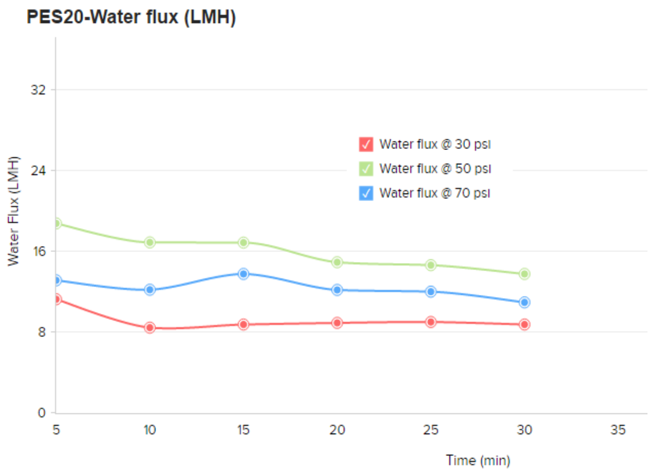

Fig 1: Effect of pressure on water flux

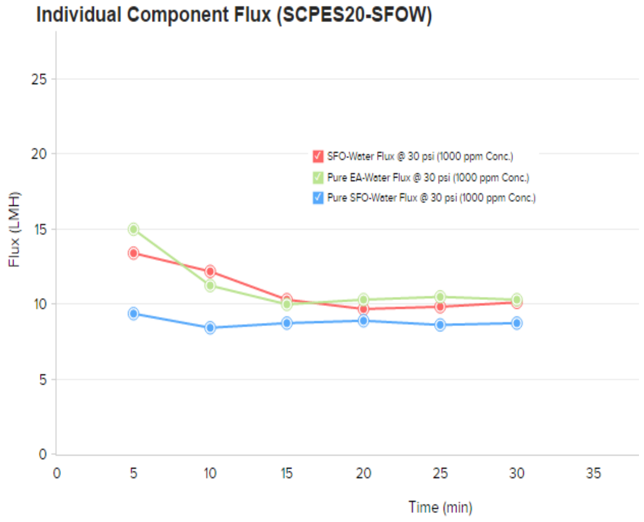

Fig 2: Effect of Pressures on sunflower oil-water emulsion flux

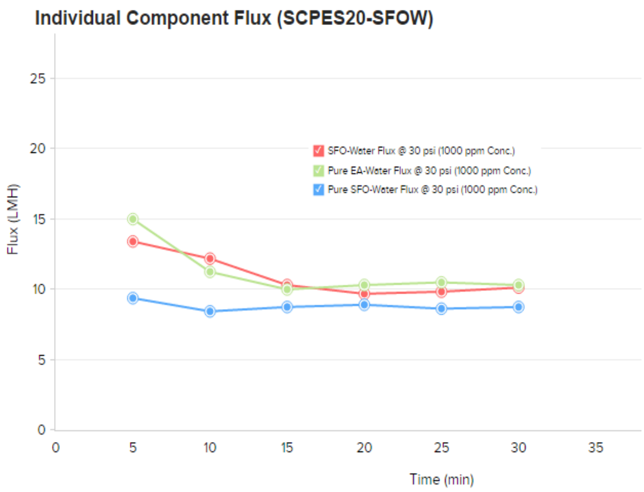

Fig .3: Flux behavior relating to individual components of the system

4. CONCLUSIONS

This study set out with the aim of accessing the viability of oily waste water treatment using polyether sulfone, crosslinked polyether sulfone, and polyacrylonitrile and polysulphone membranes.

Feasibility studies for selecting appropriate membranes for different oil-water systems have been carried out using batch test cell and industry equivalent mode of cross flow systems.

The rejection values are measured in terms of COD (combined value for individual components)

Among the four membranes cross linked poly ether sulfone have shown greater performance in terms of flux as well as rejection.

Membranes with high degree of hydrophilicity have hidden performance comparing the hydrophobic membranes.

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