International Journal of Scientific Research in Science, Engineering and Technology

Print ISSN - 2395-1990
Online ISSN : 2394-4099

OPEN ACCESS

Available Online at : www.ijsrset.com
doi : https://doi.org/10.32628/IJSRSET24114102

Studies on Recovery of Performance of Fouled Nanocomposite Ultrafiltration Membranes on Cleaning after Treatment of Oil–Water Emulsions

A. K. Ghosh*, V. S. Mamtani, A. K. Adak

Desalination & Membrane Technology Division, Bhabha Atomic Research Centre, Trombay, Mumbai, Maharashtra, India

*Corresponding author : akghosh@barc.gov.in

ARTICLEINFO

Article History:

Accepted: 25 June 2024

Published: 08 July 2024

Publication Issue :

Volume 11, Issue 4

July-August-2024

Page Number :

33-37

ABSTRACT

Herein, we discussed the pure water flux recovery of carbon based nanofillers embedded polyvinyl chloride nanocomposite membranes after physical cleaning through backwashing & flushing with flow reversal and after chemical cleaning using hydrochloric acid and caustic soda. The water flux recovery by backwashing is more than that by forward flushing for all the membrane but it is more effective in carbon black (CB) & graphitized carbon black (GCB) carbon-based membranes than relatively hydrophilic multiwalled carbon-nanotube (MWCNT) & carboxylated multiwalled carbon-nanotube (CMWCNT) based nanocomposite membranes. The highest water flux recovery was found ~96% for nanocomposite membranes by backwashing and caustic soda cleaning. The combination of backwashing and caustic soda cleaning could be the most effective method of cleaning of oil-water fouled membranes to restore the maximum water flux.

Keywords: Membrane, Polyvinyl Chloride, Nanocomposite, Fouling, Oil-Water Separation

I. INTRODUCTION

For separation of oil from oil-water mixture, membrane processes particularly, microfiltration and ultrafiltration have several advantages compare to conventional separation techniques such as higher oil removal efficiency, more compact design, easy scale up with process control and no chemical addition [1]. However, membrane fouling due to pore plugging by oil droplets and surfactant adsorption is one of the biggest challenges for oily wastewater treatment. To mitigate the fouling, one of the approaches is either to chemically modify the pure polymeric membranes or to develop fouling resistant membrane [2]. Chemical modification of pure polymeric membranes includes blending and surface modification (by coating & grafting of mostly a hydrophilic component) [3]. Another approach is to prepare nanocomposite membrane by incorporation of functional nanoparticles into the polymeric membrane which has been demonstrated the enhancement of both membrane permeability and fouling resistance [4].

Recently, our group also reported the development of nanocomposite ultrafiltration membrane by embedment of carbon black (CB), graphitized carbon black (GCB) or mesoporous carbon, multiwalled carbon-nanotube (MWCNT), carboxylated multiwalled carbon-nanotube (CMWCNT) nanofillers in polyvinyl chloride (PVC) membrane matrix for efficient separation of oil from emulsified oil/water mixture [5]. In spite of using fouling resistant membrane, it is impossible to avoid complete membrane fouling and to restore original permeability and oil rejection properties in long filtration run. Hence, cleaning of the running membrane is the common practice to avoid the fouling and to maintain the membrane performances. This cleaning process can be of physical or chemical cleaning. Flushing with deionized water in direction of flow of feed or by flow reversal mode and backpulsing/backwashing are commonly used physical cleaning methods to mitigate non-adhesive fouling but they are not effective to remove adhesive fouling. Chemical cleaning is required to remove adhesive fouling and applied when permeate flux decrease reaches 50–60% [6]. The selection of chemical for membrane cleaning depends on the nature of the foulants and the membrane materials. Both alkaline and acidic chemicals are used for cleaning of oil-water fouled membranes. It is important to study the effectiveness of different cleaning methods towards water flux recovery of fouled membranes after treatment of Oil–water emulsions. In this study, the PVC based nanocomposite UF membrane developed in our previous study [5] was used to evaluate the extent of water flux recovery of oil-water fouled membranes after physical cleaning through backwashing & forward flushing DI water and after chemical cleaning using aqueous solutions of hydrochloric acid and caustic soda.

II. EXPERIMENTAL

Membranes are prepared by phase inversion technique using casting solution prepared with 16% (w/w) PVC (Avg. Mw=43000Da), 5% (w/w) polyethylene glycol (Avg. MW= 600Da) and 79%(w/w) N, N-dimethyl formamide solvent. For preparation of carbon nanofillers embedded nanocomposite membranes, 0.8%(w/w) of the nanomaterials (carbon black nanopowder, graphitized carbon black, multiwalled carbon-nanotube and carboxylated multiwalled carbon-nanotube) were added to the PVC casting solution. The detail of the ultrafiltration (UF) membrane preparation is given in our previous paper [5].

Performance evaluation of the UF membranes in terms of product permeability and oil rejection was carried-out using a cross-flow filtration set-up (schematic details is given elsewhere [7]). The feed was prepared by mixing 1000 mg of liquid oil and 5 mg sodium dodecyl sulphate in 1 litre water and agitated for 24hours at 2500rpm. The oil separation was evaluated by measuring both turbidity and total organic carbon (TOC) of feed and permeate samples.

Fouling experiments were carried out by passing deionized (DI) water was first through the membrane at 350 kPa pressure until the water flux remained constant over 30 mins or more which took around 2.5-3.0 hours' time. The end of the stabilization period was taken as the zero time for fouling experiment [8]. The pure water flux was noted at operating pressure of 250kPa and then the oil-water emulsion was changed as feed and passed through the UF membranes at the same pressure. Permeate sample was collected at regular intervals upto 6hrs. of operation. Then, DI water was used as a feed to determine the reversibility of fouling in case of physical cleaning through backwashing & flushing with flow reversal. During backwashing, fouled membranes was put in skin side bottom position in the test-cell and DI was flushed using applying pressure of 100kPa [9]. In case of chemical cleaning, 10mM hydrochloric acid and caustic soda solutions were used independently as flushing solutions.

III. RESULTS & DISCUSSIONS

The pure water flux of all the membranes was measured at 250kPa pressure and membrane performance was evaluated in terms of percentage separation of oil from 1000ppm oil-water emulsion at the same applied pressure. All the membrane found to give more than 98.5% oil removal. The pure water flux was measured again after washing and cleaning the oil-water fouled membranes. The pure water flux of unfouled membranes as well as for washed/cleaned fouled membranes is given in Table 1. It can be seen that PVC-CB membranes has marginally lower water flux due to the non-porous nature of the carbon black which can block some of the pores in the membranes. As other nanomaterials (GCB, MWCNT & CMWCNT) have porous structure, the flux of their nanocomposite membranes was more. The water flux recovery on cleaning of fouled membranes by forward flushing & backwashing with DI water is given in Figures 1 & 2 respectively. Similarly, the water flux recovery on cleaning of fouled membranes by washing with HCl and NaOH solutions is given in Fig. 3 & 4 respectively.

TABLE 1: PURE WATER FLUX RECOVERY OF THE MEMBRANES ON DIFFERENT MODE OF CLEANING

Memb.	Pure water flux of unfouled memb. (LMH)	Pure water flux after cleaning of fouled membrane (LMH)			
		Forw-ard flush	Backwash	HCl Sol.	NaOH Sol.
PVC	62.7±3.2	31.4 ±2.8	53.3 ±2.3	52.7 ±2.5	54.6 ±2.6
PVC-CB	61.4±3.5	40.5 ±3.0	53.4 ±2.7	53.4 ±3.0	55.3 ±2.9
PVC-GCB	80.8±4.1	56.6 ±3.2	72.7 ±3.0	71.9 ±3.2	74.3 ±3.3
PVC-MWCNT	69.0±3.4	49.7 ±2.7	63.5 ±2.5	62.8 ±2.8	65.6 ±2.6
PVC-CMWCNT	85.9±3.9	65.2 ±2.9	82.5 ±2.6	79.9 ±2.9	82.5 ±2.9

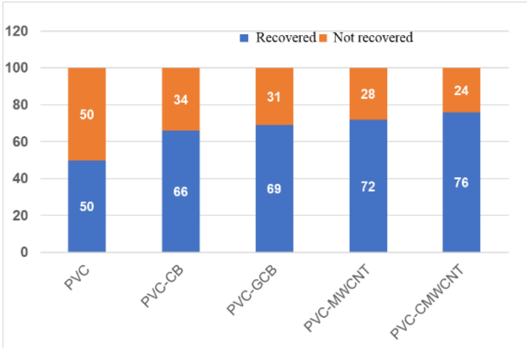

Figure 1: Flux recovery on washing of fouled membranes by forward flushing with DI water

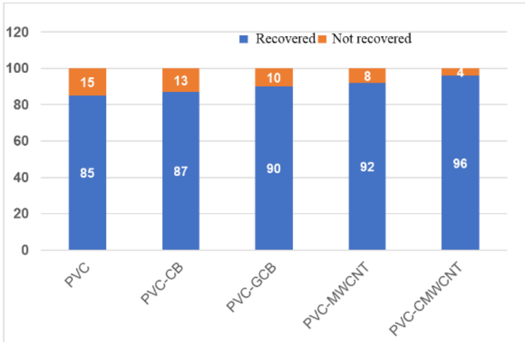

Figure 2: Flux recovery on backwashing of fouled membranes with DI water

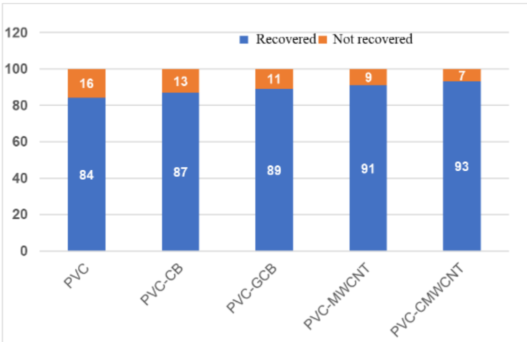

Figure 3: Flux recovery on cleaning of fouled membranes by 10mM HCl solution

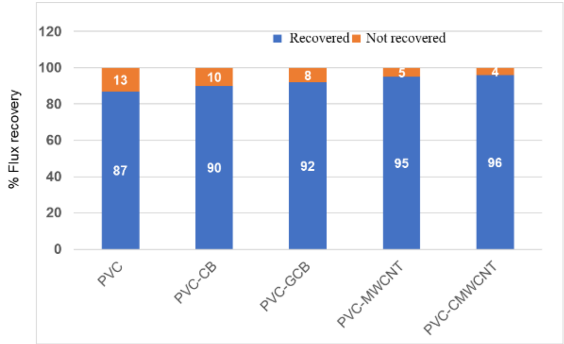

Figure 4: Flux recovery on cleaning of fouled membranes by 10mM NaOH solution

It can be seen that the water flux recovery is more for nanocomposite membranes than the pure polymeric membranes irrespective of the mode of cleaning. This could be due to the less adhesion of oil in carbon-based nanomaterials than the pure PVC polymer. The order in which the flux recovery is more is PVC-CMW-CNT > PVC-MWCNT > PVC-GCB > PVC-CB > PVC. The reason for the given order may be attributed to the due to more hydrophilic nature [5] of CMWCNT which limits interaction of oil with the membrane surface. As the hydrophilicity of the membrane decreases, the interaction between oil and membrane surface increases. Hence, water flux recovery in CNT carbon black (CB) & graphitized carbon black (GCB) carbon-based membranes are less than the relatively hydrophilic multiwalled carbon-nanotube (MWCNT) & carboxylated multiwalled carbon-nanotube based membrane. Recovery of water flux on backwashing of fouled membranes is much more than that of cleaning by forward flushing with DI water. Forward flushing can remove only loosely bound non-adhesive surface fouling but backwashing cleans foulants deposited inside the pores also alongwith the surface deposition. Both acid and alkali flushing were applied to remove the adhesive fouling and it is found that the alkali cleaning is more effective than the acid cleaning for oil-water fouled membrane. Alkalis are known to neutralize acidic organics and cause saponification of fats and oils which makes cleaning more effective [10].

IV. CONCLUSIONS

Nanocomposite membranes containing carbon-based nanomaterials gives more water permeable membranes than the pure polymer membranes. Oil caused very significant fouling in both pure polyvinyl chloride and nanocomposite membranes. Hence, identification of suitable cleaning method is required to restore the water flux of the membrane. The pure water flux recovery after fouling i.e., reversibility of fouling is more for nanocomposite UF membranes than that of pure polyvinyl chloride membranes. Among physical cleaning methods, backwashing is more effective to clean the fouled membrane. Alkali cleaning is more effective than the acid cleaning for restoration of water flux of oil-water fouled membrane.

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