# ASYMMETRIC POLY(ETHER-BLOCK-AMIDE)-1657 NANOFILTRATION MEMBRANES FOR PROCESSING OF AQUEOUS SOLUTIONS

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## ABSTRACT

*Nanofiltration (NF) is a membrane-based separation process used for treatment of industrial effluents and wastewater recycling. NF membranes have high potential to remove low molecular weight trace contaminants from aqueous and non-aqueous solutions, which cannot be removed efficiently by other conventional treatment methods. In the present study, asymmetric PEBAX-1657 (Poly ether block amide) nanofiltration (FNF) membranes were synthesized by phase-inversion technique from mixed solvent system. These indigenous membranes were characterized by Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), Thermo gravimetric analysis (TGA) and Scanning electron microscopic (SEM) studies to elucidate the structural, crystallinity, thermal stability, surface and cross-sectional morphologies of the membranes, respectively. The effect of various operating parameters such as permeate flux, total dissolved solids (TDS) and impurity rejection were studied. From the experimental results, an average flux of 68 L/m$^2$ h (Pebax-2533) and 66 L/m$^2$h (pebax-1657) were obsereved at a constant pressure of 21 kg/cm$^2$. TDS rejections were found to be 15.18 and 38.56 % respectively. A detailed economic estimation of commercial NF system for feed effluent capacity of 1 m$^3$/h is presented.*

***Keywords:*** ***Characterization, Economic estimation, Flux, Nanofiltration Membrane, Water purification***

## I INTRODUCTION

Membrane technology for Nanofiltration (NF) has gained significant importance owing to its lower energy requirements, capital investment, compactness, process safety and environmental viability (Louie et al., 2006). Poly(ether-block-amide) (PEBA) is the general name for a class of thermoplastic elastomers that consist of linear chains of rigid polyamide (PA) blocks and flexible polyether (PE) blocks. This copolymer depending on the PE block concentration becomes hydrophobic or hydrophilic. The hydrophobic grades exhibit high selectivity for extraction of aromatic organic compounds from water by pervaporation (PV) (Nunes et al., 1995). These polymers

are rubbery, thermoplastic elastomers with different degrees of hydrophilicity depending on the type of polyamide (e.g. Nylon- 6, Nylon-12) and polyether (e.g. polyethylene oxide, polytetramethylene oxide) segments (Kim et al., 2001). One way to enhance NF membrane productivity is to reduce the rate of fouling (i.e., the deposition of foreign material) on the membrane surface by coating membranes with a thin, highly water-permeable polyether-polyamide block copolymer (PEBAX) (Chan and Chen, 2004).

Recent studies of polymeric membranes for flue gas applications have focused on improving their performance to allow them to be cost-competitive with solvent absorption ( Kujawski and Roszak,2002; Wilks and Rezac, 2002 ). Structurally asymmetric or composite membranes consisting of a thin separation layer with the support of a micro-porous substrate are used in almost all industrially important gas separation processes (Scholes et al., 2008; Liu, 2004). Lua and Shen (2013) prepared a carbon–silica composite membrane using sol–gel technique, and it surpassed Robeson's upper bound limit for $He/N_2$, $CO_2/N_2$ and $O_2/N_2$ gas pairs. Liu et al. (2005) produced a PEBAX composite membrane to separate $CO_2$ from $N_2$ by dip-coating the polymer substrate and investigated the effects of operating conditions on the performance of hollow fiber membranes.

Polyether-block-amide (Pebax) has recently been studied to remove ethanol from aqueous solutions since it provides competitive permselectivity towards specific organic solvents [Scholes etal., 2005; Liu et al., 2009). Pebax is a group of copolymers containing the hard polyamide (PA) segments and soft polyether (PE) segments. In Pebax molecules, PA segments promote mechanical strength while PE segments provide good affinity to organic solvents (Brink et al., 1993; Kou et al., 2003). Depending on the nature and proportion of PA and PE segments, the characteristics of the Pebax polymer can be varied (Gilron et al., 2001; Wang et al., 2006). Generally, the higher proportion of the flexible PE segment, the more organophillic the Pebax polymer is. Among commercially available Pebax polymers (2533, 3533, 4033, 1657, 1740), Pebax 1657 has the highest content of PA segment. It is therefore expected to have the highest hydrophiliicity and maximum separation capacity for the removal of organic compounds from aqueous solutions.

In response to the challenge of developing new membrane materials, PEBAX-1657 NF membrane has been synthesized in this study for removal of inorganic and organic pollutants from industrial effluent and water treatment. The indigenous membrane was characterized by Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), Thermogravimetric analysis (TGA) and Scanning electron microscopy (SEM) to study the structure, crystallinity, thermal stability, surface and cross-sectional morphology, respectively. The effect of various experimental parameters on water flux and % rejection has been evaluated. The influence of composition in terms of total dissolved solids (TDS), conductivity, turbidity on membrane performance was determined. PEBA membrane was prepared for separation of aqueous mixtures. At first, effect of different parameters on film formation such as ratio of solvents, temperature, composition of coagulation bath (water) and concentration of polymeric solution were studied. The prepared membrane showed good performance for separation of aqueous solutions. A detailed economic estimation of a commercial NF system for processing 1 $m^3$/h of feed is also presented.

## II EXPERIMENTAL

### 2.1. Materials and methods

Pebax-1657 was chosen to enable greater interaction with $H_2O$ molecules through H-bonding as this grade of the polymer contains 40% amide groups. Pebax-1657 was purchased from Sigma-Aldrich, Mumbai, India. Pure (100%) ethanol, methanol, citric acid, HCl, EDTA, NaOH and sodium metabisulphite (SMBS) for washing and storage of the membranes were obtained from sd Fine Chemicals Ltd., Mumbai. The indigenous NF membrane of flat sheet configuration having an effective area of 0.015 $m^2$ was synthesized in the laboratory. Automatic film coater and non-woven fabric support were obtained from Permionics Membranes Pvt. Ltd., Vadodara, India. Colorimeter (Hach-DR-890, Bangalore, India), Conductivity (DCM-900) and pH meters (DPH-504) were procured from Global Electronics, Hyderabad, India, to facilitate analysis of feed, permeate and reject samples.

### 2.2. Synthesis of Pebax-1657 NF membrane

Solvent resistant membranes of Pebax-1657 were prepared by phase inversion method using a 15 wt.% Pebax-1657 solution by initially adding a small amount of the polymer pellets to a solvent mixture of 90% ethanol and 10% water. After complete dissolution of the initial polymer, remaining polymer was added gradually. The polymer was dissolved at 90 $^oC$ with rigorous stirring with constant reflux over a period of 6 h. The bubble free polymer dope solution was cast using a doctor's blade on a non-woven polyester fabric support affixed onto a clean glass plate. After 30 seconds the plate was immersed in ice cold water bath to obtain solvent resisiant NF membrane whose total thickness was 103 µm including non-woven fabric thickness. The chemical structure of Pebax-1657 is shown in Fig.1

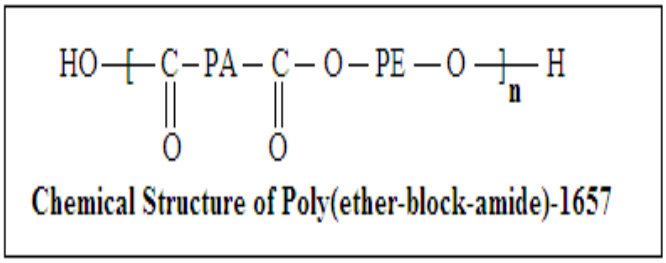


**Fig. 1: Chemical Structure of PEBAX-1657**

### 2.3. Description of NF system

The schematic of pilot scale NF system is shown in Fig. 2. A feed tank of 30 L capacity made of stainless steel was provided for storage and supply of the industrial effluent to the system. A polypropylene (PP) prefilter cartridge of 5 µm pore size was installed upstream of the flat sheet membrane module to prevent the entry of suspended solid particles. A high pressure pump (Hironisha, Japan) capable of maintaining a pressure upto 21 $kg/cm^2$ was used for transporting the feed liquid throughout the system. The pump was run by a 2 HP single phase motor (Crompton, India). The feed tank had a provision for recycling of the concentrate after it passed through a heat exchanger that

was installed for maintaining a constant feed temperature (26-28 $^{o}$C). The heat exchanger consisted of a lengthy glass shell, which was circulated with ice cold water while the effluent was flowing in concentric glass coils placed inside the shell which provided large heat transfer area. The reject coming out of the heat exchanger was then recycled to the feed tank as concentrate. A restricting needle valve was provided on the concentrate outlet at a position after the membrane module to pressurize the feed to a desired value which was indicated by a pressure gauge installed upstream of the needle valve in the reject line. Permeate and concentrate flow rates were measured using rotameters (Venkata Swamy et al., 2013).

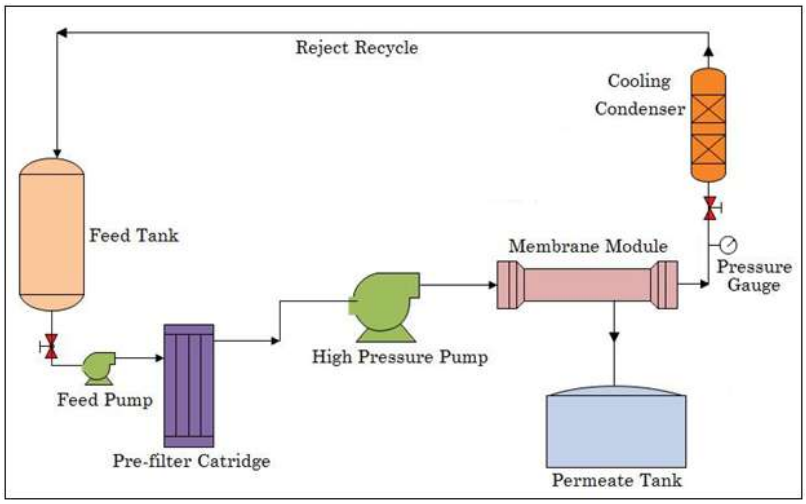


**Fig. 2: Process Flow Diagram of NF system**

### 2.3. Experimental procedure

The NF membrane was fixed in the system's flat sheet test cell, cleaned and wetted using distilled water. The experiments were then carried out with deionized water to study the effect of feed pressure on flux. Deionized water was taken in the feed tank and transported to the flat sheet NF membrane module using the high pressure pump. The concentrate outlet was left to flow into a bucket instead of being recycled to the feed tank in order to maintain a constant feed concentration. The system pressure was varied by throttling the needle valve in the concentrate line. The flow rates of permeate and concentrate were measured at each pressure. The stainless steel feed tank was then filled with 30 L of IICT bore well water feed and the system was initially run to remove 2.2 L of distilled water accumulated as dead volume in the system. A sample of initial feed was collected for analysis. The experiment was performed to study the effect of feed concentration on flux and % rejection at an optimum pressure of 21 kg/cm$^2$ with total concentrate recycle for achieving 65% water recovery. The flow rates of permeate and concentrate were measured at regular time intervals to observe any decline in flux. After a particular water recovery was attained, samples of initial feed, final concentrate and average permeate were analyzed for TDS, conductivity and turbidity values. Finally, the system was cleaned and washed with distilled water to remove solutes from the membrane surface and pores ( Venkata Swamy et al., 2013).

### 2.4. Fouling and its prevention

In general, fouling of membranes is caused by accumulation of suspended solids, salts, microbes and organic materials present in the feed water either on the membrane surface or within the pores (Luo et al., 2012). A solution of citric acid or HCl (1% w/v) was recycled through the system for about 10 min for removal of mineral scales and metal precipitates. A mixture of 1% w/v sodium hydroxide + 0.5% tetra sodium EDTA chelating agent was used to remove organic scales. On alternate occasions, 0.1% w/v of sodium lauryl sulfate, a surfactant, was added to this alkaline cleaning mixture for polishing the membrane surface.

## III MEMBRANE CHARACTERIZATION

### 3.1. Fourier transform infrared (FTIR)

The synthesized NF membrane was characterized for their intermolecular behavior. The membranes were scanned in the range 400–4000 $cm^{-1}$ wavenumber using Nicolet-740, Perkin-Elmer-283B FTIR spectrophotometer (Boston, MA, USA) by KBr pellet method.

### 3.2. X-Ray diffraction (XRD) analysis

A Siemens D 5000 powder X–ray diffractometer, NJ, USA was used to assess the solid–state morphology of TFC polyamide NF membrane. X-rays of 1.54 Å wavelengths were generated by a CuK–alpha source.

### 3.3. Thermo gravimetric analysis (TGA)

Thermal stability of NF membrane was examined using a Seiko 220TG/DTA analyzer, Japan in the temperature range of 25–800 °C at a heating rate of 10 °C $min^{-1}$ with continuous flushing using pure $N_2$ gas flowing at 200 ml/min to determine thermal stability and decomposition characteristics.

### 3.4. Scanning electron microscopy (SEM)

The surface and cross-sectional morphology of NF membrane was studied by SEM instrument of Model JEOL JSM–6380, LA, USA. In preparing the specimens, the fracture surface and cross-section of the NF membrane, ultraporous substrate and non-woven fabric polyester support were obtained by cutting the membrane samples in liquid $N_2$ to ensure smooth morphology.

## IV ANALYTICAL METHODS

The feed and permeate samples were analyzed for TDS, conductivity and turbidity according to APHA methods (1998). The conductivity of above samples was determined using the digital conductivity meter.

## V RESULTS AND DISCUSSION

### 5.1. Membrane characterization

### 5.1.1. FTIR

Fig. 3 exhibits the FTIR spectra of NF Pebax 1657 membrane. The prominent peaks of -C=O carbonyl group of stretching vibrations observed in both polyamide (PA) and polyether (PE) groups at 1541cm$^{-1}$(CO-NH) and 1715 cm$^{-1}$ (-C-O-C-) respectively.

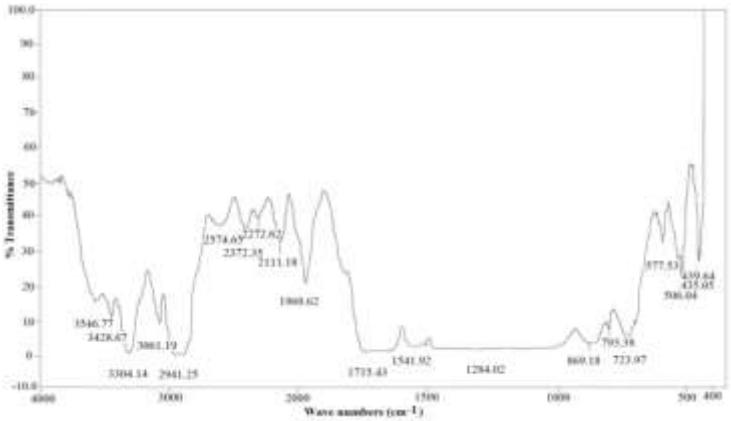


**Fig. 3: FTIR Spectra of NF membrane**

On other hand bending vibrations of amide group is noted in polyamide at 3304 cm$^{-1}$ and ether linkage of pebax (-C-O-C-) is appears in the polymer chain of 1284 cm$^{-1}$. These peaks are in accordance with the structure of pebax as confirmed by Kalyani et al (Kalyani et al., 2006).

### 5.1.2. XRD analysis

X-ray diffraction studies confirms the nature of polymer and and also indicates the intersegmental distance between polymer chains. The X–ray diffractogram of NF pebax membrane shown in Fig. 4 appears to display semi-crystalline nature. XRD pattern shows sharp diffraction peaks at around 17° ,23 and 27° of 2$\theta$ scale with *d*-spacing values of 4.92, 3.85 and 3.39 Å, corresponding to the functional groups of the copolymer. The Spectrum support the chemical structure of pebax membrane ( Krishna et al., 2012).

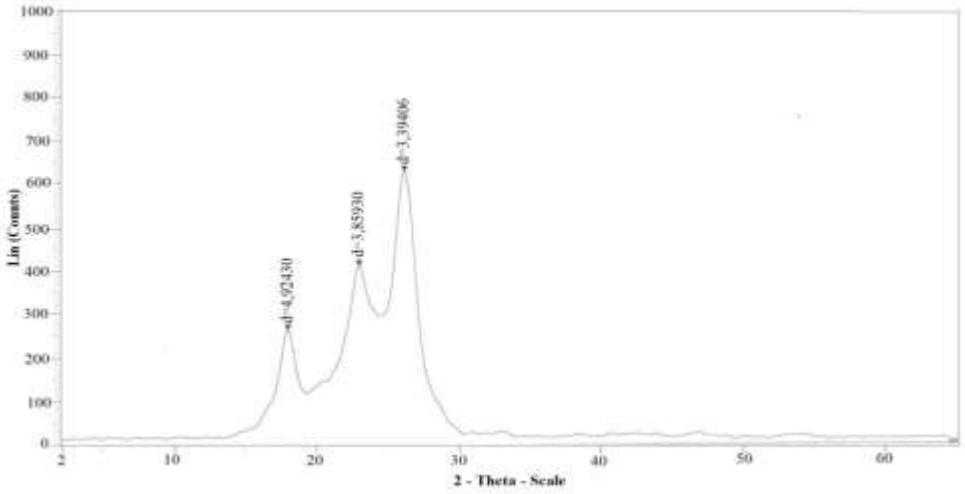


**Fig. 4: XRD Spectra of NF membrane**

### 5.1.3. TGA Studies

The TGA curve of NF membrane shown in Fig. 5 indicates the stretching weight loss at 420 °C followed by the decomposition at 470 °C, which is due to the disintegration of molecular chains.

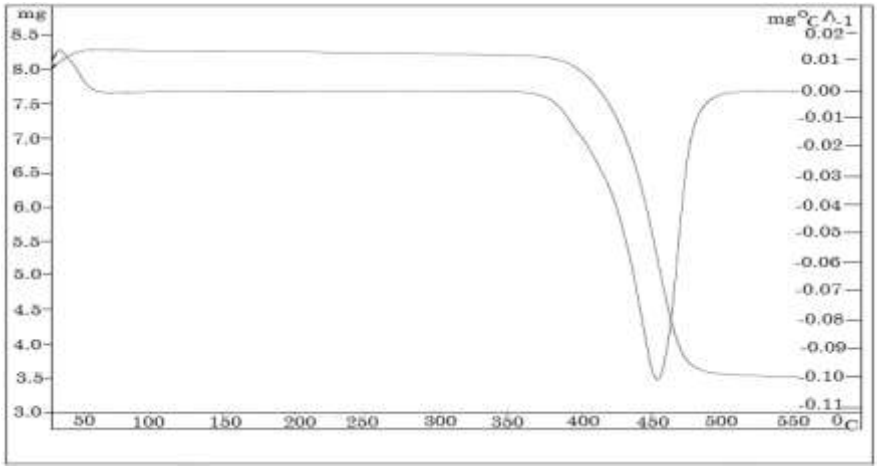


**Fig. 5: TGA Curves of NF membrane**

From the results it can be concluded that pebax membrane has high thermal stability.

### 5.1.4. SEM Analysis

The surface and cross-section of NF pebax membrane shown in Fig. 6. Surface morphology of pebax shows nanoporous openings distributed through out the membrane surface. Cross-sectional view of the membrane represents two layers, with the top layer being nanoporous pebax and non-woven polyester fabric support at lower layer with adequate interpretation of nanoporous pebax film.

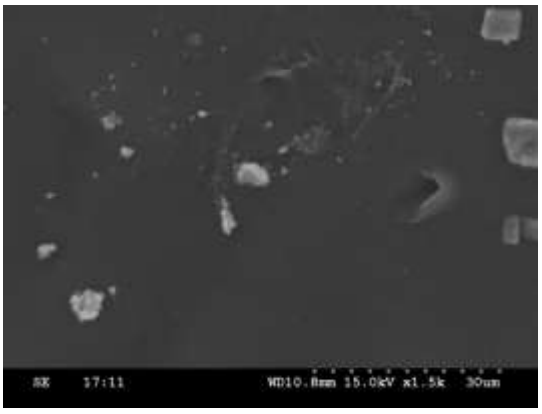

**Fig. 6 a: SEM Image of NF Membrane Surface**

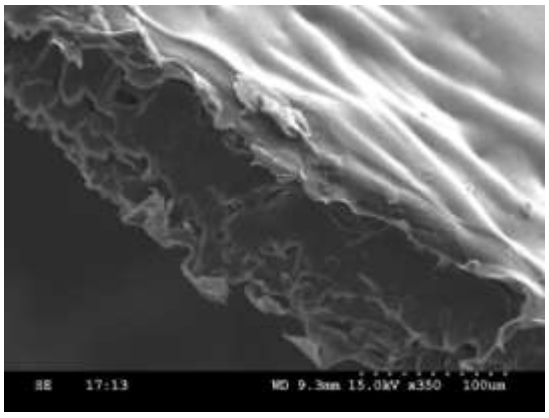

**Fig. 6 b: SEM Image of NF Membrane Cross-Section**

## 5.2. Effect of pressure on flux

The effect of pressure on (a) flux and %rejection, (b) conductivity of reject and permeate using NF process is shown in Fig. 7. As expected, a rise in pressure caused an enhancement in both flux and %rejection (Fig. 7a). Since the

driving force of the process enhances, it results in enhancement of flux due to increased affinity between $H_2O$ molecules and –CONH moiety of polyamide and –o- functional group of polyether layer.

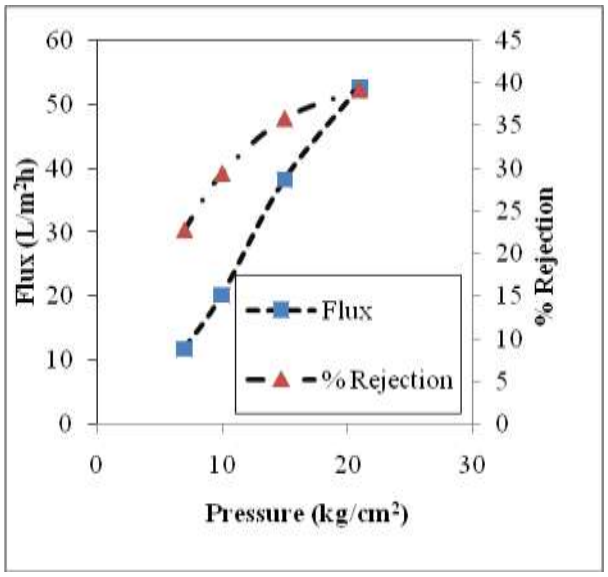


**Fig. 7 a: Effect of Pressure on Flux and % Rejection for Bore well Water**

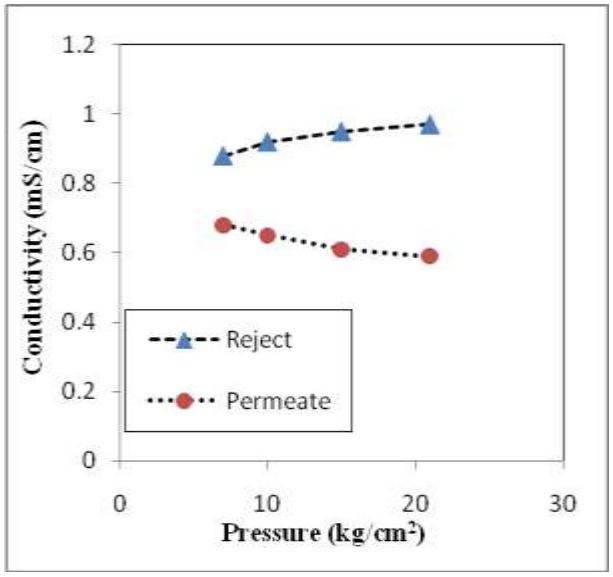


**Fig. 7 b: Effect of Pressure on Permeate and Reject Conductivities**

The flux was zero at applied pressures less than 3 kg/cm$^2$ due to high osmotic pressure arising from substantial concentration of dissolved solids in the effluent feed. The conductivity of the permeate reduced with increasing feed pressure (Fig.7b) since the solute flux remains more or less the same irrespective of the applied pressure owing to lack of any interaction with the membrane as described by the solution-diffusion mechanism (Luo et al., 2012). On the other hand $H_20$ molecules interact with the membrane and get transported at higher pressures.

### 5.3. Effect of operating time on flux, rejection and conductivity

Effect of time on flux and % rejection of bore water is graphically illustrated in Fig. 8. Flux declined from 65 to 59.61 L/m$^2$h due to increasing concentration polarization near the membrane surface arising from continuous retention of inorganic and organic solutes. On other hand, the rejection decreased from 38.56 to 32.25% over a period of 15 min (Fig. 8). Correspondingly, the conductivity of permeate increased from 0.6 to 0.63 mS/cm and that of reject from 0.89 to 0.93 mS/cm (Fig. 9) at a water recovery of 65% in permeate.

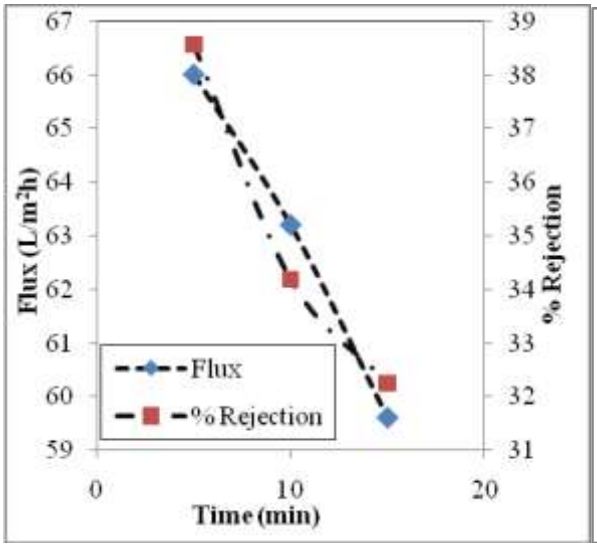


**Fig. 8: Effect of operating time on NF Membrane on Flux and % Rejection for bore water**

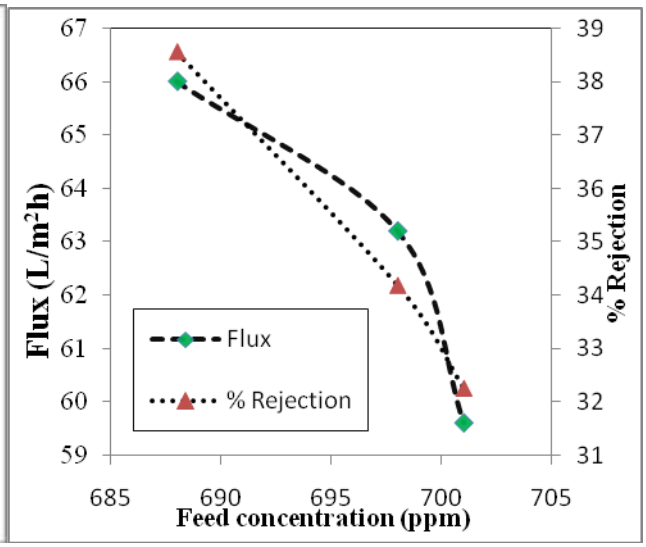


**Fig. 9: Effect of Feed Concentration on Flux and Rejection of NF membrane at 21 kg/cm$^2$**

### 5.4. Separation of TDS, conductivity, turbidity

Table 1 depicts the TDS, conductivity and turbidity values of feed, average permeate and final reject for NF process. A recovery of 65% was obtained at an average flux of 62.60 L/m$^2$h. From experimental observations it can be concluded that the membrane significantly reduces the TDS, conductivity and turbidity present in the feed. Permeate obtained during the process was analyzed as per APHA standards [19] and can be recycled for utilization in agricultural activities ,industrial cooling towers after meeting disposal standards under pollution control board norms.

**Table 1: Feed Characteristics of IICT Bore Water and Reduction of TDS, Conductivity, Turbidity and % Recovery**

| Sample | TDS (ppm) | Conductivity (mS/cm) | Turbidity (FAU) | %Recovery |
|---|---|---|---|---|
| Feed(Tap water) | 603 | 0.89 | 100 | – |
| Permeate | 576 | 0.63 | 6 | 65.00 |
| Reject | 698 | 0.93 | 150 | – |

### 5.5. Scale-up and economic estimation

#### 5.5.1. Capital investment and list of equipment for NF process

List of equipments and capital costs are provided in Table 2, in which the unit price for major accessories like high pressure centrifugal pump (Grundfos, Denmark), TFC polyamide NF membrane modules with pressure vessel and skid costs are included. The total capital investment for processing of 1 $m^3$/h of biscuit effluent is approximately 2965 USD (INR 1.77900 Lacs).

**Table 2: Capital cost of NF system**

| Item | Capacity/Size | MOC | Quantity | Total cost (USD) |
|---|---|---|---|---|
| Membrane housing | -- | -- | 1 | 200 |
| TFC polyamide membrane module | 8” dia × 40” long | -- | 1 | 1100 |
| Pressure vessel | 35 lpm | -- | 1 | 300 |
| Feed pump (2.5 HP) | 35 lpm | -- | 1 | 150 |
| 1 HP high pressure pump | 35 lpm | SS | 1 | 800 |
| Skid | 17 lpm | SS | 1 | 200 |
| Filter assembly | 35 lpm | PP | 1 | 15 |
| Accessories (Rotameters, Valves, TDS/Conductivity, pH meters) | -- | -- | 1 set | 200 |
| **Total cost** | | | | **2965 (INR 1.77900 Lacs)** |

(lpm – liter per minute; SS – Stainless steel; MOC – Material of construction; PP – Polypropylene)

**5.5.2. Operation and maintenance cost of NF process**

Operating and maintenance costs of NF system are given in Table 3, which includes membrane replacement and

**Table 3: Operation and maintenance cost of NF system**

| | |
|---|---|
| | 1 |
| | 0.85 |
| ***Feed capacity ($m^3$/hr)*** | 85 |
| ***Permeate capacity ($m^3$/hr)*** | |
| ***Module replacement cost*** | |
| Number of modules (8’’ dia, 20’’ long) | 1 |
| Price per module (USD) | 350 |
| Total module replacement cost (USD) | 350 |
| Duration of replacement (Years) | 3 |

| | |
|---|---|
| No. of working hs per day | 18 |
| Cost/hr (USD) | 0.017 |
| ***Cartridge replacement cost*** | |
| No. of cartridges | 1 |
| Price per cartridge (USD) | 8 |
| Total cartridge replacement (USD) | 8 |
| Duration of replacement (days) | 180 |
| No of working hs per day | 18 |
| Cost/h (USD) | 0.002 |
| ***Power cost*** | |
| Feed pump (kW) | 1.12 |
| High pressure pump (kW) | 1.85 |
| Dosing system (kW) | 0.015 |
| Total power consumption (kW) | 2.98 |
| Price per unit (USD) (6 Rs./unit) | 0.1 |
| Total power cost (USD) | 0.3 |
| ***Chemical consumption*** | |
| Antiscalant dosing (ppm) | 5 |
| Dosage (L/h) | 0.01 |
| Cost/lit (USD) | 6.4 |
| Hourly cost (USD) | 0.064 |
| CIP chemicals (EDTA, NaOH, citric acid) | |
| Frequency (days) | 15 |
| Total cost of CIP per hour (USD) | 0.045 |
| Labor cost per hr (USD) | 0.463 |
| Total operating cost per h (USD) | 0.89 |
| Total operating cost per year (USD) | 5847.3 |
| Depreciation cost (assuming 10% of capital cost) (USD) | 178 |
| Interest (5% of capital cost) (USD) | 63 |
| Total cost per year (USD) | 6036.3 |
| ***Permeate*** | |
| Quantity (LPH) | 850 |
| Operation time (h) | 18 |
| Quantity of permeate generated in 1 year (L/yr) | 5584500 |
| Cost of permeate per liter (USD) | $1.108\times10^{-3}$ |
| If sold at $4\times10^{-3}$ USD per liter | |

| | |
|---|---|
| Annual Profit (USD) | 16301.7 |
| Payback period (yrs) | 0.37 |

prefilter cartridge replacement costs, electric power consumption besides chemicals for cleaning and storage of the membranes. Feed capacity and recovery were assumed to be 1 $m^3$/h and 65% recovery, respectively. The operating duration was assumed to be 18 h per day and duration of membrane module replacement once every 3 years. Depreciation costs were taken as 10% of total capital investment. The cost per liter of permeate produced was found to be Rs. 0.0846 ($1.41\times10^{-3}$ USD) with a payback period of 0.55 years.

## VI CONCLUSIONS

The study revealed that indigenously synthesized Pebax-1657 asymetric nanofiltration (NF) membrane can be easily prepared and effectively used for treatment of water. The membrane was characterized by Fourier transform infrared spectroscopy (FTIR), X-ray diffraction studies (XRD), Thermogravimetric analysis (TGA) and Scanning electron microscopy (SEM) to elucidate structural nature, crystallinity, thermal stability, surface and cross sectional morphology, respectively. The flux and % rejection with respect to effluent was evaluated under various operating conditions. The membrane effectively removed total dissolved solids (TDS) and turbidity from feed to a considerable extent. Purification of bore water by NF process is more economical, energy efficient and eco-friendly when compared to reverse osmosis. The robust NF membrane exhibited considerably high water permeability and substantial anti-fouling ability which clearly offers vast scope for treatment of effluents coming from dairy, biochemical, food and bulk drug industrieswhich may contain aggressive solvents that can damage conventional RO/NF membranes.

## VII. ACKNOWLEDGEMENTS

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