

Multi- effect air gap membrane distillation process for pesticide wastewater treatment

Bhausahab L. Pangarkar^{*1,2}, Samir K. Deshmukh^{3a} and Prashant V. Thorat^{2b}

¹Chemical Engineering Department, Sir Visvesvaraya Institute of Technology, Chincholi, Nashik,
(Affiliated to SavitribaiPhule Pune University, Pune), 422 102, India

²Chemical Engineering Department, College of Engineering and Technology, Akola
(Affiliated to the SantGadge Baba Amravati University, Amravati), 444 104, India

³Chemical Engineering Department, Priyadarshini Institute of Engineering and Technology, Nagpur, 440 019,
India

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Abstract. A multi-effect air gap membrane distillation (ME-AGMD) module for pesticide wastewater treatment is studied with internal heat recovery, sensible heat of brine recovery, number of stages and the use of fresh feed as cooling water in a single module is implemented in this study. A flat sheet polytetrafluoroethylene (PTFE) membrane was used in the 4-stage ME-AGMD module. The maximum value of permeate flux could reach 38.62 L/m²h at feed -coolant water temperature difference about 52°C. The performance parameter of the module like, specific energy consumption and gain output ratio (GOR) was investigated for the module with and without heat recovery. Also, the module performance was characterized with respect to the separation efficiency of several important water quality parameters. The removal efficiency of the module was found to be >98.8% irrespective water quality parameters. During the experiment the membrane fouling was caused due to the deposition of the salt/crystal on the membrane surface. The membrane fouling was controlled by membrane module washing cycle 9 h and also by acidification of the feed water (pH=4) using 0.1M HCl solution.

Keywords: membrane distillation; multi-effect air gap membrane distillation; wastewater treatment; pesticide wastewater

1. Introduction

Nowadays, water scarcity is one of the most important problems and which is increasing due to rise in population and industrial development. Hence it is driving the implementation of wastewater treatment and water reuse on the large scale. Water recovery from wastewater treatment is an emerging and promising resource for the view of fresh water demand (Oren 2008, Poroda *et al.* 2013, Elimelech and Phillip 2011). In developed countries, widespread shortage of

*Corresponding author, Ph.D. Student, E-mail: pbl_1978@yahoo.com

^aPh.D., E-mail: samirdesh23@yahoo.com

^bPh.D., E-mail: pvthorat@rediffmail.com

water is caused due to contamination of ground and surface water by industrial effluents, and agricultural chemicals. Wastewater from manufacturing or chemical processing industries contributes to water pollution (Ali *et al.* 2012, Babu *et al.* 2011). The problem of water pollution has become still worse due to toxic organic components. Organic pollutants include pesticides, fertilizers, hydrocarbons, phenols, proteins, plasticizers and carbohydrates.

Pesticide containing industrial wastewater differentiates itself due to its toxicity in nature in the environment. The major pollution of the wastewater is released from the pesticide manufacturing industrial effluent. The pesticide contamination level in the industrial wastewater is high as 500 mg/l (Chiron *et al.* 2000). The excessive use of pesticides has resulted in surface and ground water pollution (Jani *et al.* 1991, Kumar and Singh 1997, Kumar *et al.* 1995). The various innovative technologies are used to treat the pesticide wastewater, such as Fenton oxidation, ozonation (Al-Hattab and Ghaly 2012), coagulation, photo catalytic degradation (Dong *et al.* 2010) phytoremediation (Pathak and Dikshit 2011), adsorption (Singh *et al.* 2010, Srivastava 2009), and biodegradation (Pathak and Dikshit 2011). But these techniques are cost intensive and are not eco-friendly in nature.

Recently, the membrane separation technologies like reverse osmosis (RO), nano-filtration (NF), electro dialysis (EC) (Ahalya *et al.* 2003) are gradually becoming an attractive alternative to the conventional separation processes. But these are the pressure-driven membrane technologies and more energy consuming and having osmotic pressure limitations (Elimelech and Phillip (2011)). Therefore, searching for a new alternative wastewater technology is of an interest.

A new thermal driven membrane technology such as membrane distillation (MD) process has been attracting the interest of scientific and academic communities due to its excellent performance in the desalination and wastewater treatment (Alklaibi and Lior 2004, Alkudhiri *et al.* 2013, El-Abbassi *et al.* 2013). The MD process differs from other membrane technologies in the sense that the driving force is the vapor pressure difference across the membrane. The porous hydrophobic membrane is used in the MD process (Lawson and Lloyd 1997, Curcio and Drioli 2005). The MD process is operated at lower temperatures and atmospheric pressure as compared to traditional processes. Hence it requires only low grade energy such as solar energy or waste heat (Mannella *et al.* 2010, Li *et al.* 2008). The applications of the MD process reported in the literature in the wastewater treatment of many industries like rubber industry (Mokhtar *et al.* 2015), olive oil mill (El-Abbassi *et al.* 2013), textile industry (Mokhtar *et al.* 2013) and dairy industry (Hausmann *et al.* 2013). But till today, there is no work found in the literature on the possible use of the MD process for the treatment of pesticide manufacturing industrial wastewater.

MD process found some limitations in the literature such as a low permeate flux, high thermal energy requirement, lacking in module design and high cooling water consumption as compared to other conventional membrane technologies. Hence, for the commercialization and to overcome the limitations of the MD, there is need to design a new type of multi-effect MD module. Some researchers presented the memsys vacuum multi effect membrane distillation (V-MEMD) process for the desalination purpose (Zhao *et al.* 2013, Lu *et al.* 2012, Liu *et al.* 2012). The multi-effect membrane distillation (MEMD) has advantages over the traditional MD like high permeate flux, recovery of latent heat, low cooling water consumption, low grade energy requirement, high stability, low maintenance cost and it is simple to operate. The multi-stage AGMD process is gives a high water recovery and gain output ratio (GOR) (Geng *et al.* 2015).

The air gap membrane distillation (AGMD) is one of the configuration of the MD process, in this configuration the air gap is introduced at the permeate side between the membrane and condensation surface. The AGMD has advantages like low conductive heat losses, recovery of

latent heat, low chance to membrane wetting due to air gap and low temperature polarization effect (Cipollina *et al.* 2012, Barth and Mays 1991, Liu *et al.* 1998, Warsinger *et al.* 2014, Al-Anezi *et al.* 2013, Khalifa *et al.* 2015, Zhao *et al.* 2013). Due to the above advantages of AGMD, the multi-effect concept was added in the traditional AGMD configuration. Hence this new type of module is known as multi-effect air gap membrane distillation (ME-AGMD). In this paper, the performance of this new type ME-AGMD configuration was studied for the treatment of actual pesticide manufacturing industrial wastewater.

2. Material and methods

2.1 Membrane

The flat sheet membrane made up of polytetrafluoroethylene (PTFE) polymer, which is commercially available was used in the experiment. The membrane sheet was supplied by Madhu Chemicals Pvt. Ltd. Mumbai (India). The pore size, porosity and thickness of the PTFE membrane are $0.45\ \mu\text{m}$, 70% and $175\ \mu\text{m}$ respectively. The effective membrane area of 4-stage ME-AGMD module is about $320\ \text{cm}^2$.

2.2 MEMD module preparation

The 4-stage ME-AGMD module was prepared by using the acrylic material. This module was constructed based on the air gap MD configuration. The aluminum foil was used as cooling plates in the module. In a single module 4-stages are created, hence this module is called 4-stage ME-AGMD module. The theoretical modeling and experimental validation for the saline water was studied previously (Pangarkar and Deshmukh 2015). The 4-stage module contains 03 feed channels, 02 cooling channels and 04 permeate or air gap channels, the length of each channel is about 100 and width is 80 mm. The depth of the each feed and cooling channels are fixed about 5 mm, and air gap thickness is about 2 mm. The internal channels and flow of water in the module are shown in Fig. 1. The effective membrane area for single stage is about $0.008\ \text{m}^2$ and 4-stage is about $0.032\ \text{m}^2$. In this module, one cooling channel is used commonly in the two successive stages. The fresh feed was circulated through the cooling channels for cooling purpose, for recovery of internal latent heat of vaporization. The permeate vapor was condensed on the surface of the aluminum foil. The picture of internal arrangement of 4-stage ME-AGMD module is shown in Fig. 2(a) and the assembled module picture is shown in Fig. 2(b).

2.3 Experimental setup and procedure

The schematic representation of 4-stage ME-AGMD system is shown in Fig. 3. The feed water contained in a 20 liter of the feed tank with immersion heater and thermostat. The circulation pump (0.5 hp) is used for the circulation of feed water from the feed tank to the first feed channel. Similarly 20 liter of cooling tank was used and circulates fresh feed water through the cooling channels of the membrane module. The flow rates are measured by using the Rotameter and temperatures by thermocouples of pt100 sensors. The fresh feed water was added in the first feed tank and it is re-circulated as a coolant. The latent heat is recovered during condensation of water vapor. And the sensible heat is recovered in the heat exchanger from the hot brine solution. Then

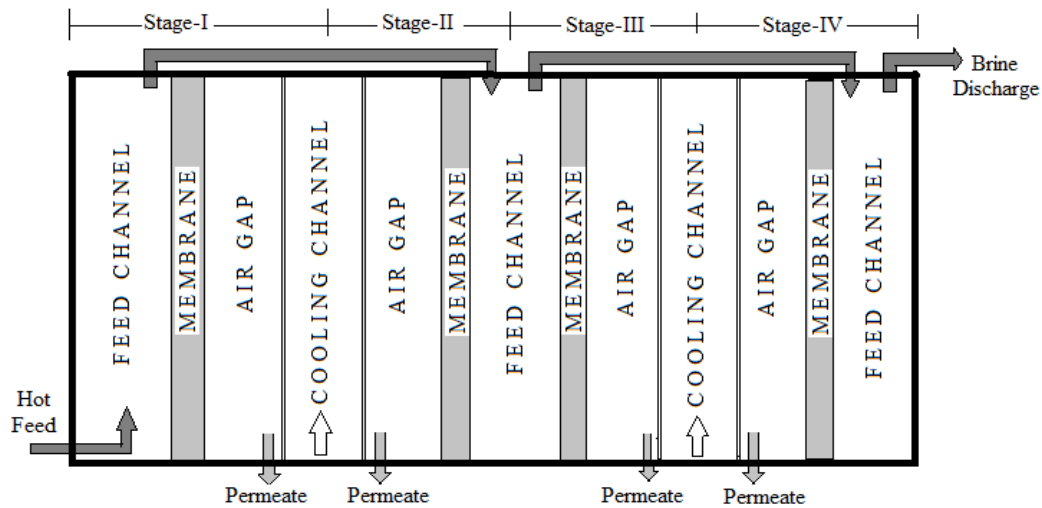


Fig. 1 Different internal channels of 4-stage ME-AGMD module

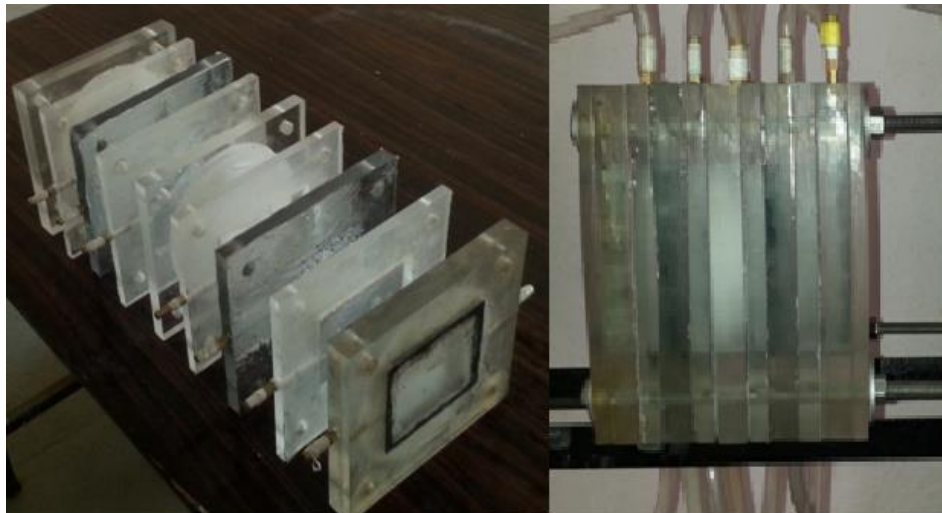


Fig. 2 (a) Picture of internal arrangement and (b) Picture of assembled 4-stage ME-AGMD module

the external heat is supplied to the second feed tank and passed in the first feed channel. The permeate flux is measured by collecting the permeate water of all stages in the measuring cylinder. The permeation rate is used to evaluate the performance of the module. The performance parameter indicators like permeate flux, separation efficiency, specific thermal energy consumption and GOR can be calculated by using the following equations.

The permeate flux, J_D (L/m^2h):

$$J_D = \frac{V}{At} \quad (1)$$

The separation efficiency of the module

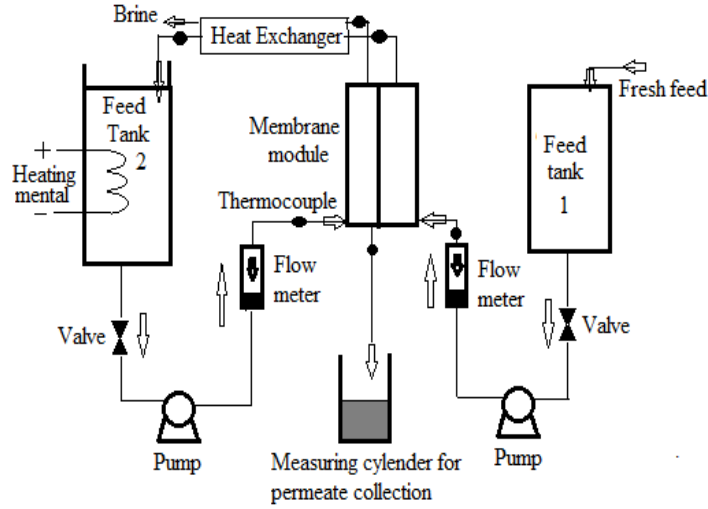


Fig. 3 Schematic diagram of 4-stage MEMD experimental setup

$$\% \text{Separation} = \frac{c_f - c_p}{c_f} \times 100 \quad (2)$$

The specific energy consumption without recovery of heat

$$\text{Specific energy consumption without heat recovery} = \frac{m_f c_{pf} (T_f - T_{fresh})}{m_D} \quad (3)$$

The specific energy consumption with recovery of heat

$$\text{Specific energy consumption with heat recovery} = \frac{m_f c_{pf} (T_f - T_0)}{m_D} \quad (4)$$

The gain output ratio (GOR) of the module after heat recovery

$$GOR = \frac{m_D \Delta H_v}{m_f C_{pf} (T_f - T_0)} \quad (5)$$

where V (L) is volume of permeate collection in time t (h), A (m^2) is membrane area, C_f and C_p is the concentration in feed and permeate respectively, m_f and m_D (Kg/s) is mass flow rate of feed and permeate water respectively, T_f , T_{fresh} and T_0 are the temperature of feed circulate through the 1st feed channel, fresh feed and feed after heat recovery (Latent heat of vaporization + sensible heat of brine) respectively, C_{pf} (KJ/kg °C) is specific heat capacity of water, ΔH_v (KJ/kg) is heat of vaporization of water.

2.4 Wastewater sample and analysis process

The wastewater samples were obtained from the nearby pesticide manufacturing industries located in the area of Nashik city, India. The fresh samples were used for physicochemical analysis

Table 1 Physicochemical analysis of feed wastewater sample

Parameter	Unit	Value
Ph	--	8.7
TDS	mg/l	5820
COD	mg/l	2530
TOC	mg/l	915
BOD	mg/l	630
TSS	mg/l	116
Conductivity	$\mu\text{s/cm}$	18350
Turbidity	NTU	166
Sulphate	mg/l	1516
Phosphate	mg/l	61
Cu	mg/l	22
Zn	mg/l	4.5
Fe	mg/l	18

and were stored at room temperature ($28 \pm 1^\circ\text{C}$). Table 1 shows the main physicochemical characteristics of the fresh feed wastewater sample which were analyzed in our laboratory. The water analysis kit (Systronics, 371) was used for the analysis of characteristics of the samples such as pH, total dissolved solid (TDS), turbidity and conductivity. The chemical oxygen method (COD) was measured by using the standard titration method (Methods 508 1975). The sulphate, phosphate and other metal ions analyzed by using the UV- spectrophotometer (Shimadzu UV-1800). Total organic carbon (TOC), total nitrogen (T-N) and biological oxygen demand (BOD) was measured in the environment laboratory located in Nashik City (India).

3. Results and discussion

3.1 Parametric study of 4-stage MEMD module

The performance of the 4-stage ME-AGMD module is evaluated by analyzing the permeate flux, specific energy consumption and gain output ratio (GOR) at maximum feed-coolant water temperature difference. The effect of the feed-coolant water temperature difference on the permeate flux in the 4-stage ME-AGMD process was shown in Fig. 4. The experiments were conducted for wastewater at a different feed temperature ($40\text{-}80^\circ\text{C}$) while the coolant temperature was kept constant at 28°C . The result shows that the permeate flux was enhanced due to increase of the feed-coolant water temperature difference. The trans-membrane driving force is the responsible for the production of permeate flux in the MD. Increase in feed-coolant water temperature difference, driving force is increased i.e., vapor pressure difference across the membrane which leads to increase the permeate flux. The temperature gradient increases across the membrane will positively effect on the diffusion coefficient which also leads to increase the vapor flux. In addition, the temperature polarization decreases with increasing the feed temperature. Hence the feed temperature is an important parameter to set in this process. In all the experiments the feed flow rate was kept constant about 0.5 L/min i.e. the Reynolds number was

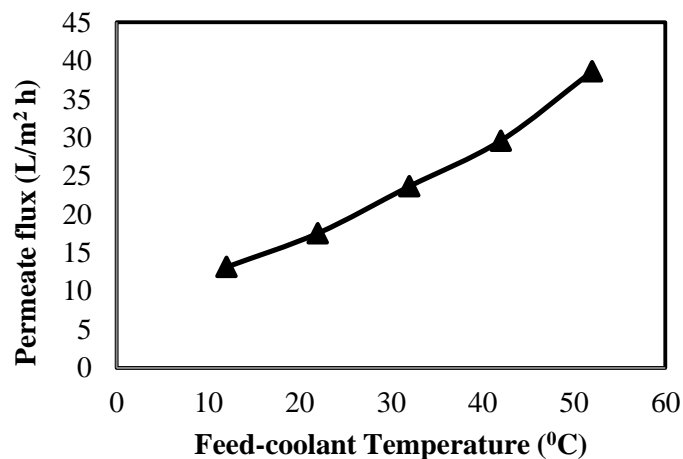


Fig. 4 Effect of feed-coolant water temperature difference on 4-stage MEMD flux at feed flow rate = 0.5 L/min, coolant flow rate in each channel = 0.25 L/min

Table 2 Effect of heat recovery on the performance of 4-stage ME-AGMD module in terms of GOR and specific energy consumption at feed-coolant water temperature difference about 52°C

	GOR	Specific energy consumption (kWh/kg)
Without heat recovery	0.425	1.48
With internal latent heat recovery	0.65	0.969
With latent + brine heat recovery	1.006	0.627

about 3182, and coolant flow rate was about 0.25 L/min in each cooling channel. The permeate flux was increased by about 195% when feed-coolant water temperature difference increased from 12-52°C. The maximum permeate flux of 4-stage ME-AGMD module was found to reach about 38.62 L/m²h at feed-coolant temperature difference about 52°C. The permeate flux obtained from each stage was found to decrease slightly due to the temperature drop in each stage of the module.

In this experiment, the fresh feed water temperature about 28°C was used as cooling water, hence there was no consumption of the cooling water and internal latent heat of vaporization to be transferred to the coolant channel via the condensation film. Also, the sensible heat of hot brine discharged from the module was recovered from the coolant water in the heat exchanger. Hence the specific energy requirement for this process was decreased by increasing the feed-coolant water temperature difference (Khan and Martin 2014). The specific energy consumption (kWh/kg) and GOR were estimated for without and with heat recovery.

Table 2 shows the specific energy consumption and GOR of 4-stage ME-AGMD process. This was determined at maximum feed-coolant water temperature difference of 52°C. The heat recovery is the key of the multi-effect process. In this module, the internal latent heat was recovered during the condensation of the water vapor in air gap channel. Due to the recovery of internal latent heat of vaporization the fresh feed (coolant) water temperature was increased. After that this coolant water was passed through the heat exchanger where exchange the sensible heat form the hot brine water. Again due to recovery of the sensible heat the feed water temperature was increased. If the temperature of the feed water is higher, then external energy requirement is

Table 3 Separation efficiency of 4-stage ME-AGMD module

Parameter	Permeate (mg/L)	% Removal
TDS	20.95	99.64
COD	26.06	98.97
TOC	2.2	99.76
TSS	0.16	99.86
Turbidity	1.99	98.8
Conductivity ($\mu\text{s}/\text{cm}$)	1.86	99.99
Sulphate	1	99.95
Phosphate	0.5	99.18
Cu	0.1	99.54
Zn	0.01	99.77
Fe	0.1	99.44

automatically decreases. Hence specific energy requirement for the ME-AGMD process was decreased due to the heat recovery as shown Table 2. The heat recovery is enhanced with higher driving forces. The GOR of the MD process greater than unity shows the good performance. In this module, the GOR was increased due to the heat recovery. The result shows, the specific energy consumption and GOR at without heat recovery is about 1.48 kWh/kg and 0.425 respectively. But after all heat recovery, the specific energy consumption decreases to 0.627 kWh/kg and GOR increases to 1.006. When the membrane area in the module increases, the water product rate also increases and GOR also increases due to higher production rate. Geng *et al.* (2015) obtained the GOR is 7.1 in AGMD after brine heat recovery and 88.2% water recovery after 14-stages of AGMD process. The high GOR and permeate flux, lower specific energy consumption gives the advantages of the ME-AGMD process for commercialization and industrialization of the process.

3.2 Water quality analysis and Separation efficiency of 4-stage MEMD module

Table 3 shows the main characteristic analysis of the permeate water. The COD removal efficiency of the water was about 98.97%. The TDS, TOC and TSS removal efficiency of the ME - AGMD process was approximately shown >99.6%. With respect to the turbidity separation efficiency, 98.64% reduction could be achieved by the ME-AGMD process. The conductivity of the permeate water was found to be 1.86 $\mu\text{s}/\text{cm}$. The result shown in Table 3 also shows the separation efficiency of the additional parameters of wastewater such as sulphate, phosphate, copper (Cu), zinc (Zn), iron (Fe) and found >99% separation efficiency of the ME-AGMD process. With this excellent performance of the ME-AGMD module for the treatment of the pesticide wastewater, the distillate produced is of high quality and could be reused in the industrial process.

3.3 Long term performance and membrane fouling of 4-stage MEMD module

Fig. 5 shows the permeate fluxes obtained by the 4-stage ME-AGMD process for pesticide wastewater as a function of time. In order to check the feasibility of ME-AGMD process for the

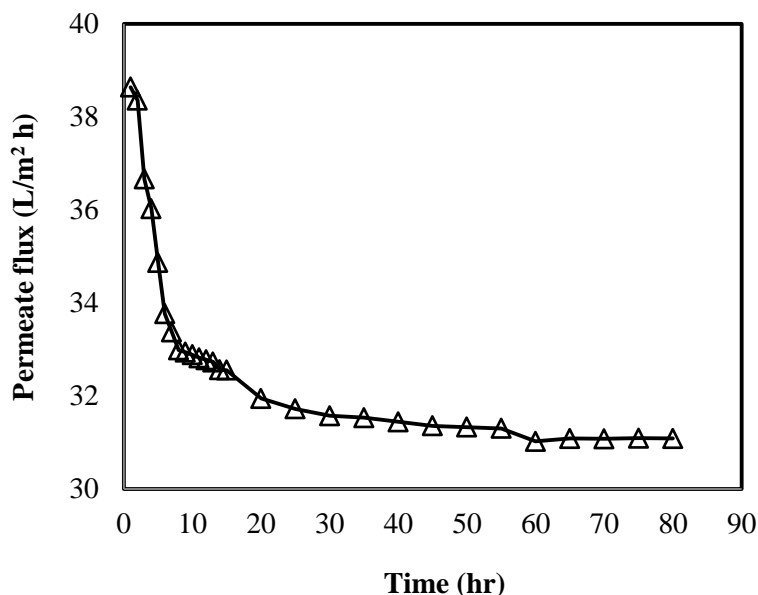


Fig. 5 Effect of time on permeate flux of 4-stage MEMD module at feed-coolant water temperature difference about 52°C

treatment of pesticide wastewater over a long period of time, the ME-AGMD experiment was carried out for 80 h continuously at feed and coolant temperature of 80°C and 28°C respectively. The fresh feed water was used as coolant water and the flow rate of each coolant channel was kept about 0.25 L/min. The feed flow rate was about 0.5 L/min. The initial permeate flux was recorded about 38.64 L/m² h. The permeate flux were decreased by 14.7% within the first initial period of 9 h continuous operations. Also the flux continuously decreases and it was more or less constant about 31.08 L/m² h. The total decline of the flux was about 19.54% within the period of 80 h operation. This flux decline is due to the feed becomes more concentrated and also due to the membrane fouling. Generally fouling is caused due to the deposition of soluble salts (Gryta 2008, He *et al.* 2008), biological compounds (Goh *et al.* 2013) and carbohydrates (Mokhtar *et al.* 2015) on the membrane surface. In this study, the fouling of membrane was caused due to the deposition of the soluble organic and inorganic compound on the surface of the membrane.

Some studies found that the fouling of membrane in the MD process can be controlled by the pre-treatment of feed and membrane cleaning along with the use of suitable MD conditions (El-Abbassi *et al.* 2013, Gryta 2010, 2007). But any pre-treatment method increases the cost of the overall process. The membrane cleaning is the simple and inexpensive method for controlling the membrane fouling. Because the loosely deposited of the salt/ crystal layer formed on the membrane surface due to lower operating pressure as compared to other pressure driven membrane technology such as reverse osmosis.

The maximum flux was decreased during the initial period of 9 h. So, the membrane cleaning cycle was fixed after every 9 h operation. The membrane cleaning was done by using DI water. The membrane was flush with the DI water, so the loose depositions of the salt/ crystals are removed from the membrane surface. After cleaning of the membrane the permeate flux of ME-AGMD was restored to an initial flux as shown in Fig. 6. The initial permeate flux and after

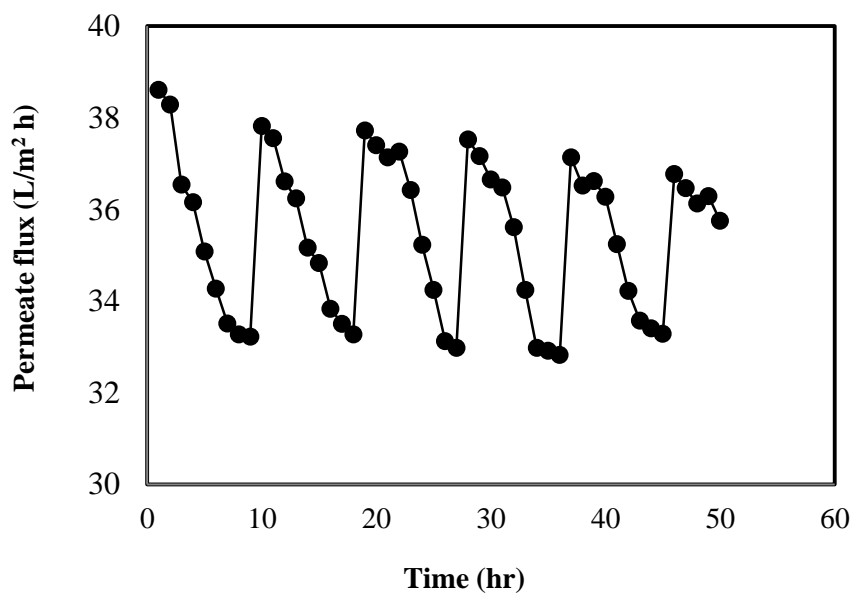


Fig. 6 Effect of time on permeate flux of 4-stage MEMD module when the membrane module washing after every 9 h operation cycle

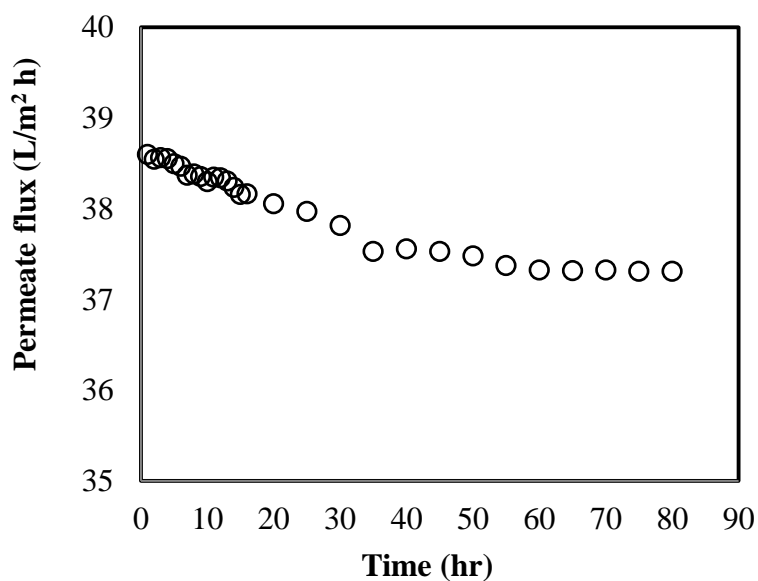


Fig. 7 Effect of time on permeate flux of 4-stage MEMD module when acidification of feed water (pH = 4) by using 0.1 M HCl solution

cleaning of membrane surface shows a variation less than 3%. Hence the fouling phenomena are highly reversible and can be easily removed the deposition from the membrane surface by using DI water washing of the membrane surface. The repetition of the module cleaning resulted in a

gradual decline of the maximum permeate flux and it is about 5.1% decreased after five times cleaning of the membrane module.

Fig. 7 shows the stability of the ME-AGMD process for the treatment of pesticide wastewater by acidification of the feed wastewater. The process was carried out at the initial feed pH about 4 adjusted by adding 0.1 M HCl to the feed wastewater. The result shows that the acidification of the feed enhances the stability of the process in a significant degree. The acidification of the feed wastewater was an efficient method for eliminating the negative effect of deposition of salt/ crystal on the membrane surface. The permeate flux was decreased about 3.54% during 80 h of continuous operation of the process. But, more work will be required for the evaluation of fouling phenomena in the ME-AGMD process for various industrial pesticide wastewater treatment.

4. Conclusions

In this study, the ME-AGMD process with internal latent heat recovery, sensible brine heat recovery, high water recovery and use of fresh feed water as a cooling water was studied successfully for the pesticide wastewater treatment. The maximum flux of the 4-stage ME-AGMD module reached to 38.62 L/m²h at feed-coolant water temperature difference of 52°C. The feed flow rate was maintained at 0.5 L/min. The fresh feed was used as a cooling water in this process for the purpose of recovery of the latent heat in each stage. The specific energy consumption and GOR of the module after the recovery of internal latent heat of vaporization and sensible heat of the brine was found to be 0.627 kWh/kg and 1.006 respectively. The permeate flux was decreased in 80 h continuous operation due to the membrane fouling. The inexpensive methods such as water washing cycle of the module and acidification of feed water (pH=4) gives the excellent performance in controlling the membrane fouling. The high quality of the pure water was obtained during the experiment via ME-AGMD process. The water quality parameters are found to be removed >98.8% from the pesticide wastewater. Hence this process can be recommended for the commercialization in the industrial wastewater treatment.

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