Advanced treatment of pharmaceutical and Tyre wastewater with the help of low-

pressure reverse osmosis membrane.

Tratamiento avanzado de aguas residuales farmacéuticas y de neumáticos con la

ayuda de una membrana de ósmosis inversa de baja presión.

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ABSTRACT

In recent years, accelerated growth of industries is hand to hand with the economic growth of developing countries. Industrial development positively impacts the living standards and economic sector, but also greater strains on available resources and severe threat to environment. Among all the industrial wastes, liquid discharge is the most dreadful concern at present. Increasingly stringent government water quality policies enhanced the necessity of proper effluent treatment technology with cost effectiveness and consistent operations. There are many methods for wastewater treatment but from last few decades, substantial use of reverse osmosis (RO) membrane for water recovery from effluent streams has strengthen the demand of different approaches for the desired efficiency of salt removal and elevated water recycle ratio. In this paper to overcome environmental problems, an effort has been made for advanced treatment of pharmaceutical and tyre industrial wastewater. The paper shows that membrane M1 when exposed to 1000 mg/l of Sodium Chloride shows the rejection of 97.46% with water flux of 33.73 (I/m²hr). Additionally, membrane M2 when exposed to 1000 mg/l of Sodium Chloride shows the rejection of 96.85% with water flux of 38.92 (I/m²hr). The results obtained highlights that the separation in RO resulted in appreciable removal of TDS from M1 are 88.31 % while from M2 is 89.23 % in case of pharmaceutical wastewater. Moreover, the performance of TDS separation in M1 is 86.69 % whilst from M2 is 87.27 % is main subject of interest in case of tyre wastewater. Based on the results obtained, it can be concluded that the RO has been effective in the reduction of COD, BOD with complete TSS removal.

Keywords: Pharmaceutical, Tyre, Wastewater, Treatment, Reverse osmosis.

RESUMEN

En los últimos años, el crecimiento acelerado de las industrias va de la mano del crecimiento económico de los países en desarrollo. El desarrollo industrial tiene un impacto positivo en los niveles de vida y el sector económico, pero también aumenta la presión sobre los recursos disponibles y amenaza gravemente al medio ambiente. Entre todos los desechos industriales, el vertido de líquidos es el que más preocupa en la actualidad. Las

políticas gubernamentales cada vez más estrictas sobre la calidad del agua aumentaron la necesidad de una tecnología adecuada de tratamiento de efluentes con rentabilidad y operaciones consistentes. Existen muchos métodos para el tratamiento de aguas residuales, pero en las últimas décadas, el uso sustancial de membranas de ósmosis inversa (RO) para la recuperación de agua de corrientes de efluentes ha fortalecido la demanda de diferentes enfoques para lograr la eficiencia deseada de eliminación de sal y una elevada tasa de reciclaje de agua. En este trabajo para superar los problemas ambientales, se ha hecho un esfuerzo por el tratamiento avanzado de aguas residuales industriales farmacéuticas y de neumáticos. El artículo muestra que la membrana M1 cuando se expone a 1000 mg/l de cloruro de sodio muestra un rechazo del 97,46% con un flujo de agua de 33,73 (l/m2hr). Además, la membrana M2 cuando se expone a 1000 mg/l de Cloruro de Sodio muestra un rechazo del 96,85% con un flujo de agua de 38,92 (l/m2hr). Los resultados obtenidos resaltan que la separación en RO resultó en una remoción apreciable de TDS del M1 son 88.31 % mientras que del M2 es 89.23 % en el caso de aguas residuales farmacéuticas. Además, el rendimiento de la separación de TDS en M1 es del 86,69 % mientras que en M2 es del 87,27 % y es el principal tema de interés en el caso de las aguas residuales de neumáticos. Con base en los resultados obtenidos se puede concluir que la RO ha sido efectiva en la reducción de DQO, DBO con eliminación completa de SST.

Palabras clave: Farmacéutica, Tiro, Aguas residuales, Tratamiento, Ósmosis inversa.

INTRODUCTION

Beyond doubt, it is well acceptable that freshwater access is the principal source of life and is crystal clear requirement of humankind for its domestic and industrial utilization. In recent years, with every four times increase in world's population, water crises have increased up to seven times and emerged as worldwide critical and an important global issue (Mayyahi et al., 2018). In view of this scarcity, there has been an overwhelming alarm to produce clean water from treatment of industrial wastewater and sea water (Elimelech et al., 2016).

In recent times, the expansion of industries has become a major part of a country's economy (Timotius et al., 2021). India being a state is hub in number of industries. However, with the progressive industrial growth, the natural pollution is becoming a serious environmental threats as different types of pollutants are being discharged directly or indirectly into nearby outlet (Nahiun et al., 2021). Wastewater generated by different pharmaceutical, personal care, and drug industries as well as in tyre and rubber industries contains many chemicals, solvents, enormous solid and hazardous wastes (Jayalekshmi et al., 2021). Discharging these effluents by conventional approaches is not merely unsafe but also does not fulfill the current state and central pollution control board guidelines (Dhote et al., 2012). In accordance, there is crucial requirement of minimization of high contamination effluent loads and environmental pollution through complete treatment of industrial wastewater (David et al., 2016, Mohidus et al., 2006). In fact, the wastewater recovery for further recycle or reuse whereby fulfilling many rigid standards of discharge is a key requisite.

The Indian pharmaceutical manufacturing industry produces an array of bulk drugs encapsulating various ingredients, raw material, and solvents in manufacture, extraction, processing, purification, and packaging processes employing many inorganic, organic and biological reactions mostly in Active Pharmaceutical Ingredient sites (Gadipelly et al., 2014). These pharmaceutical solids and liquids product are used as a valuable life-giving remedy for living beings (Sudhir et al., 2006).

Fundamental pharmaceutical wastewater treatment scheme as shown in Figure-1 as untreated effluent for primary treatment is firstly feed to equalization tank whereby dosing of Alum, Poly aluminum chloride (PAC), Polyelectrolyte (PE) is added to increase the settling process and further clear stagnant layer of water from primary clarifier is send to aeration tank and settled sludge is send to decanter. In secondary mode of treatment, in aeration tank with the help of diffusers dissolved oxygen level has been maintained which is essential for microbial growth and in next clear stagnant layer of water from secondary clarifier is send to V- notch and some part of settled sludge is continuously recycle back to aeration tank for maintaining mixed liquor suspended solid level whereas remaining part of sludge is send to decanter. From V-notch, the measured flow rate of water is sent for tertiary treatment to reverse osmosis plant. The obtained recycled water is used as feed for cooling tower and rejected water is send to multiple effect evaporator for further treatment.



Figure- 1: Fundamental Pharmaceutical Effluent Treatment Plant Scheme

Tyre manufacturing industry consumes larger amount of water, solvents, chemicals, and other utilities depending on different stages of processes (Mohammadi et al., 2010). Generally, in all types of tyre industry, foremost concern is related to environment as they cause huge number of wastes and effluent (Mohamadreza et al., 2015). For that reason, in accordance with high contamination load, the comprehensive effluent treatment is pivotal.

Complete tyre effluent treatment scheme as shown in Figure 2. Effluent treatment plant here consists of pretreatment, secondary treatment and advanced treatment which complete the ZLD scheme. Untreated effluent passes through screen chamber for preliminary treatment, then send for removal of oil and grease and next to dosing tank. In primary mode of treatment, firstly dosing of Lime, Alum, Poly aluminum chloride (PAC) are done to enhance the settling process and in equalization tank holding time is provided. Further clear layer of water from primary clarifier is send to aeration tank and the settled sludge is send for disposal. In secondary mode of treatment, in aeration tank with the help of diffusers dissolved oxygen level has been maintained which is essential for microbial growth and in next clear stagnant layer of water from secondary clarifier is send to reverse osmosis plant and some part of settled sludge is continuously recycle back to aeration tank for maintaining mixed liquor suspended solid level whereas remaining part of sludge is send for disposal. Tertiary mode of treatment occurs in reverse osmosis plant and the obtained recycled water is used as feed to cooling tower as well as the rejected water is sent to multiple effect evaporator for further treatment.



Figure-2: Fundamental Tyre Effluent Treatment Plant Scheme

As a result of rapid development and industrialization, an enhanced golden opportunity for water recycling and reuse basically for developing countries like India (Padalkar et al., 2018, Pangarkar et al., 2011). Nowadays, Reverse osmosis (RO) has made a breakthrough as a promising powerful and greener technology for retrieval of wastewater without any phase change, its simpler operation, lower energy consumption, higher separation capability and least chemical requirements (Ghanbari et al., 2015, Malaeb et al., 2011). In fact, over the last few years, RO has surpassed energy intensive classical thermal applied sciences processes such as multi-stage, multiflash etc. (Yaqub et al., 2019, Mehta et al., 2018). Furthermore, different membrane processes, like ultrafiltration (UF), microfiltration (MF), nano-filtration (NF), also reverse osmosis (RO), are applied on industrial levels for the separation purposes (Ghaffar et al., 2017, Rana et al., 2010). In addition, the different membrane processes are energy efficient, environmentally friendly and are operated in harsh conditions (Cheremisinoff et al., 2017, Dong et al., 2012). Figure-3 shows the complete classification of different membrane separation techniques along with the different parameters as pore size, pressure, and species rejection.



Figure-3: Different Membrane Separation processes with size and application.

Several works have been published on environmental issues and reverse osmosis applications for the treatment of complex concentrated water obtained from industrial effluents (Muftah et al., 2010, Subramani et al., 2014). The modern desalination industry requires treatment methods with high productivity. Among all the different methods, reverse osmosis is the crucial separation process with significant progress using hydrostatic pressure as a driving force to separate solute from solution (Dong et al., 2012, Ismail et al., 2017). Thin Film Composite (TFC) and Polyamide (PA) membranes has successfully fulfilled this criterion owing to its low hydraulic resistance (Ghanbari et al., 2015, Kulkarni et al., 1996, Cadotte et al., 1981). However, the composition and criticality of the feed water sources help in determining the type of pretreatment required (Thamaraiselvan et al., 2015, Choudhury et al., 2018). Eventually, reverse osmosis filtration process is used for the removal of multivalent, divalent ions, dissolved organic matters and to maintain the chemical oxygen demand (COD), total organic carbon (TOC), and biological oxygen demand (BOD) in required range (Zhaohuan Mai, et al., 2022, Pandey et al., 2012).

The objective of the present work is to demonstrate the Pharmaceutical and Tyre industrial wastewater treatment with the help of low-pressure reverse osmosis membranes. The wastewater treatment can be significantly achieved by reverse osmosis processes fulfilling dual objective of minimization in volume of wastewater as well as generation of good quality of wastewater fulfilling legal requirements. Experimental

MATERIAL AND METHODS

Two sets of membranes were purchased and used for testing viz. standard polyamide thin-film composite (TFC) spiral-wound RO membrane element BW60-1812-75 (DOW FILMTECH) and industrial TFC DTRO membrane cushion (RO CHEM) respectively. BW60-1812-75 has pressure range of 50 psig with 99% salt rejection whereas in RO CHEM membrane the working pressure is 100 psig with average salt rejection of 98.50 %. Laboratory grade (LR) Sodium chloride (Himedia laboratories Pvt. Ltd.) and Isopropanol (Rankem laboratory reagents) were used.

Experimental setup design: A laboratory bench scale cross flow RO test cell was fabricated from a local manufacturer. The membrane test unit consists of a PVC membrane cell, pump, pressure measuring meter and a feed reservoir. The membrane cell consisted of a circular plate-and-frame unit, which contained a flat membrane sheet placed on a Top Cell. Both the top and bottom discs of cell are each of 75 mm diameter and 55 mm thickness. The bottom plate was milled to accommodate a 47 mm diameter sintered plate that fitted flush with its top surface.

The laboratory bench scale cross flow RO test kit consists of the following parts as shown in Figure 4:

Feed vessel with capacity of 5-10 liter.

Diaphragm pump (Model no. VT-100-1-A) is used to pump the feed at a pressure of 90-110 psi.

Pressure Gauge of 0-160 psig.

Reverse osmosis cell- 75 mm diameter and 55 mm thickness (dimensions)



Figure-4: Schematic diagram of laboratory scale cross flow RO test kit cell

The flat-sheet membrane was cut and fitted into a 47 mm disc and positioned upon the sintered plate. The effective surface area of the membrane is 0.001734 m². A 3 mm thick Rubber gasket was placed over the top to clamp the membrane in position, produce a hydraulic seal and provide a space between the membranes. The permeate flow rate was measured with the help of measuring cylinder per unit time. Permeate and concentrated water were recirculated back to the feed reservoir. Figure 5 shows snapshots of the Lab Scale Cross flow RO membrane cell setup.

Evaluation of wastewater properties: The wastewater from pharmaceutical and tyre was analyzed for Chemical Oxygen Demand (COD), Biological Oxygen Demand (BOD), pH, Total Suspended Solids (TSS) and Total Dissolved Solids (TDS). COD was analyzed by digester (Spectra lab 2015 M1) using APHA AWWA-05 5220B method. BOD was determined through incubator (VELP Scientific FTC 120) by IS: 3025 (Part-44) method. pH was measured employing Hanna (HI-8314) meter, TDS was determined by conductivity meter (CON-700) and TSS was obtained manually with the help of Whatman filter paper NO. 41/42 using APHA AWWA-05 2540D method.



Figure- 5: Snapshots of (A) Top Cell (B) Bottom Cell (C) Side View of RO Membrane Cell Setup (D) Lab Scale Cross flow RO membrane cell setup.

Membrane Performance: The membrane performances were investigated on the designed cross flow membrane testing kit with circular membrane coupons of 0.001734 m² area at operating pressure of 90 psig at 25°C feed water temperature after compacting the membranes at 100 psig pressure for 20 minutes. Permeate flux and solute rejection for both the membranes were determined with NaCl solution at 2000 mg/L concentration. The selectivity or salt rejection % (R) of the membranes were calculated using the following equation.

R= $(1 - C_p/C_f)$ *100(i)

Here, C_p is conductivity of permeate, $C_{f is}$ conductivity of feed.

Additionally, the permeate flux (J_v) were determined by dividing the permeate volume collected per unit time on a unit membrane area (I/m^2hr) in the standard crossflow membrane testing kit. Treatment performances of both the reverse osmosis membranes were evaluated with different industrial wastewater samples obtained from pharmaceutical and tyre industries respectively.

Fouling behavior with both pharmaceutical and tyre industrial wastewater were performed for eight hours at 100 psig in recycle mode and moreover the flux decline of fouled membrane was measured by collecting each hour permeate. Finally, the Flux Recovery Ratio (FRR) was determined by measuring the pure water flux before fouling (J_{w1}) at 90 psig as well as with after fouling membranes cleaning with water (J_{w2}) .

FRR (%) = (J_{w2}/J_{w1}) *100.....(ii)

RESULTS AND DISCUSSION

Membrane Performance (Salt-rejection and Water-flux): The performance of the fabricated reverse osmosis cross flow setup kit was tested in terms of different membrane performance as water flux and salt rejection as well as with respect to wastewater treatment efficiency. Separation performances in terms of water-flux and salt-rejection of both the membranes viz. M1 (DOW FILMTECH) and M2 (RO CHEM) were found out after 10 min compacting sample in the designed reverse osmosis membrane kit at 90 psig pressure and tested for brackish water concentration of 1000 mg/l of Sodium Chloride. From Figure 6: it can be seen that the membrane M1 when exposed to 1000 mg/l of Sodium Chloride shows the rejection of 97.46% with water flux of 33.73 (I/m²hr).



Figure 6: Separation performance of Sodium Chloride through M1 (DOW FILMTECH) at 1000 mg/l.



Figure 7: Separation performance of Sodium Chloride through M2 (RO CHEM) at 1000 mg/l.

Similarly, from Figure 7: It can be seen that the membrane M2 when exposed to 1000 mg/l of Sodium Chloride shows the rejection of 96.85% with water flux of 38.92 ($Lm^{-2} hr^{-2}$)

Reverse Osmosis membrane performance in wastewater treatment

Treatment efficiency of Pharmaceutical industry waste water

Pharmaceutical wastewater usually arises from the synthesis and formulation of variety of drugs. As a result of different industrial products many types of effluents are generated in composition depending on production rate, processes etc. [30]. Table 1. Shows the results obtained from analysis of nearby collected pharmaceutical wastewater samples at inlet wastewater for treatment and at reverse osmosis feed respectively.

Parameters	Inlet wastewater	Reverse osmosis feed
рН	6.0-7.2	6.8-7.2
TSS (mg/L)	50-150	12-16
TDS (mg/L)	1300-1700	1162-1300
COD (mg/L)	1000-1100	560-720
BOD (mg/L)	500-650	30-40

Significant parameters have been considered while feeding wastewater to reverse osmosis for removing variety of dissolved impurities such as inorganic salts and organic matter. After sequential operational treatment (as shown in Figure 1) wastewater is feed to reverse osmosis. Table 2. Shows the wastewater composition at reverse osmosis inlet, after treatment through M1, after treatment through M2 and permissible limit respectively.

Table 2. Pharmaceutical	wastewater com	position of reverse	osmosis feed, afte	r treatment through M1
and M2 along with the permissible	limits.			

Parameters	Reverse osmosis	After M1	After	M2	Permissible Limit
	feed	wastewater	wastewater		(As per CPCB norms)
рН	7.2	7.2	7.2		6.0-7.5
TSS (mg/L)	15	0	0		100
TDS (mg/L)	1198	140	129		2100
COD (mg/L)	648	154	150		250
BOD (mg/L)	30	20	19		30

The results as shown in Figure 8 prove that the significant reduction in pollution load after treatment. It can be seen that there is decline in parameters as pH, TSS, TDS, COD, and BOD along with complete TSS removal. The appreciable removal is shown in case of TDS from M1 is 88.31 % while from M2 is 89.23 %. The overall results gave an increase in COD reduction from M1 to 76.23% and M2 to 76.85%. Similarly, BOD reduction of 33.33 % from M1 and 36.67% from M2 respectively.



Figure 8: The reduction in various parameters after treatment through M1 & M2.

Reverse Osmosis membrane Fouling Behavior in Pharmaceutical wastewaterMembrane fouling resistance was studied with the industrial pharmaceutical wastewater by analyzing the corresponding decline in flux every hour at 90 psig for 10 hr. as shown in Figure 9. Table 3 display the permeate water flux of first hour & ten hours respectively along with the percentage decrease observed in permeate water flux at the end of ten hours. From this experiment, it was seen that the permeate flux decline in case of M1 was 46.80% while with M2 was 33.18 %. Flux Recovery ratio was found out by measuring the water permeate flux after fouling the membrane and cleaning with distilled water and water permeate flux before fouling and with fresh water. Moreover, the Flux Recovery Ratio obtained for M1 was 85.56 and with M2 was 81.75; proves that the non-degradable pollutants get accumulated causing fouling and decreases the rejection. This can be concluded that the wastewater contains a lot of organic compounds resulting in fouling.



Figure 9: Permeate flux with pharmaceutical wastewater fouling through M1 & M2.

Membrane	l st hour Flux (L/m ² .hr)	10 th hour Flux (L/m ² .hr)	% decline in water flux after 10 hr (L/m ² .hr)
M1	32.53	17.30	46.80
M2	38.58	25.78	33.18

Table 3 display the permeate water flux of first hour & ten hours respectively, and decrease observed in permeate water flux at the end of ten hours of experiment.

It can be seen that the main foulants in pharmaceutical industry wastewater contributing fouling are organic load. Hence, pharmaceutical wastewater consists of high number of organic pollutants, saline compounds and bio toxic which makes it's of high COD load. RO membranes are effective in removal of organic as well as inorganic impurities and for active pharmaceutical compounds. However, due to deposition of non-degraded foulants resulted in fouling and decline the treated product water.

Treatment efficiency of Tyre industry wastewater: Tyre products are manufactured in various stages and there are huge concerns of pollution during operational stages [34]. Appropriate and cost-effective treatment methods are vital in dealing with major water pollutants as shown in Table 4 after laboratory analysis of nearby collected tyre wastewater samples of inlet wastewater for treatment and at reverse osmosis feed respectively.

Parameters	Inlet wastewater	Reverse osmosis feed
рН	7.5-7.7	6.9-7.5
TSS (mg/L)	165-170	20-40
TDS (mg/L)	1800-1980	1300-1400
COD (mg/L)	260-300	70-100
BOD (mg/L)	70-90	20-30

Table 4. Composition of Tyre wastewater generated.

To achieve the removal of a variety of dissolved impurities such as inorganic salts and organic matter; wastewater in tyre industries are feed to reverse osmosis. The variety of certain major parameters considered for removal from reverse osmosis are TSS, TDS and to some extent of COD as well as BOD after secondary treatment (as shown in Figure 2). Table 5. Shows the wastewater composition at reverse osmosis inlet, after treatment through M1 and M2.

Table 5. Tyre wastewater composition of reverse osmosis feed, after treatment through M1 and M2 along with the permissible limits.

Parameters	Reverse osmosis feed	After M1 wastewater	After M2 wastewater	Permissible Limit (As per CPCB norms)
рН	7.5	7.5	7.5	6.5-8.5
TSS (mg/L)	34	0	0	100
TDS (mg/L)	1390	185	177	2100
COD (mg/L)	72	23	21	100
BOD (mg/L)	24	10	9	30

The results as shown in Figure 10 prove that the significant reduction in pollution load after treatment. It can be seen that there is decline in parameters as pH, TSS, TDS, COD, and BOD along with complete TSS removal. The remarkable separation especially in case of TDS from M1 is 86.69 % while from M2 is 87.27 % is main subject of interest. This paper indicated on the whole an enhanced COD reduction from M1 to 68.06 % and M2 to 70.83%. Further, BOD reduction of around 58.33 % from M1 and 62.50% from M2 respectively.



Figure 10: The reduction in various parameters after treatment through M1 & M2.

Membrane Fouling Behavior in Tyre wastewater: Membrane fouling resistance was checked with the industrial tyre wastewater by analyzing the corresponding decline in flux every hour at 90 psig for 10 hr. as shown in Figure 11. Table 6 display the permeate water flux of first hour & ten hours respectively along with the percentage decrease observed in permeate water flux at the end of ten hours. From this experiment, it was seen that the permeate flux decline in case of M1 was 43.23% while with M2 was 30.93%. The Flux Recovery Ratio obtained for M1 was 82.90 and with M2 was 80.44, it can be concluded that the wastewater contains a lot of TDS resulting in fouling.

Table 6 Permeate water flux of first hour & ten hours respectively, and decrease observed in permeate water flux at the end of ten hours of experiment.

Membrane	l st hour Flux (L/m ² .hr)	10 th hour Flux (L/m ² .hr)	% decline in water flux after 10 hr. (L/m ² .hr)
M1	32.01	18.17	43.23
M2	36.33	25.09	30.93

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Figure 11: Permeate flux with tyre wastewater fouling through M1 & M2.

It can be seen that the main foulants in tyre industry wastewater contributing fouling are inorganic load. Hence, tyre wastewater consists of large amount of TDS, SS with small amount of carbohydrates, protein, lipids, and carotenoids. RO membranes are effective in removal of inorganic impurities, but due to deposition of nondegraded foulants resulted in fouling and decline the treated product water.

As conclusion, this paper extensively complies and proves the specific requirements of effective treatment of pharmaceutical and tyre industrial wastewater fulfilling the discharge quality goals. The effective treatment of both the industrial wastewater is of global interest which can be fulfilled by reverse osmosis treatment. Keeping in view, the mentioned facts both membranes have shown the significant reduction in total dissolved solids, organic pollutants, COD, BOD and total removal of suspended impurities. It was concluded after series of experiments that the M2 membranes shows better treatment efficiency with better flux and rejection.

Membrane M1 when tested to 1000 mg/l of Sodium Chloride shows about 1% more rejection than M2 while in flux the M2 shows 5.19 (l/m²hr) more flux than M1.

The results obtained highlights that the separation in RO resulted in appreciable removal of TDS from M1 are 88.31 % while from M2 is 89.23 % in case of pharmaceutical wastewater. Moreover, the performance of TDS separation in M1 is 86.69 % whilst from M2 is 87.27 % is main subject of interest in case of tyre wastewater.

Additionally, based on the excellent results another important consideration is overall reduction in other significant parameters of wastewater as chemical oxygen demand, biological oxygen demand as well as completely removal of turbidity.

The flux recovery ratio shows the less fouling tendency in case of M2. This present work clearly indicates the noteworthiness of reverse osmosis membrane processes as an advanced treatment in industrial wastewater treatment with more energy saving and economically viable solution.

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