

# TREATMENT OF DISTILLERY SPENT WASH BY COMBINED UF AND RO PROCESSES

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## ABSTRACT

The present study deals with the treatment of distillery spent wash by an ultrafiltration (UF) membrane followed by reverse osmosis (RO) membrane on a pilot scale membrane setup. The performance of the system was evaluated by varying applied pressure on the thin-film composite polyamide (TFC-PA) UF and RO membranes. In the first stage, UF experiments are carried out for concentration of effluents by removing the suspended solids (SS). RO has effectively separated the total dissolved solids (TDS), chemical oxygen demand (COD), biochemical oxygen demand (BOD), sulphate and potassium with the rejection efficiency of 97.9%, 96.8%, 97.9%, 99.7% and 94.65%, respectively. UF was effective for the separation of SS with the rejection efficiency of 95.5%. The pollutant level in permeate are below the maximum contaminant level as per the guidelines of the World Health Organization and the Central Pollution Control Board (CPCB) of India specifications for effluent discharge (less than 1000 ppm of TDS and 500 ppm of COD).

KEYWORDS: Distillery effluent; Spentwash; Ultrafiltration; Reverse osmosis.

## 1. INTRODUCTION

The Indian distillery units use sugarcane molasses as a preferred raw material because of its easy and large scale availability. There are about 579 sugar mills and 295 distilleries in India. Alcohol is produced from molasses by two types of fermentation processes, Praj type and Alfa Laval distillation. In Praj type, for one liter of alcohol produced, about 12-15 liters of spentwash is generated, whereas, in the Alfa Laval continuous fermentation and distillation process only 7-8 liters of wastewater per liter of alcohol is produced as it uses evaporators for concentrating the effluent. Currently, about 40.72 million m<sup>3</sup> of spentwash is generated annually from distilleries alone in India (Saha et al., 2005; Pant and Adholeya, 2007; Belkacemi et al., 2000; Dahiya et al., 2001; Sangave and Pandit, 2006). Ground water is the main water source for these operations. Significant volume of water is consumed for molasses preparation, yeast propagation etc. in the range of 14–22 I I<sup>-1</sup> of alcohol production. The production and characteristics of spentwash are highly variable and dependent on feed stocks and various aspects of the ethanol production process. The spentwash is acidic (pH 3.94 - 4.30) dark brown liquid with high BOD (45000-100000 mg 1) and COD (90000-210000 mg  $\Gamma^{1}$ ), and emits obnoxious odour. Although it does not contain toxic substances, its discharge without any treatment brings about immediate discolouration and depletion of dissolved oxygen in the receiving water streams, posing serious threat to the aquatic flora and fauna (Mane et al., 2006). Membrane based separation processes like ultrafiltration (UF), nanofiltration (NF), reverse osmosis (RO) and membrane bioreactor (MBR) have been applied for treating distillery effluent (Nataraj et al., 2006; Couallier et al., 2006; Zhang et al.,

2006). Distilleries are one of the 17 most polluting industries listed by the Central Pollution Control Board (CPCB) of India (CPCB, 2003). Indian distilleries employ various forms of primary, secondary and tertiary treatments of wastewater. The typical treatment sequence is screening and equalization, followed by biomethanation. Ferti-irrigation and biocomposting with sugarcane pressmud are the most widely used options for effluent disposal (Ramana et al., 2002). However, these methods are highly energy intensive and hence quite expensive. These disadvantages emphasized the need for further research using novel separation methods.

The main objective of this study is to purify the wastewater by removing the colour and the contaminants by using UF and RO membranes. Experiments are conducted at different operating pressures. The results are presented for the waste effluent samples collected from the local distillery industries.

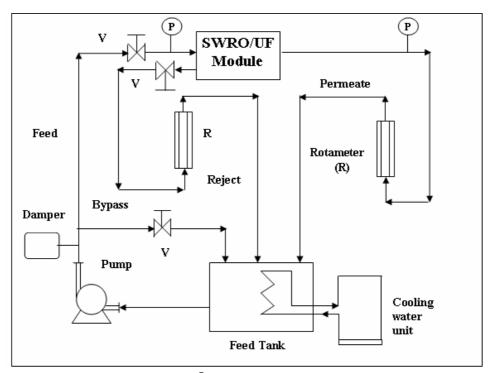
#### 2. MATERIALS AND METHODS

The experiments are performed on a Perma<sup>®</sup>-pilot scale membrane system (Permionics, Vadodara, India) consisting of a spiral-wound TFC-PA UF and RO membrane modules. These membranes have three layers. The first layer (also known as skin layer) is of  $5 - 20 \,\mu m$ thick TFC-PA layer that does the actual separation. The second layer is made of polysulfone of about 50 µm thickness, which provides support to the first layer (skin layer) as a substrate for cross-linking of composite layer. The third layer, used for resistance and strength, is made of polyester with a thickness of about 200 µm. The Perma-TFC membranes are capable of withstanding pH in the range 2–12, pressure up to 30 atm and temperatures up to 50°C with an effective area of 1.0 m<sup>2</sup>, module length 21 inches and diameter 2.5 inches. The experimental set-up is shown in Figure 1. A damper is provided to regulate the flow fluctuation due to the reciprocating action of the pump. A feed tank of 25 I capacity, made of stainless steel-316 is provided for storage and supply of effluent to the system as well as collection of the recycled concentrate. A cooling coil is installed inside the feed tank for circulating cold water to maintain constant feed temperature within the range of 28–30°C. The effluent sample is collected from the local distillery industry situated in south Gujarat, India. Twenty five liters of the spentwash feed is poured in the feed tank after thoroughly cleaning the membrane systems and wetting with deionized water. The high pressure reciprocating pump is employed to transport the feed to the spiral wound membrane module. With a control valve, the retentate flow rate is maintained constant (15 I min<sup>-1</sup>) throughout the experiments, to ensure the steady hydrodynamic conditions inside the membrane module. Fouling of the membrane surface is the bane of membrane operations. Fouling layers are removed by using appropriate cleaning. The CaCO<sub>3</sub> scaling is controlled by treating the membrane at the end of the day's study with dilute HCl solution, which converts the carbonate to CO<sub>2</sub> and also removes other metal precipitates and mineral scales. To prevent biological fouling, the membrane is washed thoroughly with deionized water and stored in a 0.5% solution of sodium bisulfate (NaHSO<sub>3</sub>) at the experiment runs. Washing with a 1-1.5% aqueous solution of tetrasodium EDTA, a chelating agent useful in removing organics and silt, is done on alternate days. The important parameters like colour, pH, suspended solids (SS), total dissolved solids (TDS), COD, BOD, potassium, sulfate and chlorides are measured for the feed and permeate samples, according to standard methods (Clesceri et al., 1998).

In RO/NF/UF membrane processes, separation performance of the membrane is judged by the percent observed solute rejection ( $R_o$ ) of SS, COD, TDS and other feed components which is calculate as (Murthy and Gupta, 1997):

$$\% R_o = \left(1 - \frac{C_p}{C_f}\right) \times 100 \tag{1}$$

where  $C_f$  and  $C_p$  are the individual component concentrations in the incoming feed to the membrane system and in the permeate from the system, respectively.



*Figure 1.* Perma<sup>®</sup>-pilot scale membrane system

## 3. RESULTS AND DISCUSSION

#### 3.1 UF experiments

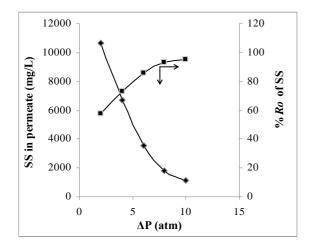
The characteristics of the distillery spent wash used for this study are given in Table 1. In the first stage, UF experiments were carried out for concentration of effluents by removing the suspended solids.

Parameter	Unit	Value
рН	-	4.01
SS	mg l⁻¹	25040
TDS	mg l⁻¹	38140
COD	mg l⁻¹	125000
Colour	Pt. Co. Scale	800
Chloride	mg l⁻¹	59981
Sulphate	mg l⁻¹	25920
BOD	mg l <sup>-1</sup>	41946
Potassium	mg l⁻¹	4360

Table 1. Characteristics of distillery spent wash

UF has effectively separated the SS from 25040 to 1127 mg l<sup>-1</sup>; COD from 125000 to 4600 ppm at pressure 10 atm, with  $R_o$  of 95.5% and 63%, respectively. Effect of pressure on the removal efficiency of SS and COD by UF is shown through Figures 2 – 3.

It can be seen from Figures 2 and 3 that the percentage rejection of SS increased from 57.3 to 95.5% with increasing the pressure from 2 to 10 atm, while COD rejection increased from 26.4 to 63%. With the pressure range of 2-10 atm, experimental results show that the removal of BOD is from 41946 to 24127 ppm, colour from 800 to 398 Pt-Co unit and potassium from 4360 to 2674 ppm with  $R_o$  being 42%, 50% and 39%, respectively. There was a marginal reduction by UF in TDS from 38140 to 33561 ppm with  $R_o$  of 12%. Effect of feed pressure on the removal efficiency of different ions through UF module is shown in Table 2.



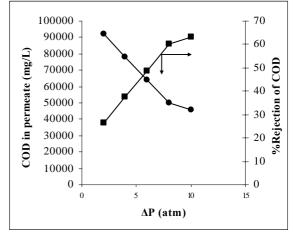


Figure 2. Removal of SS as a function of pressure with UF process

Figure 3. Removal of COD as a function of pressure with UF process

Table 2. Effect of pressure on UF permeate characteristics							
Pressure	SS	COD	Colour	Chlorides	Sulphate	BOD	K⁺
(atm)	(mg l⁻¹)	(mg l⁻¹)	(Pt. Co. Scale)	(mg l⁻¹)	(mg l⁻¹)	(mg l <sup>-1</sup> )	(mg l <sup>-1</sup> )
2	10678	92000	724	52430	18792	27342	4165
4	6700	78000	560	50895	12160	36829	3854
6	3521	64000	501	48212	6840	26124	3420
8	1780	50000	430	47585	3200	25161	3056
10	1127	46000	398	45120	2164	24127	2674

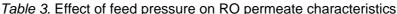
#### 3.2 RO experiments

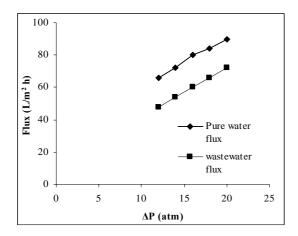
In the second stage, permeate obtained from the UF experiment was used as a feed for RO experiments. Several parameters have been studied with RO. A comparison of pure water flux, when distilled water and spent wash were used as feed, is shown in Figure 4, which clearly demonstrates the effect of osmotic pressure in RO transport. The flux for feed increased linearly from 12-20 atm. The comparison shows that the performance of the membrane was not affected by the fouling. In the pressure range of 12-20 atm, permeate was transparent. Effect of pressure on the removal efficiency of COD by RO is shown in Figure 5. It can be seen from Figure 5 that the  $R_o$  of COD increased from 91.3 to 96.8%. Effect of feed pressure on the removal efficiency of MO module is shown in Table 3.

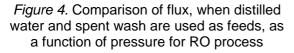
It can be seen from Figures 6–9 and Table 3 that the TDS permeate concentration was reduced from 33,561 to 68 mg  $\Gamma^1$  ( $R_o$  =97.9%), BOD from 24,127 to 493 mg  $\Gamma^1$  ( $R_o$  =97.9%), colour from 398 to 27 Pt-Co unit ( $R_o$  =93.2%), chlorides from 45,120 to 76 mg  $\Gamma^1$  ( $R_o$  =99.8%), sulphate from 2,164 to 6.4 mg  $\Gamma^1$  ( $R_o$  = 99.7%) and potassium from 2,674 to 143 mg  $\Gamma^1$  ( $R_o$  = 94.65%).

Similar results were obtained by Nataraj *et al.* (2006), but with a hybrid NF and RO processes for the removal of colour and contaminants in the distillery spent wash and for a good water recovery rate. The reclaimed water by NF and RO processes could be reused for either domestic or industrial purposes. This hybrid process has proven its potential technological applicability for treating domestic and other wastewaters, by producing excellent effluent quality for safe and environmentally begins discharge, even under variable feed conditions (Couallier *et al.*, 2006).

Pressure	SS	COD	Colour (Pt. Co.	Chlorides	Sulphate	BOD	K⁺
(atm)	(mg l <sup>-1</sup> )	(mg l⁻¹)	Scale)	(mg l⁻¹)	(mg l⁻¹)	(mg l⁻¹)	(mg l⁻¹)
12	431	4000	42	297.9	25.6	1333.3	718
16	240	2400	38	124	2.6	774.1	302
20	94	1480	27	75.97	6.4	493.3	143







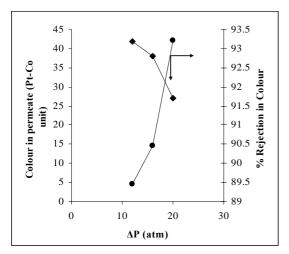


Figure 6. Removal of Colour as a function of pressure with RO process

# 4. CONCLUSIONS

The present membrane based separation study demonstrated that the UF and RO processes can be successfully used for the removal of colour and other contaminants from the distillery effluents. In the present case the best operating conditions for RO were observed to be 15 L/min feed flowrate and 20 atm applied pressure, which gave the rejection efficiency of 97.9% and 96.8% for TDS and COD, respectively. The absence of heat application and a high rejection efficiency by RO shows that clean water can be produced economically instead of being vaporized by the energy intensive evaporation process. Recycling purified water could also enable to spare a major part of the ground water used in the distilleries at the fermentation stage. The main interest of recycling water is to be related with the management of the effluents by reducing fresh water consumption and wastewater treatment costs, small

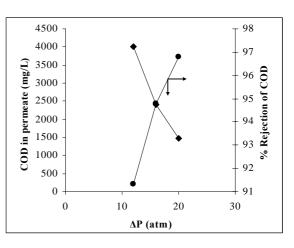
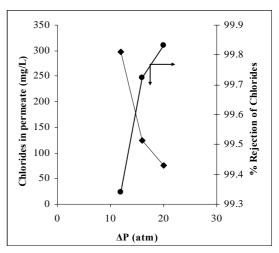


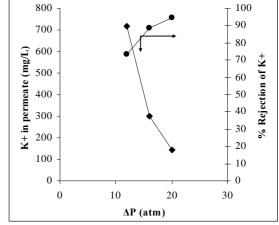
Figure 5. Removal of COD as a function of pressure with RO process



*Figure 7.* Removal of Chlorides as a function of pressure with RO process

1400 98.5 98 1200 97.5 BOD in permeate (mg/L) BOD 1000 97 800 ection of 96.5 96 600 Rej 95 5 400 ~ 95 200 94.5 0 94 10 20 30 0 ΔP (atm)

Figure 8. BOD removal as a function of pressure for RO process



*Figure 9.* Potassium removal as a function of pressure for RO process

#### ACKNOWLEDGEMENTS

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disposal volumes which will minimize the waste disposal costs and reduction in regulatory

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pressure.