Reclamation of end-of-pipe textile effluent using low energy membrane systems

P. Xaba[1], S. Rathilal[1], L Pillay[2]
[1]Durban University of Technology, Department of Chemical Engineering, Durban, South Africa
[2]Univesity of Stellenbosch, Faculty of Engineering, Stellenbosch, South Africa

Abstract

In this study, the reclamation of end-of-pipe textile effluent originating from a reactive dyeing textile mill was investigated using low energy membrane based processes. Effluent quality, salt recovery and membrane recovery were the main parameters used to evaluate the membrane process. Flat sheets of the SR90 and NF90 Dow FilmTech nanofiltration (NF) membranes were used on a pilot scale membrane system. The UF multipore membrane was used in the pretreatment runs. The quality of reclaimed water was measured against the specified water quality in terms of the internal criteria for effluent recycle (ICFER) provided by the mill. It was required to optimize the SR90 and NF90 membrane performances so as to set operating parameters for pilot study. The average critical flux for the SR90 membrane was found to be ±31 LMH at 5 bar TMP when directly feeding the effluent without any pretreatment. The average critical flux for the NF90 membrane was found to be approximately ±15 LMH at 10 bar TMP when no pretreatment was used. All rejections were found to be >90% for all specified parameters as per Table 1. The quality results from the SR90 and NF90 membranes with pretreatment showed that the permeate could be recycled and reused in the dying processes within the mill. It was however concluded that the NF90 membrane produces high quality effluent as compared to the SR90. It was of importance to determine if the SR90 and NF90 membranes could be recovered and reused after directly feeding un-pretreated feed effluent. The flux recoveries on the SR90 and NF90 membranes were 84.72% and 82.17%, respectively after chemical cleaning. It was further concluded that the salt initially fed in the dying process could be recovered from the waste stream especially when using the SR90 membrane for reuse. However, the colour parameter was found to be slightly out of specification when using the SR90 membrane. To improve the colour of effluent produced when using the SR90, it was recommended that a granular activated carbon filter needs to be employed downstream of the process to polish the effluent.

Keywords: Pollution control, Membrane technology, Textile effluent

1. Introduction

The textile processing industry encompasses a diverse group of organizations that process and produce textile related products. The production of textile products involves processes like preparation (winding, warping, sizing and knitting/weaving), dyeing, washing, finishing and drying. These processes have different water usages depending on the material being processed (Agenson and Urase, 2007). Large amounts of dyestuffs, reagents, salts and other chemicals are used in different stages during the processing of these products. Resulting effluents generally contain suspended solids, dissolved solids, salts, dye and other chemicals (Al-Amoudi and Lovitt, 2007). However, the dye house (dyeing stage) produces highly polluted effluent when comparing it to the other processes. To minimize the amount of pollutants found in these effluent streams, several operating methods have been set up and implemented (Blanpain-Avet et al, 2009) (Calvo et al, 2011) & (Fersi, C. and Dhahbi, M. 2008). Such methods include the use of less concentrated dyeing chemicals, discontinuation of run-off rinsing and separation of hot and cold effluent (Blanpain-Avet et al, 2009) (Fersi and Dhahbi, 2008). These manufacturing methods have proved to be significant in reducing the organic and inorganic load of reactants but the resulting effluent still does not meet municipal discharge specifications. Numerous wastewater treatment methods have been established to treat and remove dyes from textile effluent (Robinson et al, 2001), (Wolmarans, 2011). These methods are divided into chemical degradation techniques, physical, biological and mechanical treatment (Sojka-Ledakowicz et al, 1998). These methods have been found to be inefficient due to operating costs, labor skill requirements and overall quality effectiveness (Petrinića et al, 2007), (Wolmarans, 2011). Most recently, reverse osmosis has been used to treat these effluents, however, due to its extensive energy use and the fact that it removes all salts (including useful salts) it has been found to be inefficient. The development of nanofiltration (NF) which uses significantly less energy and potentially recovers the needed salts has been seen to bring promising solutions (Wolmarans, 2011), (Liikanen et al, 2002), (Robinson et al, 2001). This project was undertaken to investigate the feasibility of using NF membrane based processes in the treatment of end-of-pipe textile effluent. The option of using an ultrafiltration (UF) membrane as a pretreatment to nanofiltration was also investigated. Treated water ready for the dyeing process was analyzed and used as a permeate quality indicator. It was termed internal criteria for effluent recycling (ICFER) measure for the treatment.

2. Materials and methods

2.1 Effluent and descriptions

Three different end-of- pipe textile effluents were processed using UF and NF membranes. The first effluent was termed EFF1, second effluent was termed EFF2 and the third effluent was termed EFF3. All these effluents were sampled independent of each other at different time intervals. A treated effluent sample was taken from the mill to determine quality requirements to be met by the UF/NF systems. The qualitative analysis was termed ICFER (internal criteria for effluent recovery). The permeate from EFF1 was termed P1 OR Perm1, from EFF2 was termed P2 OR Perm 2 and from EFF3 was termed P3 OR Perm3.

2.2 Experimental approach

The experimental work composed of two stages i.e. pretreatment using UF and filtration using NF. The effluent from the mill was analyzed for quality prior to it being pretreated. The UF test rig was designed with three membranes in parallel, however during optimization only one membrane was used since the same performance was expected and the other two membranes were therefore only used for validation purpose. It was important to determine optimum operating conditions in terms of operating pressure, temperature and filtration time. At the second stage, the NF membranes were characterized using pure water and the effluent was used to determine critical operating parameters for the quality and productivity experiments. The NF membranes were first run without effluent pretreatment while in the last stage; they were run with pretreated effluent from the UF.

2.3 Experimental setup

2.3.1 Ultrafiltration experiments

The membranes used were first characterized for permeability by running pure water to determine the pure water flux (PWF). The operating pressure was 100 kPa. The membrane pore size was 0.2 µm. Refer to Figure 2 for the process flow diagram.

5L of pure water was fed into the feed tank. The pumping speed was set to 100% and pure water was circulated to and from the feed water tank while adjusting the back pressure to 100 kPa. Permeate was sampled in 15 minute time intervals for a total period of 2 hours. The permeate volume collected over the 15 minute period was measured.

2.3.2 Operational procedure for NF experimentation

The same operating procedure was followed when characterizing both the SR90 and NF90 membranes. Refer to Figure 1 for the process flow diagram of NF test rig. Membranes were first cut to size and placed in purified water for 24 hours. Purified water was filled into the NF feed tank. Individual membranes which were soaked for 24 hours were fitted into the membrane cell and the cell was secured in the NF rig. The cooling system was started and the membrane unit was switched on. The operating pressure was adjusted to 2.5 bar. Permeate was collected in 10-15 minute time intervals for a total period of 2-3 hours. Differential pressure across the NF membrane was varied with 2.5 bar increments until maximum pressure of 14 bar was reached. The permeate volume collected over the 15 minute period was measured and the experiment was repeated for repeatability and statistical analysis.

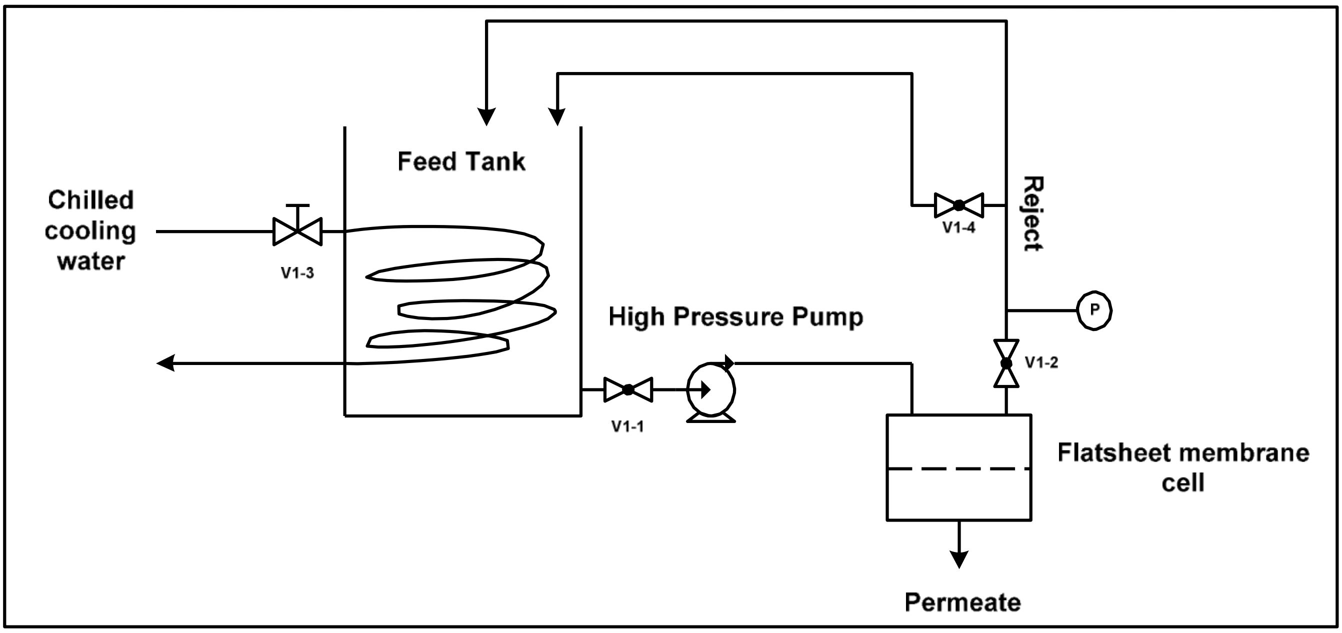

Figure 1: Nanofiltration test rig setup

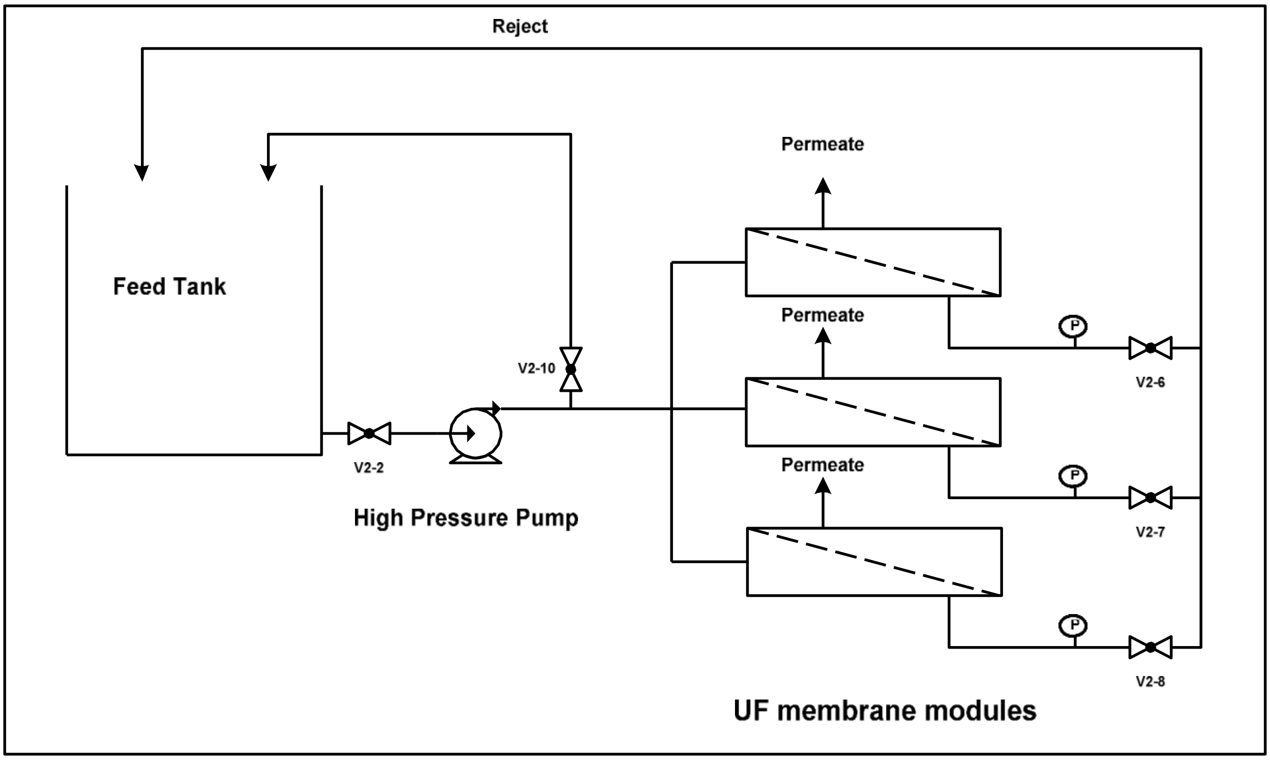

Figure 2: Ultrafiltration test rig setup

2.4 Analytical Instruments and analysis methods

Table 1: Instruments and analytical methods

Parameter	Analytical Instrument	Analytical method
*COD	DR 3900 Benchtop Spectrophotometer	****USEPA Reactor Digestion Method
Colour	DR 3900 Benchtop Spectrophotometer	Platinum-Cobalt Standard Method
Conductivity	Hach SensION5	Direct method
pH	pH meter	Standard method
**TSS	DR 3900 Benchtop Spectrophotometer	Photometric Method
***TOC	DR 3900 Benchtop Spectrophotometer	Platinum-Cobalt method
*****TDS	Hach SensION5	Direct method
Turbidity	Turbidmeter	Direct method

*COD –Chemical Oxygen Demand, **TSS – Total suspended solids, ***TOC- Total organic compound, **** USEPA - United States Environmental Protection Agency, *****TDS – Total dissolved solids

3. Results and Discussion

3.1 Feed and permeate analysis for the NF membranes with pretreatment

Table 2 and Table 3 shows the results found when the UF membrane was used to pre-treat effluent EFF1, EFF2 and EFF3 prior to the NF membranes. The pretreated effluent was used as feed to the SR90 and NF90 membranes. The permeate quality was compared to the specified quality specified by MTM in the form of the ICFER (internal criteria for effluent recovery) specification for both Table 2 and Table 3. The optimized pressures of 5 bar and 10 bar were used for the SR90 and NF90 membranes and 1 bar for UF membrane respectively. The feed temperatures were kept at ±18 ºC in both membrane processes.

Permeate COD's of both the SR90 and NF90 were found to be on specification except for permeate Perm 2 of the SR90 membrane. When comparing the COD reduction rates of the SR90 and the NF90 membranes, it was noted that the differences could be theoretically justified by the secondary layer formed by dye molecules on the membrane and the individual intrinsic properties of the membranes. It was expected that higher COD rejections could be achieved after pretreating the raw feed effluent with the UF membrane.

The improvement in COD reduction was also attributed to the fact that the UF membrane had removed larger organic pollutants and the NF membranes did not have heavy pollution loads to reject. The specified permeate colour was 0 PtCo and it was seen that only the NF90 membrane was able to meet the specification. The highest permeate colour was found to be 0.9 PtCo while processing EFF2 using the SR90 membrane. The lowest SR90 permeate colour was 0.23 PtCo. Even though the SR90 did not meet the specification, it was seen that the UF had improved the colour reduction rate.

Permeate conductivities from both the SR90 and NF90 membranes were all within specification as the specified conductivity was <86.4 µS/cm. When comparing the conductivity of Perm 1, Perm 2 and Perm 3 in both membranes, it was seen that as the conductivity was increasing, the rejection rate was decreasing. This decrease in rejection was attributed to the shield effect caused by increased feed salt concentration as both membrane systems were operated in cross flow circulation modes.

Different authors have reported that major problems in treating effluent with high TDS rise from the fact that there is accumulation of dissolved solids on membrane surfaces which accelerates fouling thereby decreasing the membrane performance (Robinson et al, 2001). (Calvo et al, 2011), (Robinson et al, 2001), (BTTG, 1999) reported that even though permeate cannot potentially have low levels of TDS, organic compounds and colour, it can still be recycled in the processing plant for different purposes.

It was noted that the feed turbidity's for EFF1 and EFF3 were in specification before the effluents were processed using the SR90 and NF90 membranes. The final turbidity of P2 was found to be 0.92 NTU for the SR90 membrane and 0.65 NTU for the NF90 membrane. The 0.92 NTU was out of specification as the specified turbidity range was 0-0.9 NTU.

The permeate TOC from the SR90 and NF90 membranes were all within specification as the specified value was <31 mg/L. The TSS of the SR90 membrane was found to be out of specification in all three permeates. The TSS of Perm 1 and Perm 2 from the NF90 was out of specification while that of Perm 3 was on specification. (BTTG, 1999) reported that the reuse criteria in terms of COD, conductivity and colour was specified to be <80 mg/L,

<100 mS/m and 20 PtCo respectively. When comparing the quality of permeate found experimentally with that specified by (BTTG, 1999). It was seen that the quality of permeate produced met the specifications and could be reused in the dyeing process.

The UF membrane qualitatively improved the performances of the SR90 and NF90 membranes. It was expected that the improvement rates in all quality parameters were going to be low since the UF membrane was not able to reduce smaller pollutants such as dissolved dyes, dissolved solids and dissolved salts. However it was expected that the UF membrane would significantly reduce the fouling rate of both membranes since the pollution loads were decreased.

Table 2: Comparison of UF+ SR90-400 permeate and the specified water quality

Parameters	**Units**	**UF permeate (NF-feed)**			**SR90 NF Permeate**			**ICFER Analysis**
		EFF1	**EFF2**	**EFF3**	**Perm 1**	**Perm 2**	**Perm 3**	
COD	mg/L	181	184	176	14	17	12.3	<15
Colour	*PtCo	483	456	422	0.6	0.9	0.23	0
Conductivity	**µS/cm	4390	4510	4103	44.5	53.2	44.1	<86.4
TDS	mg/L	2200	3100	2183	20.1	24.3	21.6	<44.2
Turbidity	NTU	0.82	24.3	0.76	0.41	0.92	0.47	0-0.9
TOC	mg/L	0.6	85	0.63	0.49	7.6	0.37	<31
pH		9.6	9.7	9.6	6.5	6.1	6	6.5-8.4
TSS	mg/L	13	34	13.2	0.9	1.3	0.51	<0

*PtCo – Platinum-cobalt, **µS/cm – Micro-Siemens per centimeter

3.2 Performance of the SR90 and NF90 membrane with pretreatment

Figure 3 shows the performance of the SR90 membrane in terms of flux when the feed of EFF1, EFF2 and EFF3 was pretreated using the UF membrane. The average flux while processing the effluent was found to be 44.94 LMH and the flux improved by an average of 5.40 LMH. The slight decline in fluxes was attributed to the fact that the dyes were being adsorbed onto the surfaces of the membrane and the same phenomenon was explained by (Agenson and Urase, 2007) when explaining the decline in flux which could not be fully accounted for.

Figure 4 shows the relationship between flux and time while processing the prefiltered effluent EFF1, EFF2 and EFF3 with the UF membrane. All three effluents were processed for 3 hours and it was noted that all three graphs were seen to have remained constant with insignificant flux decline. The lowest flux was found when EFF2 was processed. The average flux over the 3 hour filtration time was 18.6 LMH.

Table 3: Comparison of UF+ NF90 permeate and the specified water quality

Parameters	Units	UF permeate (NF-feed)			SR90 NF Permeate			ICFER Analysis
		EFF1	EFF2	EFF3	Perm 1	Perm 2	Perm 3	
COD	mg/L	181	184	176	11	10.3	8.3	<15
Colour	*PtCo	483	456	422	0	0.3	0	0
Conductivity	**µS/cm	4390	4510	4103	28.3	50.4	25.3	<86.4
TDS	mg/L	2200	3100	2183	13.6	25.1	11.6	<44.2
Turbidity	NTU	0.82	24.3	0.76	0.43	0.65	0.49	0-0.9
TOC	mg/L	0.6	85	0.63	0.29	1.4	0.12	<31
pH		9.6	9.7	9.6	6.5	6.5	6.1	6.5-8.4
TSS	mg/L	13	34	13.2	3	0.3	0	<0

*PtCo – Platinum-cobalt, **µS/cm – Micro-Siemens per centimetre

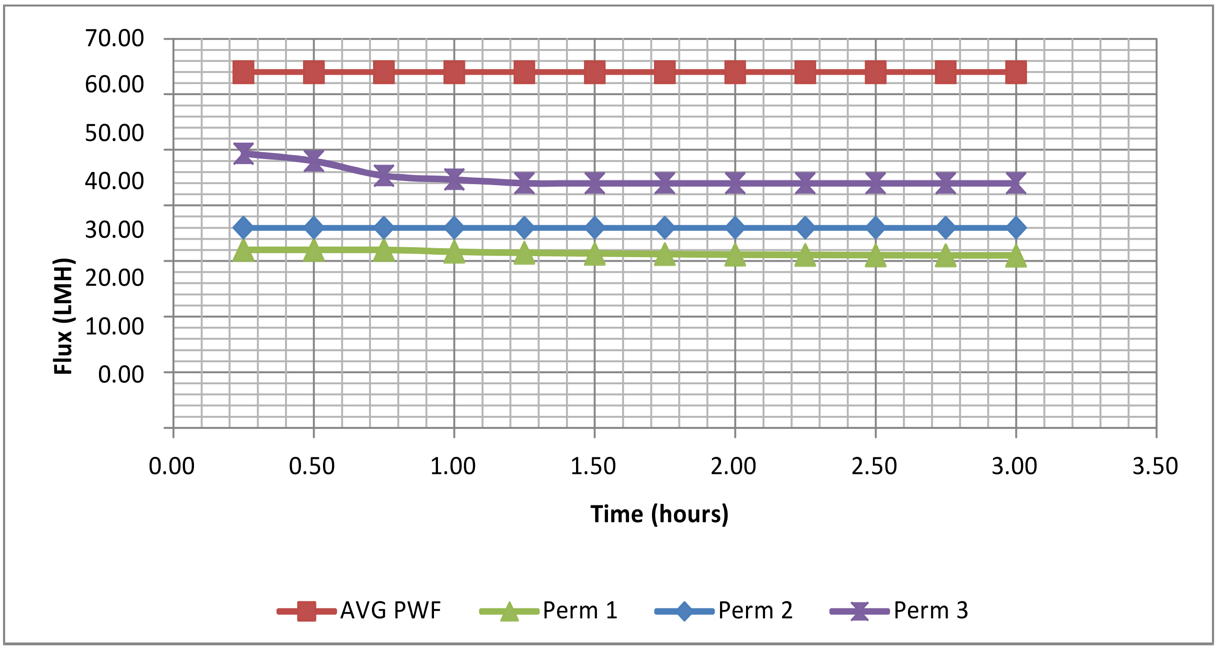

Figure 3: UF + SR90 membrane performance using effluent EFF1, EFF2 and EFF3 at 18ºC

It was noted that the average flux when processing EFF1 was 30.6 LMH. The flux when processing EFF1 had improved by 19.56 LMH. The improvement in the Perm 1 flux meant that EFF1 had high concentration of pollutants which were rejected by the UF membrane such that when the NF90 membrane was used, the major pollutants were already rejected.

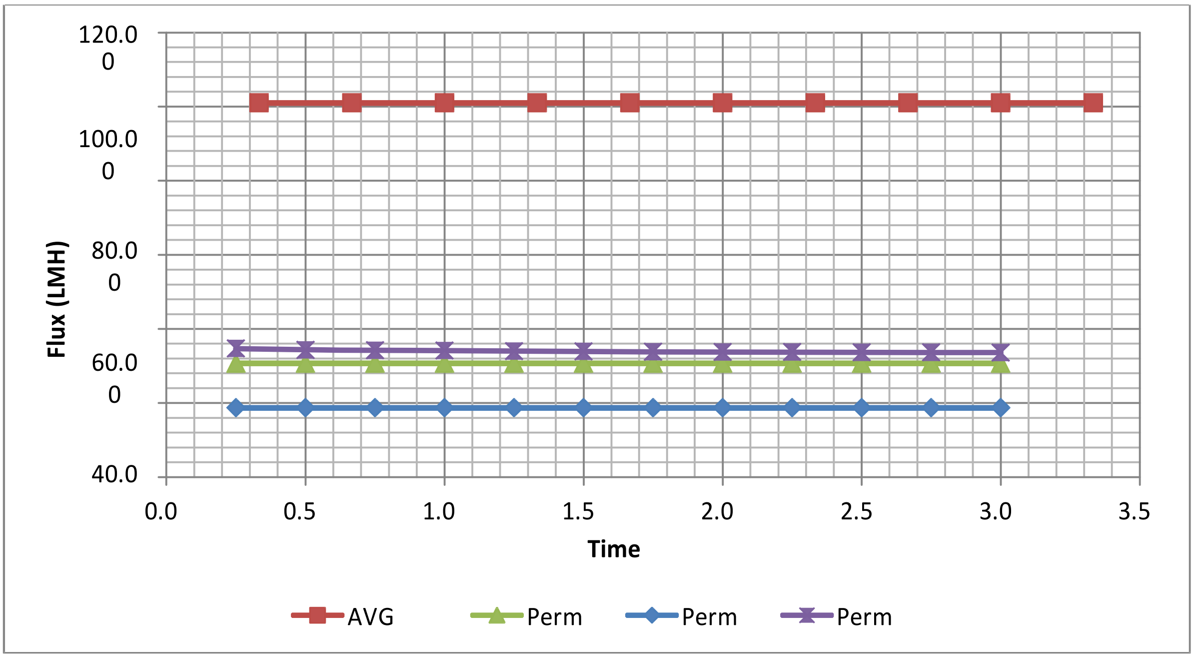

Figure 4: UF + NF90 membrane performance using effluent EFF1, EFF2 and EFF3 at 18 ºC

3.3 Membrane cleanability and recovery

Figure 5 shows the relationship between flux and pressure. Two effluent processing runs and two membrane cleaning runs were performed to establish flux recovery of the SR90 membrane.

The average flux decline was around 15.27%. It was seen that the membrane flux could not be recovered completely. Only 84.72% of the flux was recovered after the membrane was cleaned on the first run while processing EFF2. After the membrane was cleaned, it was used to process EFF2 on the second run. The flux was seen to have been stable at 6.5 bar rather than the optimized 5 bar. After processing the second run of EFF2, the membrane was cleaned. After the second run cleaning, the average flux recovered after the second run was 70%.

Figure 6 shows the relationship between flux and pressure while processing EFF2. The parameter which remained constant for the duration of this experiment was the feed temperature at 18ºC. EFF2 was not pretreated; it was used directly with no pretreatment. The membrane was chemically cleaned after processing EFF2 on the first run. The average flux recovery was 82.5%. The cleaned membrane was subjected to the second run of EFF2 and the stable flux was 10.1LMH at 10.5bar pressure. The membrane was then cleaned after the second run of EFF2 was processed. It was seen that the average flux recovery was at 59.8% after cleaning the membrane on the second run.

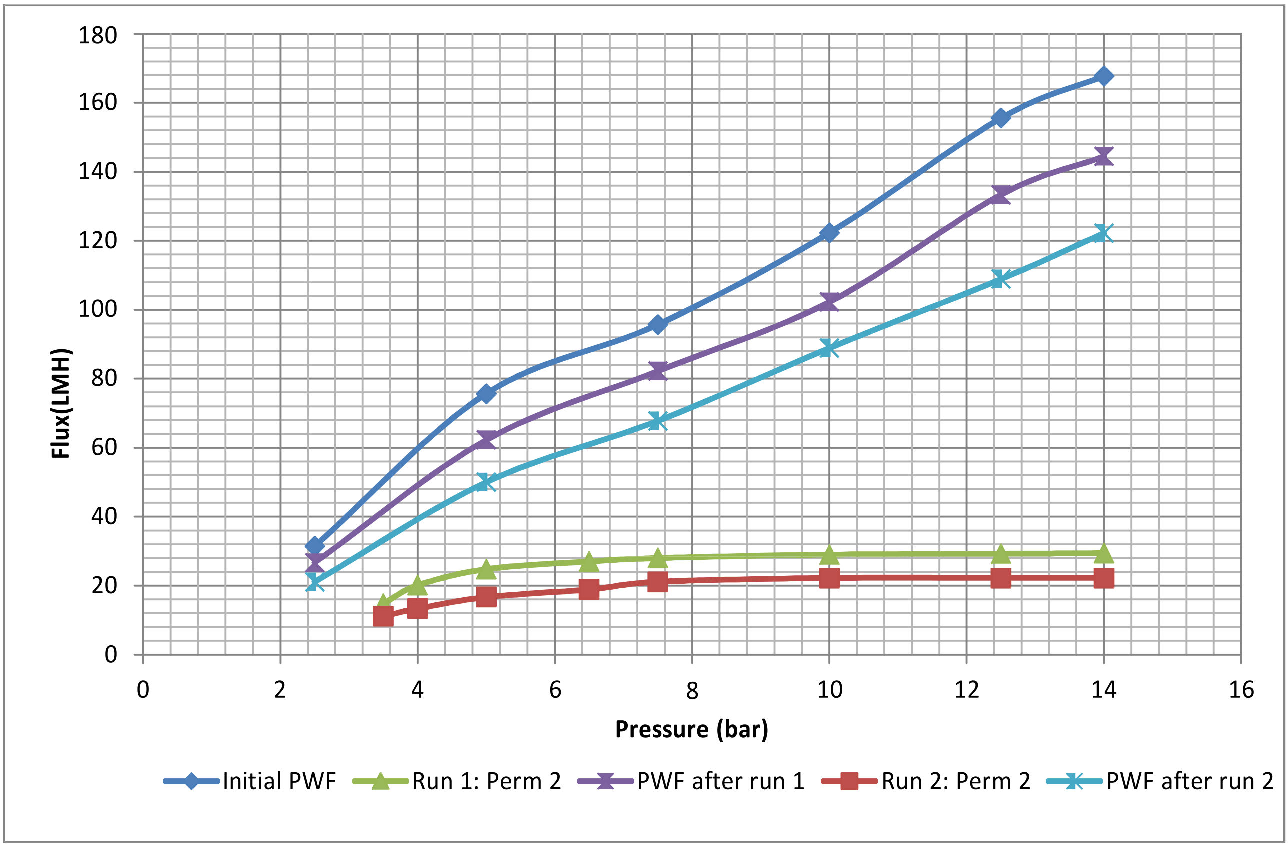

Figure 5: Variation of pressure on flux for the SR90 membrane

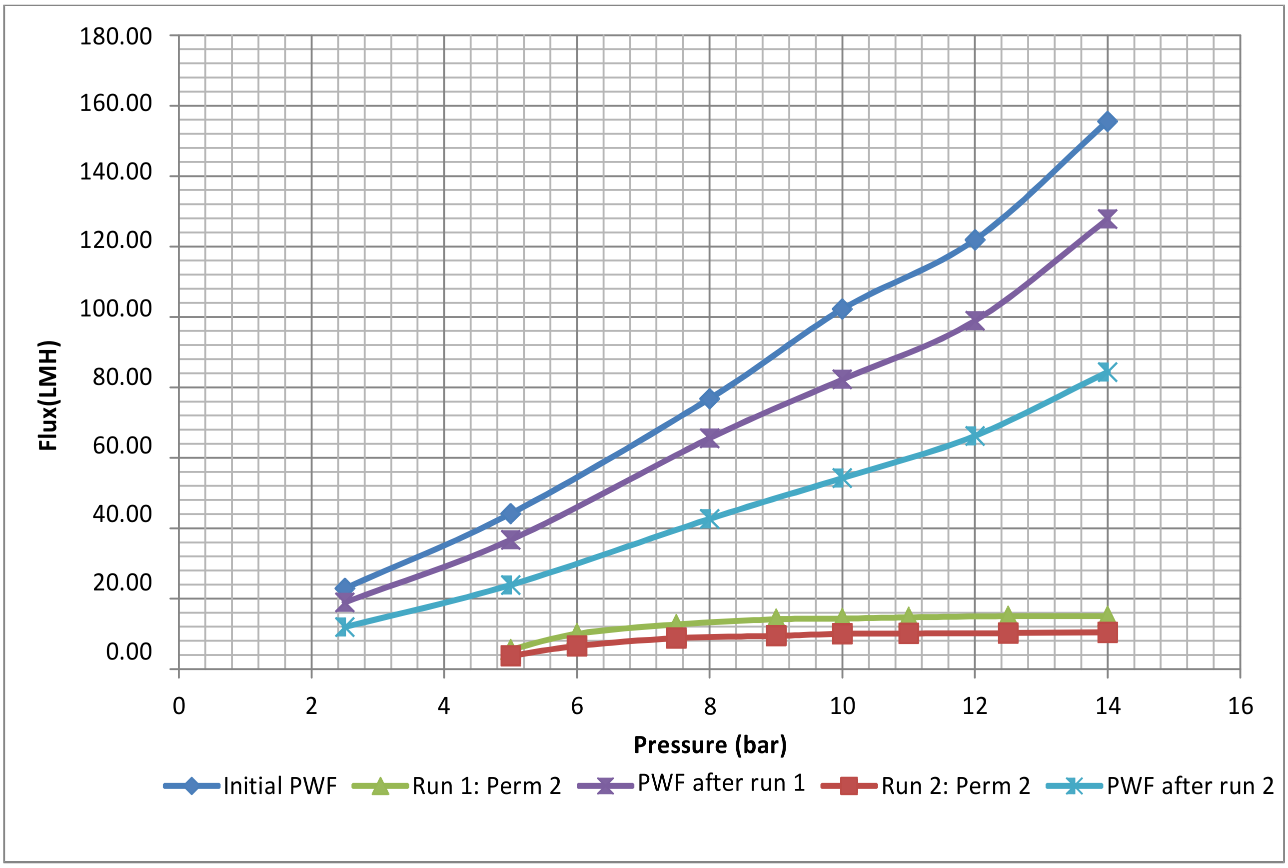

Figure 6: Variation of pressure on flux for the NF90 membrane

4. Conclusion

When comparing the fluxes of the SR90 and NF90 membranes with pretreatment and without pretreatment, it was seen that the SR90 membrane gave the highest flux. The specific fluxes for the SR90 membrane were higher than that of the NF90 membranes. Qualitatively, the NF90 membrane performed better than the SR90 membrane. Flux recoveries in both membranes suggested that both membranes could be recovered and be reused. Based on the performances of both the SR90 and NF90 membranes, it was concluded that membrane based processes can be applied in the treatment of end of pipe textile effluent.

5. References

Agenson, K. O. and Urase, T. 2007. Change in membrane performance due to organic fouling in nanofiltration and reverse osmosis applications. Separation and Purification Technology, 55, 147-156.

Al-Amoudi, A. & Lovitt, R. W. 2007. Fouling strategies and the cleaning system of nf membranes and factors affecting cleaning efficiency. Journal of Membrane Science, 303, 4-28.

Blanpain-Avet, P., Migdal, J. F. & Bénézech, T. 2009. Chemical cleaning of a tubular ceramic microfiltration membrane fouled with a whey protein concentrate suspension—characterization of hydraulic and chemical cleanliness. Journal of Membrane Science, 337, 153-174.

BTTG 1999. Waste Minimization And Best Practice. In: 5, R. (Ed.).

Calvo, J. I., Peinador, R. I., Prádanos, P., Palacio, L., Bottino, A., Capannelli, G. & Hernández, A. 2011. Liquid–liquid displacement porometry to estimate the molecular weight cut-off of ultrafiltration membranes. Desalination, 268, 174–181.

Fersi, C. & Dhahbi, M. 2008. Treatment Of Textile Plant Effluent By Ultrafiltration And/Or Nanofiltration For Water Reuse. Desalination, 222, 263-271.

Ginsberg, G. & Barclay, S. 2012. Mediterranean Textile Mills Resource Efficiency & Cleaner Production Audit. In: 001 (Ed.).

Hermanutz, F., Oppermann, F. & Ozonationèan, W. 1994. Important technique to comply with new German laws for textile wastewater treatment. Journal of the Society of Dyers and Colourists, 30, 255-263.

Liikanen, R., Yli-Kuivila, J. & Laukkanen, R. 2002. Efficiency of various chemical cleanings for nanofiltration membrane fouled by conventionally-treated surface water. Journal of Membrane Science, 195, 265–276.

Nowak, K. M., Winnicki, T. & Wisniewski, J. 1989. Effect of flow conditions on ultrafiltration efficiency of dye solutions and textile effluents. Desalination, 71, 127/135.

Petrinića, I., Andersenb, N. P. R., Šostar-Turka, S. & Marechala, A. M. L. 2007. The removal of reactive dye printing compounds using nanofiltration. Dyes And Pigments, 74, 512–518.

Robinson, T., Mcmullan, G., Marchant, R. & Nigam, P. 2001. Remediation of dyes in textile effluent: a critical review on current treatment technologies with a proposed alternative. Bioresource Technology, 77, 247-255.

Sojka-Ledakowicz, J., Koprowski, T., Machnowski, W. & Knudsen, H. H. 1998. Membrane filtration of textile dyehouse wastewater for technological water reuse. Desalination 119, 1-10.

Wolmarans, J. 2011. The impact of trade policies on the south african clothing and textile industry: a focus on import quotas on chinese goods. Masters of Business Administration, University of Stellenbosch.