Desalination and Water Treatment
www.deswater.com

25 (2011) 133–142
January

doi: 10.5004/dwt.2011.1137

Application of the combined ultrafiltration and reverse osmosis for refinery wastewater reuse in Sinopec Yanshan Plant

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Received 16 January 2009: Accepted in revised form 29 June 2010

ABSTRACT

The refinery wastewater reuse system of Sinopec Yanshan Plant in Beijing (China) has been in operation for more than four years. The water reuse system combines biological treatment, media filtration with a combination of ultrafiltration (UF) and reverse osmosis (RO). After more than 30 times of chemical cleaning, the current RO system salt rejection is still above 97% at 80% system recovery. The normalized permeate flow of the three RO trains vary with the operation time but after chemical cleaning, they recover to above the design flow of 100 m^3/h. The data presented in this study indicate the fouling nature of the RO feed water on the 1st stage RO. However, according to the experience of Sinopec Yanshan Plant the output water quality meets the customer requirements. This is one of the first publications which show that the combination of UF and RO technology can be applied to reuse the refinery wastewater. The TOC rejection of the UF process is determined at 34%, which is highly dependent on the molecular weight of the organics. Low molecular weight organics could pass the UF unit and foul the RO membrane surface, causing serious organic fouling. Furthermore, the periodic pressure drop increase of the 1st stage RO system showed that there was serious bio-fouling. Therefore, addition of other pretreatment technology before UF, such as activated carbon cartridge filter and dosing non-oxidized biocides, are proposed alternatives that could help to increase the life-span of UF and RO elements.

Keywords: Refinery wastewater; Water reuse; Membranes; Ultrafiltration; Reverse osmosis

1. Introduction

The industrial growth in China over the last 15 years has dramatically increased the discharge volumes of wastewater from all industrial sectors. According to the China Environmental Statistic Report, in 2006 the total discharge volume of the industrial wastewater in China was 24 billion tons [1]. The COD (chemical oxygen demand) discharge volume in industrial wastewater was 5.4 million tons and the ammonia discharge volume was 425 thousand tons. The top five industries with the largest discharge volume are pulp and paper, chemical, power, textile and metallurgy. The chemical manufacturing industry mainly involves refineries and petrochemical industries.

The Chinese government is imposing increasingly stringent restrictions to encourage industries to treat and reuse their wastewaters. However, refinery and

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petrochemical wastewater is very complex and contains both high levels of TOCs (total organic compounds) and TDS (total dissolved solids) and many kinds of soluble organic compounds, most of which are non-biodegradable. Therefore the reuse of refinery and petrochemical wastewater is quite limited. Wastewater in the refinery and petrochemical industries is currently treated by an ASP (active sludge process) using an oil/water separator as pretreatment. There are limited studies (both pilot and full scale) on the treatment and reuse of refinery and petrochemical wastewater using advanced technology. Nevertheless, innovative technologies such as membrane technology and more advanced degradation techniques like photo-catalytic degradation and advanced oxidation process are required to comply with the tightening wastewater discharge and water reuse regulations.

In China, membrane technologies, including MF (microfiltration), UF (ultrafiltration), NF (nanofiltration) and RO (reverse osmosis), have been widely applied in water treatment and wastewater reuse for power and metallurgy industry [2,3]. In refinery and petrochemical industry, membrane technology is not widely applied yet, with only few recent and successful cases in China. This paper, presents the oldest case history of refinery wastewater reuse in China (Sinopec Yanshan Plant, Beijing) which has been operating for more than four years. An overview of the operation and the performance of the ultrafiltration and RO elements are discussed.

2. Backgroud

2.1. Literature

Recently, some bench scale studies have been performed with UF membrane systems to treat wastewater contaminated by organic compounds from refinery or petrochemical industries. UF and MF processes are being used as an alternative or as an additional step to the conventional clarification and filtration methods.

A laboratory scale study was conducted by Zhang et al. [4] to investigate the effectiveness of UF technology for treatment of refinery wastewater using powdered activated carbons and coagulant. Flux decline, cleanability by backwashing and removal rates of TOCs were studied. In this study, the removal rate of TOCs was reported at more than 99%. Fang [5] applied the combined MMF (multi-media filters) and UF technology to treat refinery wastewater. COD rejection was about 50–70% and COD of the UF permeate water was less than 32 mg/L. Both the turbidity and SDI (silt density index) of the UF permeate water met the pretreatment requirements and limits for RO feed water quality (Turbidity < 1 NTU, SDI < 5). Li [6] applied a combined UF and RO membrane system to reuse the petrochemical wastewater. The pretreatment before UF was contact oxidization and coagulation settlement, and the coagulant and flocculant were PAC (polyaluminium chloride) and PAM (polyacrylamide), respectively. SDI of the UF permeate was less than 3 and oil was less than 1 mg/L.

MBR (membrane bioreactor) technology has been tested recently on a laboratory scale for re-use potential of refinery wastewater. Hu [7] compared the removal efficiency of MBR and UF for turbidity, COD and SDI of a petrochemical waste stream. The pilot test showed that the permeate water quality of MBR and UF can both meet the feed water requirement of RO system (turbidity < 1 NTU, SDI < 5, COD best < 10 mg/L for surface water, COD tolerable < 40 mg/L for wastewater). The average level of COD, SDI and turbidity of the UF effluent were 22 mg/L, 2.5 and 0.18 NTU, respectively, while those of the MBR effluent were 20 mg/L, 2.2 and 0.14 NTU. Stability of the turbidity and SDI of MBR effluent was better than that of the UF effluent and the authors concluded that MBR can withstand much higher COD fluctuation compared to UF. In another bench scale study, an anoxic/aerobic concept MBR was tested under different conditions by Qi et al. [8]. The results showed the feasibility to treat the refinery wastewater using MBR technology. The sustainable membrane flux and applicable HRT (hydraulic retention time) of the process were obtained. The treated water quality consistently met the requirements for discharge while segregation of the streams with high TDS was required to reclaim the water for reuse. COD in the product was consistently less than 100 mg/L although feed COD fluctuated from 700 to 2000 mg/L. This level of COD removal efficiency (more that 93%) was also reported elsewhere [9] using cross-flow MBR to treat wastewater discharged by a petroleum refinery plant. In the recent study of Viero et al. [10] it was proved that the membrane had a key role in the MBR process, since it improved COD and TOCs removal efficiencies by 17 and 20%, respectively, in comparison with the results obtained by the biological treatment only.

Since polymer membranes are sensitive to both polar and chlorinated solvents, as well as high oil fractions, ceramic membranes are expected to have a wider application range to treat refinery wastewater. Zhong et al. [11] reported that ceramic membranes, particularly the zirconia membranes, show better separation performance such as higher flux, less fouling and higher oil rejection.

2.2 Case history of refinery and petrochemical wastewater reuse in China

In recent years, several refinery and petrochemical companies have begun to reuse their wastewater through a combined UF and RO membrane process. The wastewater reuse system of Sinopec Yanshan Plant, which is the earliest one (started operation in November 2004) and still the biggest, has been operating for more than four years. Table 1 [12–15] presents a list of the major petrochemical wastewater treatment projects (both discharge and reuse)

Table 1
Major refinery and petrochemical wastewater treatment projects applying membrane technology (MBR, UF and RO/NF)

Company name	Capacity (m^3/h)	Treatment process	Commission date	Reference
Kelamayi	170	Refinery wastewater + flotation + BAC + NF	Jun. 2004	[13]
Yanshan	800	Petrochemical wastewater + pretreatment + disinfection + UF + RO	Aug. 2004	[14]
Yanshan	410	Refinery wastewater + BAF + coagulation settlement + UF + RO	Oct. 2004	[12]
Daqing	500	Refinery and petrochemical wastewater + coagulation + cartridge filter + UF + oxidization + ACF + RO	Oct. 2005	[14]
Jingmen	200	Petrochemical wastewater + BAF + MMF + UF	2006	[15]
Haerbin	130	Refinery wastewater + coagulation + sand filtration + MF + ACF + RO	Mar. 2006	[14]
Qilu	150	Petrochemical wastewater + UF + RO	Jun. 2006	[14]
Dagang	320	Refinery wastewater + BAC + UF + RO	Nov. 2006	[14]
Jinan	230	Refinery wastewater + fiber filtration + BAC + electric flocculation + MMF + UF + RO	Dec. 2006	[14]
Jinzhou	600	Refinery wastewater + flotation + oxidization + BAC + UF + RO	Jul. 2007	[14]
Dushanzi	200	Petrochemical wastewater + BAF + MMF + UF + RO	Dec. 2007	[14]

which applied membrane technology in China. The earliest adaptor to use UF and RO technology was Sinopec Yanshan Plant in 2004. For reuse needs of wastewater, the combined UF and RO technology has been applied in many cases. BAF (biological aerated filter) and MMF are applied as the pretreatment of UF to protect both the UF and RO systems. BAC (biological active carbon) and ACF (active carbon filter) are also applied to remove organics in the treatment process.

3. Wastewater reuse system design of Sinopec Yanshan Plant

The wastewater reuse system in Sinopec Yanshan Plant is presented in Figs. 1 and 2. Fig. 1 shows the process flow of the refinery wastewater reuse system. The wastewater which originates from the refinery process is treated by a BAF (biological aerated filter) and coagulation settlement followed by chlorine treatment. Additionally, this water is treated by fibrous filtration (multimedia filter) and ACF (active carbon filter) to remove soluble organics prior to filtration with advanced filtration technologies for water reuse.

The integrated membrane solution for this water reuse opportunity is a combination of outside-in pressurized hollow fiber ultrafiltration (DOW Ultrafiltration™ SFP2660) and spiral wound fouling resistant brackish water membranes (Dow FILMTEC™ BW30-365 FR) (Fig. 2). The characteristics of both products are presented in Table 2. In total 600 UF elements are used with a total capacity of 560 m^3/h. One train capacity of UF system is 56 m^3/h. Fouling resistant RO membranes are used in the

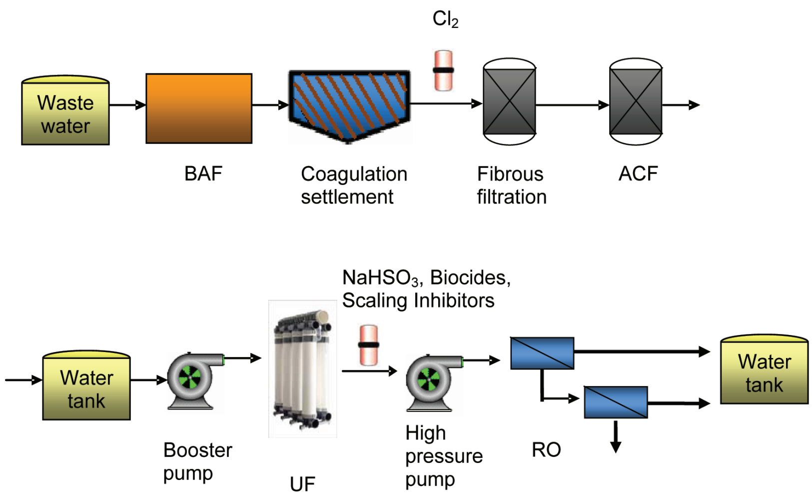

Fig. 1. Flowchart of the refinery wastewater reuse process.

Table 2
Characteristics of SFP2660 and BW30-365 FR

DOW Ultrafiltration™ SFP2660		FILMTEC™ RO BW30-365 FR	
Base polymer	PVDF	Membrane type	Polyamide (FTC)
Module surface area, m^2	33 (355 ft^2)	Nominal active surface area, m^2	34 (365 ft^2)
Filtrate flux @ 25°C, lmh	40–120	Permeate flow rate, m^3/h	860 (9500 gpd)
Diameter, cm	16.5 (6.5 inch)	Feed spacer thickness, mm	0.864 (34 mil)
Nominal pore diameter, μm	0.3	Stabilized salt rejection, %	99.5

Fig. 2. RO and UF system.

Table 3
UF and RO systems summary

Module model	UF	RO
Capacity, m^3/h	560	309
Number of skids	10 (8R/2S)	3
Number of modules per skid	60	138
Total number of modules	600	414
Capacity per skid, m^3/h	70	103
Recovery	>95%	80%
Design flux, l/m^2h	37.2	22

Table 4
Feed water quality of the refinery wastewater reuse system [12]

pH	7.0~8.4
COD_{Cr}, mg/L	20–50
NH_3-N, mg/L	0–10
TDS, mg/L	900–1400
Hardness, mg/L	300–500
Ca^{2+}, mg/L	200–360
Alkalinity, mg/L	50–150
Conductivity, us/cm	1400–1900
T, °C	25–38
Turbidity, NTU	1–6
Cl^-, mg/L	150–400
SO_4^{2-}, mg/L	150–360
Silica, mg/L	8–11
Oil, mg/L	0–1.2
Bacteria, unit	103–105
Total Fe, mg/L	0.5

three RO trains with 270 elements in the first stage and 144 elements in the second stage. The capacity of one train of the RO system is 103 m^3/h. A summary of the UF and RO systems is shown in Table 3. The UF permeate water is pumped directly into the RO system by high pressure pump after dosing scaling inhibitors (Flocon Plus, BWA™), reducing agents ($NaHSO_3$) and biocides (Flocide 380, BWA™).

Table 4 shows the feed water quality of the refinery wastewater reuse system, which is the secondary effluent of refinery wastewater [12]. The main characteristics are the high level of COD and the presence of some oil. The rejection of UF to COD is relatively low, thus the main problem of RO may be bio-fouling and organic fouling.

4. Results and discussion

4.1. UF system performance

The UF elements that are used can withstand a high concentration of oxidants (max. 5000 ppm NaClO, normal 2000 ppm NaClO cleaning) which is ideal to control bacteria growth [16]. The outside-in hollow fiber design enables demanding cleaning conditions like high turbulent air scouring. Table 5 shows the UF system operation process. The UF units were backwashed every 30 min for 60 s, and air scoured with backwash every 6 h for 60 s, then air scoured with forward flush every 6 h for 60 s. No chemically enhanced backwash (CEB) or chlorine was dosed during the backwash operation. Clean in place (CIP) (0.2% NaClO at pH 12, 0.5% oxalic acid) was performed every 2–5 months to remove the foulants from the fiber surface thoroughly. The UF system was operated at a water recovery of 92–95%.

Table 5
UF system operation process

Serial No.	Contents	Frequency	Duration	Chemical consumption
1	Filtration		30 min	
2	Upstream backwash	After every filtration	30 s	None
3	Downstream backwash	After upstream backwash	30 s	
4*	Air scour with backwash	6 h	60 s	768 Nm3/d
5*	Air scour with forward flush	6 h	60 s	
6*	Forward flush	After backwash	30 s	
7	CEB	None	None	None
8	CIP	2–5 months	8 h	Acid: 0.5% oxalic Alkaline: 0.2% NaClO at pH 12

* Repeat 4-5-6 several times per 12 h operation.

The UF units are used as a pretreatment to protect the RO elements from suspended solids, colloids and some large molecular weight organics. The SDI of the UF permeate was always below 3 (SDI unit). The turbidities of UF feed and permeate water are about 5 and 0.3 NTU respectively. Fig. 3 shows the trans-membrane pressure (TMP) and permeate flow of the UF units in 10 months. The average TMP was around 0.6 bar, which indicated that the UF fouling was well managed. This UF system has had 5 CIPs in the reported period, as indicated by the arrows in Fig. 3. In these 10 months of operation, the highest TMP was about 1.6 bar and far away from the design limit of 2.5 bar [16]. The permeate flow was around 60 m^3/h, which is slightly higher than the design flow 56 m^3/h.

4.2. RO system performance

Fig. 4 shows the operation performance of the three RO trains in the refinery wastewater treatment plant in recent four months, including feed pressure, permeate flow, and salt passage system. The system recoveries of the three trains were all stable at 80%. The feed pressure increased with operation time but could be recovered after chemical cleaning as is shown in Fig. 4. The permeate flows of the three trains were all kept at about 100 m^3/h. The salt passages of three trains were very low and less than 3%.

The operation data can only reflect the observed performance of the current RO systems, thus performance normalization was also done to show any performance

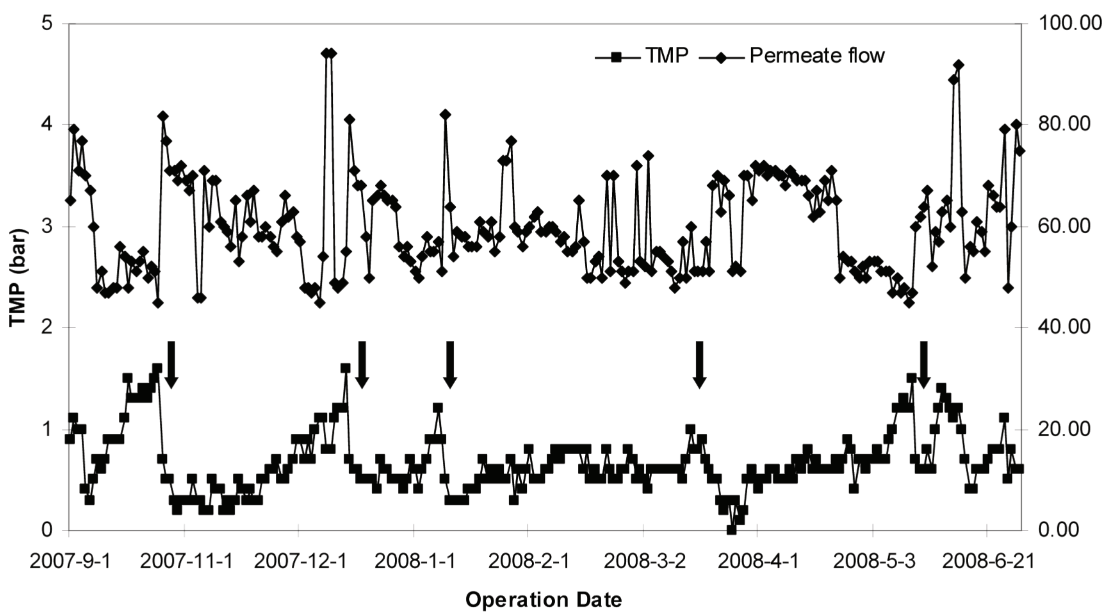

Fig. 3. Trans-membrane pressure and permeate flow of the UF units. The arrow (↓) indicates a chemical cleaning.

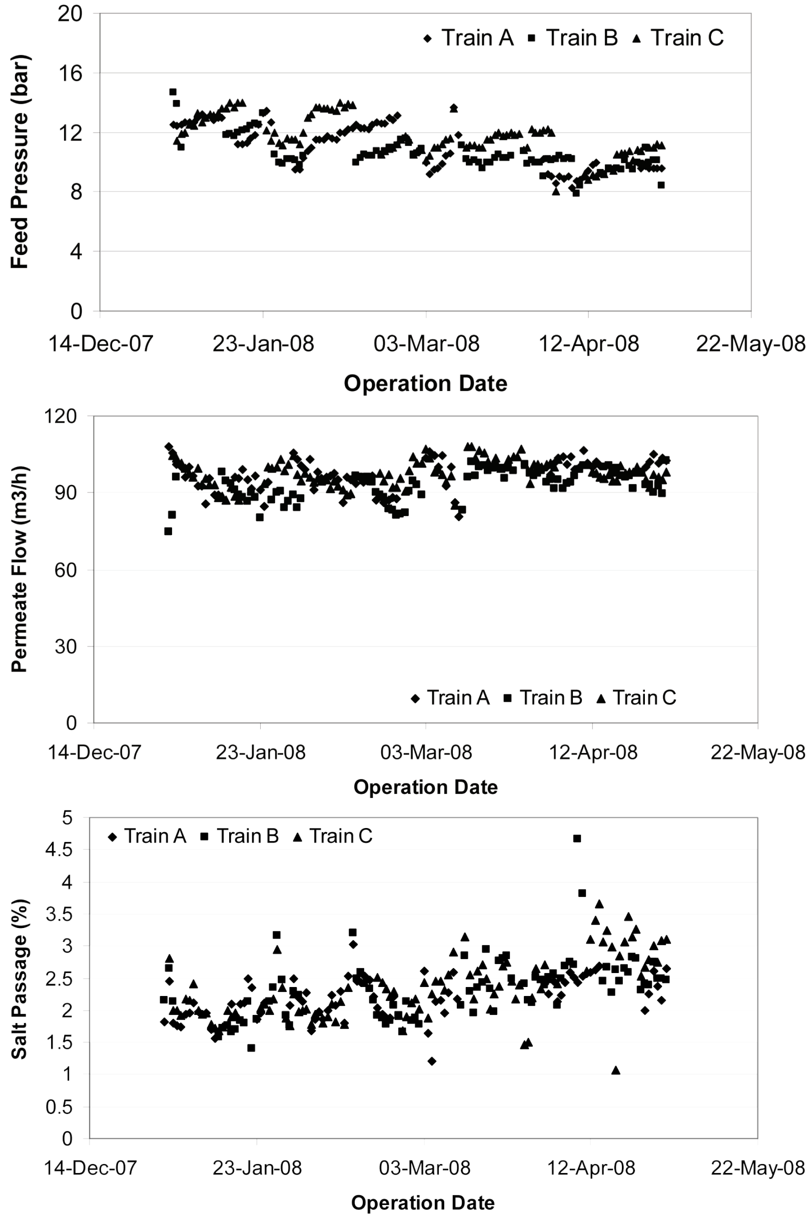

Fig. 4. Feed pressure, permeate flow, and salt passage of the three RO trains vs. operation time.

changes between day one and the actual date. Normalization is a comparison of the actual performance to a given reference performance while the influences of operating parameters are taken into account (feedwater composition, feed pressure, temperature and recovery). The reference performance may be the designed performance or the measured initial performance. In this case, normalization was done on the basis of the system performance on the start-up date. Fig. 5 shows the normalized salt passage of one of the RO trains. As shown in the figures, the normalized salt passages were close to 1.5% and all less than 2%, and also shown a little bit increase over time. The normalized permeate flows of the three RO trains varied with the operation time. After chemical cleaning, they could be recovered to above 100 m^3/h.

Fig. 6 shows the pressure drop of the RO train C changing with the operation time. The data show that the pressure drop of the 1st stage increased with the operation time from 2 bar to 5 bar, then dropped back to 2 bar after chemical cleaning. The arrows in Fig. 6 indicate the time of each chemical cleaning and show a cleaning frequency of once per month. This indicates a serious fouling concern in the 1st stage of the RO system. After the chemical cleaning though, the pressure drop of stage one decreased to the original level of 2 bar and the permeate flow also increased. The pressure drop of the 2nd stage was about

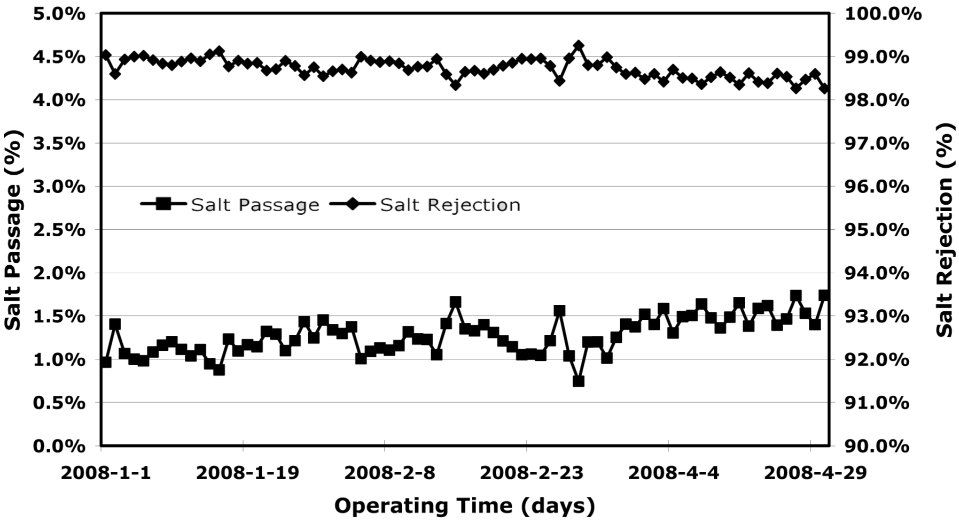

Fig. 5. Normalized salt passage of RO A vs. operation time.

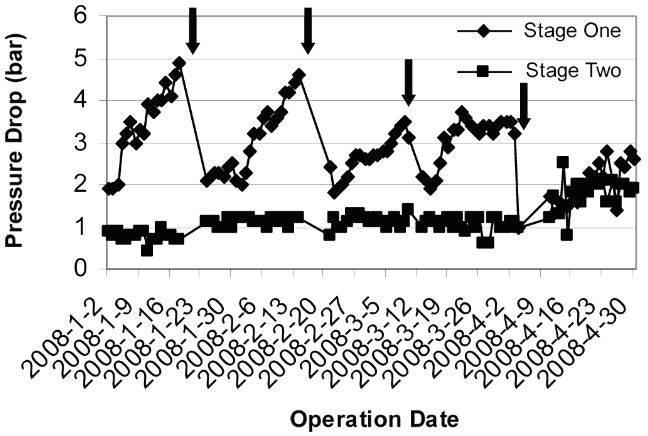

Fig. 6. Pressure drops of RO train C vs. operation time.

Table 6
UF Feed water, UF and RO permeate quality of the refinery wastewater reuse system

Items	Feed water	UF permeate	RO permeate
pH	7.42	7.39	5.99
HCO_3^-, mg/L	102.4	114	2.2
NO_3^-, mg/L	56	51	3.4
SO_4^{2-}, mg/L	359	353	1.2
Cl^-, mg/L	221.17	222.56	5.9
Ca^{2+}, mg/L	146.7	156.5	1.0
Mg^{2+}, mg/L	41.1	43.0	0.2
K^+, mg/L	5.94	6.12	0.3
Na^+, mg/L	168.8	176.6	5.4
TOC, mg/L	10.63	7.00	1.1
TDS, mg/L	1197	1232	24

0.5–1.5 bar which was lower and much more stable than that of the 1st stage.

4.3. Permeate water quality

Table 6 shows the UF feed water quality and the UF and RO permeate water quality in one sampling process in 2009. The data shows that the rejection to TOC of UF membrane is about 34% and RO has about 84% rejection to TOC. The salt rejection of RO is about 98% as shown previously. For different ions, the lowest anion rejection is nitrate which is about 93% and the lowest cation rejection is potassium which is about 95%. After more than four years operation, the salt rejection of RO is still good and the TOC rejection of UF is also acceptable.

4.4. RO fouling evaluation and cleaning protocol

Two elements (the first element in the 1st stage and the last element in the 2nd stage) have been sent back to the RO manufacturer to evaluate the fouling status of the RO system. The EPAS (element performance analysis service) test of these two elements were done with the standard test condition: 2000 ppm NaCl as test solution, at 225 psig (15.3 bar) operation pressure, 77°F (25°C), pH 8, and 15% recovery. The results showed that the flow rates of the two fouled elements were almost 30% lower than the standard flow rate and the pressure drops of a single element were also close to the limited condition (1 bar for single element), which indicated a serious blocking of the feed channel. The first element in the first stage showed more severe fouling than the last element in the second stage, which was consistent with the observation of the system pressure drops in Fig. 6. In order to have more understanding of the membrane fouling, the first element in the first stage was cleaned to help find the best chemical cleaning method, while the last element in the second stage was autopsied to analyze the characteristic of the foulant.

4.4.1. Cleaning tests

In each chemical cleaning, a caustic-acidic-caustic cycle was performed and the cleaning conditions were described as below:

- NaOH, pH 12, 35°C. Circulated for 60 min and flushed for 30 min.
- HCl, pH 2, 45°C. Circulated for 30 min and flushed for 30 min.
- NaOH, pH 13, 35°C. Circulated for 60 min and flushed for 30 min.

In the two high pH caustic cleaning, the solutions were both caramel colored, while the solution was clean after low pH acidic cleaning. The product flow rate increased 13% overall. The salt rejection was still between 99.35 to 99.43%. The cleaning results showed that high pH caustic cleaning can partially remove the foulant from membrane surface and recover the product flow to a certain level, but not to the initial value; low pH acidic cleaning almost had no effect on foulant removal. Salt rejection was not influenced by two high pH caustic cleaning and one low pH acidic. However, the pressure drop of the element only showed a little bit decrease after cleaning. It indicated that the chemical cleaning could only partially remove the foulant attached to the feed-spacer and membrane surface. More vigorous cleaning might be needed to peel it off.

4.4.2. Autopsy inspection and foulant ignition test

Fig. 7 shows the photos of the fouled membrane surface. It can be seen that the whole membrane leaves were covered by brown and caramel colored foulant, which also stuck to the feed spacer screen when the screen was removed from the membrane leaf. The foulant was easy to smear from the membrane surface but hard to rinse away by water. The acid and caustic drip tests showed no reaction or effect to the foulant, which explained the poor pressure drops decrease during chemical cleaning test.

The foulant was scrubbed from the membrane and sent for loss of ignition test. The foulant was first dried at 110°C. The test results showed that dry substance was 17.2% of the whole foulant, as shown in Table 7. A number less than 20% typically indicated that the foulant was mainly composed of biofouling. The dry substance was then dried at 550°C. This procedure destroys the organic material in the foulant sample. In the test 100% of the dry substance was proved to be organics. It can be concluded that the foulant on the membrane surface was bio-fouling and organics.

In order to address the issue of organic fouling and

Fig. 7. Photos of the fouled membrane surface.

Table 7
Loss of ignition test results of the foulant

3 samples average	
Dry substance, %	17.20
Foulant distribution, g/m²	22.39
Inorganics, %	0.00
Inorganics distribution, g/m²	0.00
Organics, %	100.00
Organics distribution, g/m²	22.39
Sample surface area, m²	0.0232

biofouling, some actions have been taken to prevent bacteria growth. For example, NaClO was dosed in the UF permeate pipes and keeping a 0.5 ppm free chloride in the UF permeate tank. At the same time, nonoxidizing biocides (such as DBNPA) were also dosed intermittently prior to the RO unit. The amount of DBNPA used depends on the severity of the biological fouling. 10–30 mg/L of the active ingredient from 30 min to 3 h every 5 d is minimum for this kind of feed water. Since reducing agents (sodium bisulfite) were used for chlorine removal, the concentration of DBNPA should also be increased by 1 ppm of active ingredient for every ppm of residual reducing agent in the RO feed water. To remove the dead biofilm, an alkaline cleaning is also recommended. Sometimes, the end user applied two kinds of biocides in turn to destruct the immunity of bacteria to one biocide.

The chemical cleaning procedure was adjusted according to the lab cleaning test results. A caustic-acid-caustic approach was adopted, since caustic cleanings are needed both for organic removal and for dead biofilm removal. As indicated both by the pressure drop data and membrane autopsy, the 1st RO stage was much more fouled than the 2nd stage, thus NaOH alkaline cleaning (pH = 12) was first performed in the 1st stage (recycling 2–4 h, soaking for 10 h). Then, acid cleaning (pH = 2) was performed in two stages together to avoid any possible scaling of insoluble salts (recycling 2~4 h, soaking for 10 h). Finally, alkaline cleaning (pH = 12) was performed again in two stages together to further clear the residue oragnics/biofilm (recycling 2–4 h, soaking for 10 h). The CIP frequency was kept within 1 month, which was acceptable by the end users.

5. Conclusions

The refinery wastewater reuse system of Sinopec Yanshan Plant in Beijing has been in operation for more than four years. In the beginning, the RO cleaning frequency was about one chemical cleaning every three months. Now the cleaning frequency is about once per month. After more than 30 times of chemical cleaning, the current system rejection is still above 97% at a system recovery of 80%. The normalized permeate flows of the three RO trains vary with the operation time but can be recovered fully after chemical cleaning, to above 100 m^3/h. The pressure drop of the 1st stage increases with the operation time from 2 bar to 5 bar, then drops back to 2 bar after each chemical cleaning. The pressure drop of the 2nd stage is lower and much more stable than that of the 1st stage, which is about 0.5–1.5 bar. This observation indicates a serious fouling concern in the 1st stage RO.

UF could protect RO elements and remove suspended solids, bacteria and colloids effectively. However, TOC rejection of UF was 34% in the case study presented here. Other research showed that the COD rejection is highly dependent on the molecular weight of the organics. The rejection data that were obtained in the Sinopec Yanshan Plant are in line with the 0~60% COD rejection observed by Wu et al. [17]. For refinery and petrochemical water with high organic contaminants, coagulation/flocculation and multimedia filter can remove most of the large molecular weight organics, while active carbon filter as pretreatment before the UF can adsorb small molecular weight organics. In the case study presented here, the authors believe that some low molecular weight organics could pass the UF unit and foul the RO membrane surface, causing serious organic fouling. For this reason, some research institutes suggested to use ACF + UF as organic removal process [17] to increase the lifetime of both the UF and RO elements.

Furthermore, the periodical pressure drop increase of the 1st stage RO system strongly suggests that biofouling occurs. Element autopsy and foulant analysis further confirmed the cause. This can be induced by the secondary contamination of the pipes or water storage tanks. This problem can be solved by dosing NaClO in the UF permeate pipes and keeping a 0.5 ppm free chloride in the UF permeate tank. A precaution should be taken to add the appropriate amount of reducing agent ($NaHSO_3$) before the RO operation to protect RO membranes from damage by oxidation. Alternatively, non-oxidized biocides could be dosed prior to the RO operation to reduce the biofouling on the RO elements and increase their life-span.

According to the experience of Sinopec Yanshan Plant, the combined and integrated use of UF and RO technology suits their need to reuse wastewater. The output water quality meets the customer requirements. However, some operational concerns like organic fouling and bio-fouling of the RO elements demand the use of more stringent or alternative pretreatment of the membrane operation.

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