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Advanced treatment of biologically treated heavy oil wastewater for reuse as boiler feed-water by combining ultrafiltration and nanofiltration

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ABSTRACT

This work developed a new treatment system, which could effectively purify biologically treated heavy oil produced wastewater to the acceptable levels in the reused boiler at the Liaohe Oilfield, China. The ultrafiltration was used as pretreatment for the nanofiltration process. The filtration experiments of the selected wastewater were performed by modifying transmembrane pressure of the membranes. Results showed that the water quality of final permeate was improved after filtration with the ultrafiltration–nanofiltration membranes to successfully meet the admissible limit set by China's Environmental Protection Agency. After coupling ultrafiltration with nanofiltration process, a stable permeate flux was observed, which was kept above 25 L/m^2 h during 40 min of continuous treatment. It was also found that a flux recovery of 0.96 was obtained for the fouled ultrafiltration membrane after chemical cleaning with the mixture of NaOH and sodium dodecylsulphate (both at 0.2 wt.%).

Keywords: Produced water; Thermal exploitation; Hardness; Ultrafiltration; Nanofiltration

1. Introduction

In China, heavy oil reservoirs are widely distributed, with a geological reserve of about 1.64 × 10^9 tons. Thermal exploitation using steam soak is a mature process for enhancing recovery of heavy oil, and it has been widely applied in many oil fields at home and abroad [1–3]. This process consumes vast volumes of freshwater and generates much produced water (PW) in high temperature (50–70˚C). PW contains elevated levels of salt as well as other inorganic and organic components. The characteristics of PW can vary greatly depending on factors such as geographic location, contact time with the oil in the formation, method of extraction, treatment chemicals, and physiochemical compositions [4].

PW discharged to water body would make the environment greatly polluted. It is of significance that PW should be reused for enhancing oil recovery or discharged into the environment after proper treatment [5].

It is valuable to treat and reuse heavy oil PW in the boiler for reinjection into the reservoirs [6], which can solve the problems of environmental pollution caused by discharge of PW and water shortage for thermal exploitation in oil fields. Beneficial use of PW has become an attractive solution to PW management by providing additional water supplies and reducing the cost for disposal of PW. However, PW contains high concentrations of oil, polymers, and salts, etc. which makes PW not meet the water quality requirements for the boiler [6].

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High-pressure membranes such as reverse osmosis have been used to desalinate seawater and brine water for a long time and can offer a possible solution for a beneficial treatment of PW. It has been shown that water with good quality could be generated by removing a large fraction of organic and inorganic constituents from PW using reverse osmosis [7]. However, the application of reverse osmosis was limited by membrane fouling, which resulted in quick deterioration of the membrane performance and increased operation costs [8,9].

Nanofiltration has been widely used for removing organic and inorganic substances from aqueous phase with lower operating pressures than reverse osmosis; however, membrane fouling is also a major constraint to the further application of nanofiltration [4,10,11].

The inorganic carbon content of PW is made up of water-soluble components like carbonate and bicarbonate salts that are not expected to be retained by ultrafiltration membrane. However, some researchers have demonstrated that modifying nanofiltration membrane by layer-by-layer assembly of polyelectrolyte thin films increases salt rejection [12,13]. Thus, ultrafiltration membrane can be used as an organic prefilter to a nanofiltration unit to reduce membrane fouling of nanofiltration.

The objective of this study was to explore the feasibility of reusing PW in a boiler after being purified by combination of ultrafiltration and nanofiltration. The membranes were evaluated in terms of solute rejection, membrane permeability, and antifouling performance.

2. Materials and methods

2.1. Characterization of the wastewater

The PW used in this study was obtained from the secondary settling tank of a wastewater treatment plant based on anaerobic–aerobic activated sludge process located in Liaohe Oilfield, Liaoning Province, China. The effluent was taken from the final discharge point of the existing wastewater treatment plant. The effluent quality of the process is listed in Table 1.

Table 1
Characteristics of the biologically treated PW effluent used in this study

Parameter	Range	Average
pH	6.2–6.8	6.5
COD (mg/L)	92–135	106
Oil (mg/L)	3.6–7.5	5.8
TDS (mg/L)	8,230–9,340	8,650
SS (mg/L)	15.3–18.6	16.5
Turbidity (NTU)	115–172	138
Hardness (mg/L as $CaCO_3$)	1,232–1,315	1,266
Si^{2+} (mg/L)	14.5–16.1	15.3
Ca^{2+} (mg/L)	486–522	507
Mg^{2+} (mg/L)	45–51	48

Notes: COD: chemical oxygen demand; TDS: total dissolved solids; and SS: suspended solids.

2.2. Membranes

Composite ultrafiltration membrane (model KL-UF-4040) with a nominal molecular weight cutoff of 30 kDa and a maximum transmembrane pressure (TMP) of 0.3 MPa was purchased from Hangzhou Kailv Membrane Technology Co., Ltd, China. Nanofiltration membrane (model NF90-4040), purchased from Film-Tec Dow Chemical Company (USA), is made of polyamide thin-film composite with 7.2 m^2 nominal active surface area and 7.6 m^3/d permeate flow rate at 25°C and 0.48 MPa pressure.

Membranes were first flushed with 20 L/m^2 of deionized water and then equilibrated with 20 L/m^2 of 0.1 M NaOH and immersed in 20 L/m^2 of a solution of 3-bromopropanesulfonic acid sodium salt in 0.1 M NaOH for 48 h [14]. Afterward, the membranes were flushed with 100 L/m^2 of 0.1 M NaOH followed by 100 L/m^2 deionized water.

2.3. Experimental setup

To remove some suspended solids (SS) and organic substances, the biologically treated PW was pretreated using hydrophilic polyethylene hollow fiber microfiltration membrane with pore size of 0.1 μm and surface area of 0.05 m^2. Afterward, ultrafiltration and nanofiltration treatments were carried out sequentially with a P-28 membrane unit from CM-Celfa Membrantechnik AG (Seewen, Switzerland), which were operated in cross-flow mode (feed stream flowing tangentially to the membrane surface). The main elements of this unit have been described by others [15].

A standard protocol for ultrafiltration and nanofiltration experiments was composed of two steps. At first, deionized water was pumped into the system to measure the water permeability of the membrane. Secondly, the system was emptied and filled with feed to perform the continuous experiments under varied pressures. The permeates of both ultrafiltration and nanofiltration units were collected and analyzed. The backwashing process was carried out for the ultrafiltration membrane every 40 min at a pressure of 0.06 MPa for 30 s.

The fouled ultrafiltration and nanofiltration membranes were chemically cleaned according to the protocol proposed by Muñoz-Aguado et al. [16], using HCl, NaOH, NaClO, sodium dodecylsulphate (SDS), or mixture of NaOH and SDS (1:1, m/m) as a cleaning agent. The concentrations of individual cleaning agents ranged from 0.05 to 1 wt.%. Fresh chemical cleaning solution was prepared daily by dissolving each cleaning reagent in deionized water. The SDS solution was prepared 8 h before performing chemical cleaning experiments to ensure complete dissolution of the foam residue. For starting cleaning process, the fouled membrane was first washed with deionized water for 5 min to remove unbounded substances from membrane surface. This was followed by washing the membrane with a cleaning agent at ambient temperature without pressure within 20 min. Membranes were washed again with deionized water to remove any chemical agent within 10 min, and then, flux of water was determined. Each test was conducted with a new membrane filtering PW under controlled environment. Flux recovery (R) is calculated according to Eq. (1) [17]:

$$R = \frac{J_c}{J_0} \tag{1}$$

where J_c is the flux after the application of a certain cleaning solution and J_0 the flux of the virgin, unfouled membrane.

Membrane fouling potential was assessed by the silt density index (SDI) calculated from the rate of plugging of a 0.45-μm membrane filter at 30 psi using the standard test method D4189-95 according to Eq. (2) [18]:

$$\mathrm{SDI} = \left(\frac{\%P_{30}}{T}\right) = \left[1 - \left(\frac{t_i}{t_f}\right)\right] \times \frac{100}{T} \tag{2}$$

where $\%P_{30}$ is the percent at 30 psi feed pressure, T the total elapsed flow time (min), t_i the time to collect initial 100 mL of sample (s), and t_f the time to collect final 100 mL of sample (s) after the time T.

2.4. Analytical methods

All pH values were determined by means of a model PHS-3E pH meter (Shanghai Precision & Scientific Instrument Co., Ltd, Shanghai, China). Conductivity was measured using an electric conductivity meter (DDS-11A, Shanghai Yilong Instrument Co., Ltd, China). The following water quality parameters were measured according to standard methods [19]. In brief, oil was measured using an infrared spectrophotometry oil-measuring instrument (H3-OCMA-350, Japan) after dichloromethane liquid–liquid extraction. The concentrations of chemical oxygen demand (COD) were determined with $K_2Cr_2O_7$ and H_2SO_4 in a 1:1 ratio by the open reflux method with $AgSO_4$ as a catalyst and $HgSO_4$ to remove Cl^- interference. Excess dichromate was titrated with Fe^{2+} using phenanthroline as an indicator. Si^{2+}, Ca^{2+}, and Mg^{2+} ions were detected using inductively coupled plasma-atomic emission spectrometry (Thermo Electron Co., Franklin, USA). Total dissolved solids (TDS) were determined gravimetrically after evaporation. SS was measured by filtration through glass fiber filters. Turbidity was determined using a portable turbidimeter (model 2100P, Hach Company, Loveland, Colorado, USA), and results were reported in nephelometric turbidity units (NTU). Hardness was measured by EDTA titrimetric method, and results were expressed as mg/L as $CaCO_3$.

All experiments in this study were performed in triplicate, and results represented the average of three parallel experiments.

3. Results and discussion

3.1. Ultrafiltration performance

Increased temperature may reduce solution viscosity, resulting in increment in the permeate flux of membranes. The temperature of oil field PW is generally in the range of 40–80°C [20]. However, the temperature of PW can decrease continuously to ambient temperature with treatment time. Thus, filtration treatments were performed at room temperature in this study.

3.1.1. Influence of TMP

TMP is a major factor influencing permeate flux of membranes. Ultrafiltration experiments of the wastewater were performed in batch mode, by modifying TMP of the membrane.

The permeate flux, obtained as a function of time for the ultrafiltration membrane at different TMP with a constant $v = 1.5$ m/s, is plotted in Fig. 1. As can be seen, the initial permeate flux increased with increasing TMP, and the permeate flux gradually decreased with time for all the three applied pressures. Regular backwashing using water could alleviate flux decline, but it could not fundamentally eliminate concentration polarization of solutes, deposit of solute or particle into membrane pores, and hole wall adsorption of membranes [21]. Thus, mass transfer resistance may

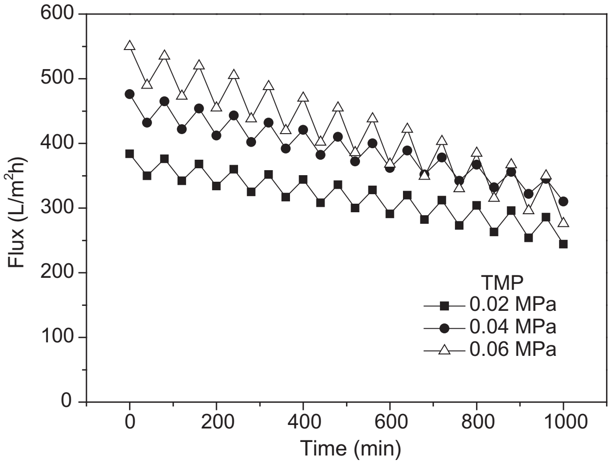

Fig. 1. Evolution of permeate flux with processing time for the wastewater filtration experiments performed with the ultrafiltration membrane. The first data at time = 0 represented the permeate flux of distilled water.

increase with processing time, resulting in continuous decrease of flux. Additionally, Fig. 1 shows that higher pressures were associated with a greater rate of flux decline. At the same operation time, the permeate flux was reduced by 32.6, 41.3, and 45.2% for 0.02, 0.04, and 0.06 MPa of TMP, respectively. The use of 0.06 MPa pressure generated the highest initial flux, but also gave the greatest flux decline. Higher TMP caused greater flux decline, higher energy consumption, and shorter backwashing period. Thereupon, 0.04 MPa was selected as the optimum pressure for the ultrafiltration process. At this TMP, the oil concentration of filtrate was below 0.05 mg/L, and SDI was lower than 5 (Table 2), which could meet the influent quality standard of nanofiltration membrane.

The increase in flux decline was connected with the polarization of particle deposition on the membrane surface and with pore blockage. In general, the permeate flux increases initially with the applied TMP, and then decreases with continued increase in the TMP [22]. Increasing the TMP means increasing the concentration polarization and thus increasing the number of collisions between particles.

Table 2
The oil content of permeate and SDI at various TMP during ultrafiltration experiments

TMP (MPa)	Oil content (mg/L)	SDI
0.02	0.040	3.81
0.04	0.042	4.73
0.06	0.045	5.18

3.1.2. Chemical cleaning of ultrafiltration membrane

Membrane fouling is one of the main factors accounting for flux decline. The above results (Fig. 1) showed that the efficiency of hydraulic cleaning was not satisfactory; thus, chemical cleaning was required for recovery of permeate flux. In this study, the efficiency of chemical cleaning was examined with HCl as an acidic solution, NaOH as an alkaline solution, NaClO as an oxidant, and SDS as an anionic surfactant. These chemical agents are common ingredients in commercial chemical cleaning solutions for organic and inorganic fouled membranes. When the permeate flux decreased by 40%, chemical cleaning was conducted and permeability was measured using pure water.

The cleaning efficiency with different cleaning solutions is compared in Fig. 2. It is clear that under the same condition, the cleaning efficiency was dependent strongly on the nature and concentrations of the cleaning agents. Fig. 2 shows that the cleaning efficiency was generally elevated with increasing the cleaning agent concentration. However, an optimum concentration was observed for some of the cleaning agents, and increasing the concentration of cleaning agents above the optimum levels did not improve the cleaning efficiency but rather reduced the cleaning efficiency. This is in agreement with other reports [23,24]. The cleaning efficiency with HCl solution was quite poor for all applied concentrations, indicating a minor impact of inorganic foulants on the membrane fouling. As an oxidant, NaClO can oxidize and remove organic foulants from the membrane. Within the investigated concentration range, the maximum

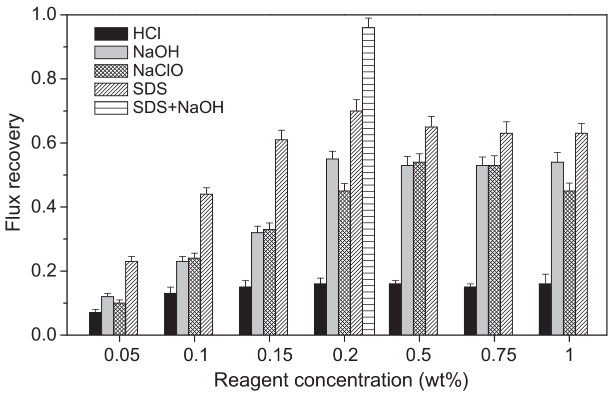

Fig. 2. Flux recovery of the ultrafiltration membrane in cleanings applying cleaning chemicals at various concentrations. In the case of NaOH + SDS, the concentration of NaOH and SDS was both 0.2 wt.%. The error bars represented the standard error of the mean (SE) ($n = 3$).

flux recovery reached 0.54 with 0.5% NaClO. Higher concentration of NaClO did not improve the cleaning efficiency; it even caused slight reduction of flux recovery. For NaOH and NaClO, the highest flux recovery was less than 0.6 (Fig. 2), suggesting that oil was not the sole cause of the membrane fouling. Nowadays, surfactants and polymers have been widely used for enhancing oil recovery in China's oil fields including Liaohe Oilfield [25]. Thus, these chemicals also played an important role in the membrane fouling in this study. The highest flux recovery of SDS was only about 0.7 (at 0.2 wt.% concentration), whereas the mixture of NaOH and SDS (both at 0.2 wt.% concentration) yielded a flux recovery of 0.96 (Fig. 2).

The selection of preferable cleaning agent depends on feed characteristics [26]. Generally speaking, acid solution is convenient for the removal of salt precipitates. Alkaline solution increases the dissolution of organic substances. SDS can efficiently break down the organic gel network by disarranging the complex between the organic foulants and the divalent action Ca^{2+} and Mg^{2+} [23]. However, SDS alone was not effective at solubilizing organic foulants due to foulant re-adhesion and consolidation [27]. Beyer et al. [27] reported that cleaning of organically fouled membranes using a 10 mM SDS solution at pH 11 resulted in excellent flux recovery (as high as about 1.3). Some chemical cleaning processes increase the flux recovery higher than 1. This may be due to (i) damage of the membrane or ridding the pores of the material that is left from the membrane preparation process and (ii) altering the surface more hydrophilic by the adsorption of the chemicals [23].

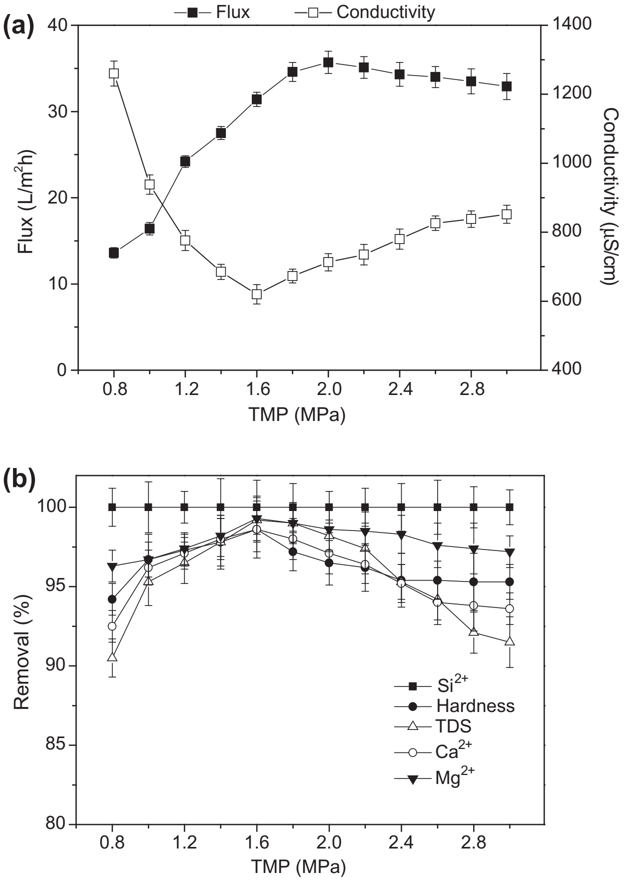

Fig. 3. Effect of TMP on permeate flux and conductivity (a) and ion removal (b) during nanofiltration experiments. The error bars represented the standard error of the mean (SE) (n = 3). The time and volume of each test was 6 h and 45 L, respectively.

3.2. Nanofiltration performance

The ultrafiltration permeate from the PW filtration was further filtered using nanofiltration. The effect of TMP on the permeate flux and conductivity can be observed in Fig. 3(a). As can be seen, the flux increased markedly with increasing TMP from 0.8 to 2.0 MPa, followed by a slight decrease from 2.0 to 3.0 MPa. This can be explained by that an increase in TMP leads to an increase of the permeate flux, with the permeate thus becoming more dilute [28]. The flux reduction at higher TMP can be caused by the concentration polarization, by the formation of a cake layer on the membrane surface, or by the internal occlusion of the pores.

Fig. 3(a) also shows that TMP affected especially the permeate conductivity. With increasing the TMP, the conductivity at first decreased and then suffered a gradual increment. The inflection point of the TMP–conductivity curve can be ascribed to the decrease of retention ability of the membrane toward salts at higher TMP caused by the gradual saturation of the nanofiltration membrane.

Calcium and magnesium are known as the dominant species for water hardening [29]. Water hardness is responsible for the formation of precipitates in boilers. Formation of deposits may cause a decrease of heat transfer in boilers, a decrease of fluid rate, bursting of water pipelines and boilers. Thus, the water of steam injection boilers in oil fields should be free of scale-forming ions as much as possible. Fig. 3(b) shows the effect of TMP on ion removal during nanofiltration experiments. As shown, the removal efficiency of Si^{2+} was almost 100%, which was not detected in the permeate. Silicate scale is harmful to the run of steam boilers in the production in oil fields. The nanofiltration system exhibited high removal efficiency toward Ca^{2+}, Mg^{2+}, hardness, and TDS

(Fig. 3(b)). Their removals increased gradually with increasing TMP up to 1.6 MPa, and then slightly decreased. This variation trend was just like that of the permeate conductivity as plotted in Fig. 3(a). Thereupon, 1.6 MPa was taken as the suitable pressure for nanofiltration treatment in this study.

3.3. Performance stability of the integrated ultrafiltration–nanofiltration system

Continuous filtration treatment was performed on the PW during a 40-min period with 0.04 and 1.6 MPa of TMP for the ultrafiltration and nanofiltration units, respectively. Fig. 4 shows the permeate flux and the removal efficiency of hardness, SS, and TDS within 40 min during the integrated ultrafiltration–nanofiltration. It can be found that the integrated system showed high performance, with removal percentages of 98.0–98.6%, 98.4–99.2%, 97.6–98.5% for hardness, SS, and TDS, respectively. Meanwhile, high permeate flux was obtained during the treatment. As listed in Table 3, the concentrations of various indexes in the PW were reduced after filtration with the ultrafiltration–nanofiltration membranes to successfully meet the admissible limit set by China's EPA. Several studies have demonstrated that ultrafiltration is an appropriate technique as a pretreatment of nanofiltration process to reuse or purification of wastewater [30–32].

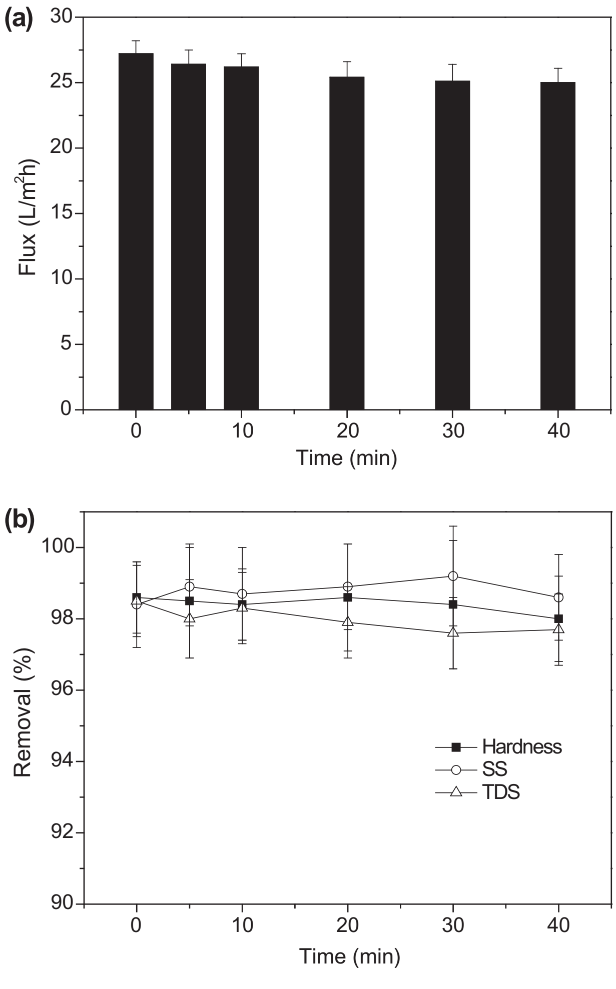

Fig. 4. Permeate flux (a) and removal efficiency of various indexes (b) as a function of processing time during the integrated ultrafiltration–nanofiltration. The TMP of ultrafiltration and nanofiltration was 0.04 and 1.6 MPa, respectively.

Table 3
Overall water quality parameters measured for the effluent of the ultrafiltration–nanofiltration hybrid system and the admissible limit set by China's EPA for boiler

Parameter	Value	Limit
Oil (mg/L)	ND	<2.0
Hardness (mg/L)	8.3–12.6	<2,000
SS (mg/L)	0.21–0.53	<2.0
TDS (mg/L)	113–152	<230
COD (mg/L)	11–14	<50
pH	7.8–8.1	7.0–11
Turbidity (NTU)	1.1–1.4	<5.0

Note: ND: not detected.

4. Conclusions

The results of this study showed that using ultrafiltration membrane as a pretreatment in an integrated nanofiltration system could produce high-quality water with potential for reuse in boils in oil fields. The fouled ultrafiltration membrane could be well cleaned by rinsing with the mixture of NaOH and SDS (both at 0.2 wt.%). The mechanism of ultrafiltration and nanofiltration membranes in purifying PW needs to be investigated in further depth.

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