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Purification of UF-treated anaerobically digested manure wastewater by two-pass reverse osmosis

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ABSTRACT

Membrane technology provides a potential promise towards manure treatment as well as nutrients concentration and recovery in the livestock industry. A two-pass reverse osmosis (RO) system was described in this study for the purification of UF-treated anaerobically digested manure wastewater (ADMW) with respect to permeate flux, ionic rejection, permeate quality, and membrane cleaning. The permeate flux fluctuated at 26.6–33.4 and 73.7–91.2 $\mathrm{L\,m^{-2}\,h^{-1}}$ for the first- and second-pass RO processing, respectively, at operating pressure of 1,500 kPa in the temperature range of 24–34˚C. The overall rejections of both Na^+ and K^+ by the two-pass RO were greater than 99%, whereas in case of ammonia, it was approximately 88%. The significant presence of molecular NH_3 at relatively high pH in ADMW solution probably induced a lower rejection of total ammonium, which also largely led to the low rejection of alkalinity and further pH increase in the RO permeate. The two-pass RO treatment afforded a complete rejection of Cl^-. The overall rejections of dissolved solids (DS) were estimated to be 94% with the first-pass RO and 98.8% after the two-pass RO resulting in product water containing only 45 mg/L of DS. Chemical cleaning of used membrane was successfully performed by successive flush of specific acid and base cleaning agents.

Keywords: Anaerobically-digested manure wastewater; Reverse osmosis; Ammonia; Purification

1. Introduction

A resurgent interest in anaerobic treatment of organic wastes has been stimulated by the increasing concerns over the environment pollution and the rising costs for energy [1–5]. Anaerobic digestion of organic wastes and the corresponding nutrient recovery provides bioenergy and bio-based fertilizer, and simultaneously diminish the waste disposal, odor emissions and some other negative impacts on environment [6–8]. The resulted wastewater from anaerobic digestion, nevertheless, will contribute to lake eutrophication, dissolved oxygen depletion, fish toxicity in surface water, and even groundwater contamination, if it is directly discharged into the environment. To avoid the severe impacts on the environment, anaerobically digested wastewater needs

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further treatment, such as the advanced solid–liquid separation, nutrient control, and recovery [9].

Following anaerobic treatment, a subsequently aerobic biological process might enhance the settleability of the resulted biowaste and provide the feasibility in the removal of the high-strength ammonium remaining in the rejected wastewater [10,11]. However, a typical nitrification/denitrification biological process requires a large amount of external electron donors, such as methanol, acetate, and ethanol which makes biological denitrification quite expensive [12]. There appear some novel biological processes, such as partial-nitrification/denitrification, which is specifically developed for the elimination of high concentration of ammonium in this kind of wastewater treatment. However, most of these new techniques are still in the laboratory stage without stable practice in full-scale facilities [13,14]. Furthermore, the deduced large footprint and complexity make the incorporation of an alternative aerobic process less attractive.

Integrated membrane technology, such as ultrafiltration/microfiltration followed with reverse osmosis (RO) is widely applied for the wastewater treatment and process water reuse, particularly in the field of food industry [15,16], juice production [17], textile industries [18], and municipal wastewater reclamation [19,20]. We developed an integrated manure utilization system (IMUS) for the treatment of anaerobically digested manure wastewater (ADMW), which consisted of the integrated uses of ultrafiltration (UF), RO, steam-stripping (ST) and ion-exchange (IE). Tubular UF was firstly employed to realize the ultimate solid–liquid separation of the biowaste produced by anaerobic digestion. Subsequently, the filtered solution was treated by RO to remove large amounts of the dissolved solids (DS). Finally, the ammonia accumulated in the RO concentrate was recovered by the ST [21] and that retained in the RO permeate could be further eliminated by the IE using natural zeolite [22,23]. Although there have been some reports available on the RO treatment of wastewaters having similarities with ADMW, purification of ADMW using RO processing, to our best knowledge, is poorly documented yet [9]. In the context of permeate flux, ion rejection rate, water quality, and membrane cleaning, a two-pass RO system was to be described in this study for the purification of UF-treated ADMW.

2. Materials and experimental methods

2.1. UF-treated ADMW

Anaerobically digested cattle manure wastewater was obtained from the IMUS large-scale plant, which was located in a farm nearby the town of Vegreville, Alberta, Canada. The cattle manure originally contained 22 wt.% dry matter and was diluted to 10–12 wt.% total solids before the digestion. The anaerobic digestion process was operated at thermophilic temperature (50–57˚C) with 15 days of hydraulic retention time. ADMW produced in the plant firstly experienced a preliminary solid–liquid separation by onsite press screw fan separator (Fan Separator GmbH, Germany), by which the total solids content was reduced from 8% in the anaerobic effluent to 2–4%, and then was pumped into a nearby lagoon for the restoration. The wastewater for use in membrane treatment was pumped from the lagoon and transported by truck to IMUS pilot-scale plant. After the UF treatment, the characteristics of the resulted wastewater are exemplified in Table 1.

2.2. RO system

The experimental RO system, as schematically shown in Fig. 1, was provided by Applied Membrane Corporation, USA. The system was equipped with thermometers, flow meters, pressure gauges, and control valves to determine and control the relative parameters of effluent in and out. The UF-treated ADMW was driven by a booster pump and a high pressure pump, firstly through a pre-filter with 5 μm of pore size, and then into two connected spiral-wound membranes (thin film composite membranes, Model L-125A-116, Applied Membrane Corp. USA) each with length of 102 cm, diameter of 6.1 cm, and membrane area of 1.5 m^2. The stabilized salt rejection was 99% determined with a 2,000 mg/L NaCl feed solution at an applied pressure of 225 ψ (1,550 kPa) and 25˚C of feed water temperature. The permitted operating pH recommended for the membranes ranged from 2 to 11. The system was operated at the recommended operating pressure (1,500 kPa) and the measured tap-water flux was 78.7 $L\,m^{-2}\,h^{-1}$ at 15˚C. In order to yield product water with properties suitable for reuse in the anaerobic digestion process, a two-pass RO system was used to treat the targeted wastewater. The first-pass RO permeate containing NH_4^+-N up to 250 mg/L would pass through a second-pass treatment. Both first- and second-pass RO are used the same system in feed-and-bleed mode. The operating pressure of two-pass RO was kept constant at 1,500 kPa. Near the end of the experiment of first-pass RO, the permeate was directed into a new tank to be used as the feed for the second-pass RO. The recoveries of the first-pass and second-pass were regulated constantly at 70 ± 2 and 80 ± 2%, respectively, yielding a 56% total recovery by the

Table 1
The characteristics of UF-treated ADMW (The variation of individual component in the feed wastewater covered ± 10%)

Variable	Concentration	Variable	Concentration
Dissolved solids	3.75 g/L	K^+	1,004 mg/L
Suspended solids	Undetected	Ca^{2+}	21 mg/L
Turbidity	5 ntu	Mg^{2+}	17 mg/L
Alkalinity ($CaCO_3$)	2,867 mg/L	Cl^-	705 mg/L
pH	8.5	SO_4^{2-}	10 mg/L
Na^+	366 mg/L	PO_4^{3-}	27 mg/L
NH_4^+-N	836 mg/L	NO_3^- NO_2^-	Undetected

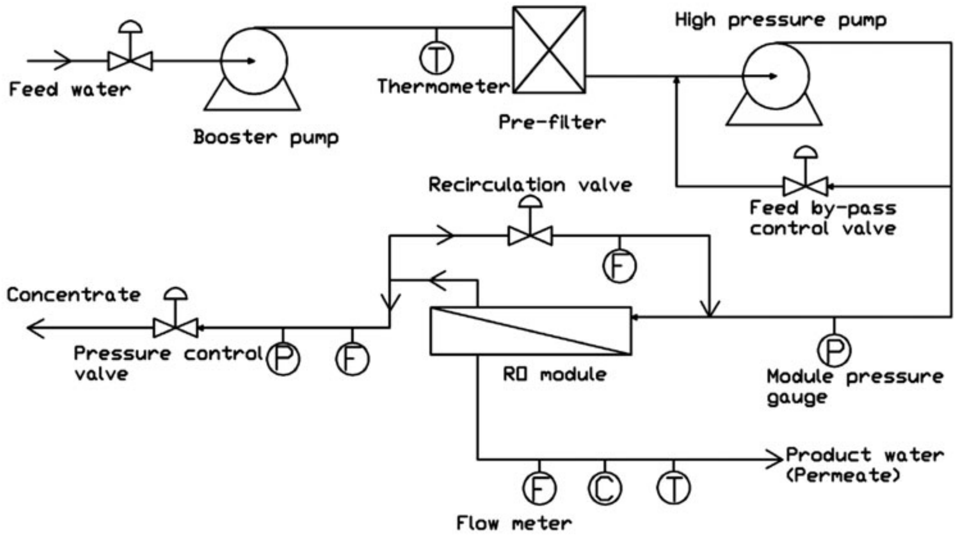

Fig. 1. Pilot-scale RO system.

adjustment of recirculation valve to control the volume ratio of permeate to concentrate. The used membrane was cleaned by flush of 20 L cleaning agents, weak acid A-11 (diluted with pure water to pH = 3), and weak base A-22 (adjusted with pure water to pH = 10), successively at 480 kPa and 25 ℃ for 30 min. The cleaning process was operated in a batch mode and the effluents from both the permeate and concentrate side were redirected to flush the membranes. The cleaning agents were provided by the same corporation as the membrane modules.

2.3. Chemical analysis

Over the course of 13 h of RO operation, the samples were collected for each determined time interval of 2 h. The water specimens for the measurement included feed water, RO concentrate, and RO permeate. The collected samples were analyzed for the DS, pH, turbidity, and alkalinity by standard method [24]. Several inorganic ions, including Na^+, NH_4^+, K^+, Mg^{2+}, Ca^{2+}, Cl^-, NO_2^-, NO_3^-, PO_4^{3-}, and SO_4^{2-} were analyzed by a Dionex ion chromatograph system (ICS-1000, USA) which is integrated with the injection valve, pump, guard and separation column (cationic or anionic ion-exchange column), column heater, suppression vary, and conductivity detector.

3. Results and discussion

3.1. Permeate flux in the two-pass RO system

Fig. 2 shows the evolution of permeate flux with time for the two-pass RO system and the corresponding variation of operating temperature over the experimental duration. In the exception of considerable permeate flux drop observed during the beginning few minutes of first-pass RO operation (data not shown), further permeate flux degradation was unnoticeable over 13 h of operation period and the permeate flux varied in the level of 26.6–33.4 L m^{-2} h^{-1} at operating pressure of 1,500 kPa in the temperature range of 24–34°C. The initial drop of the permeate flux was not observed for the second-pass RO and the permeate flux ranged at 73.7–91.2 L m^{-2} h^{-1}over the course of experiment. The permeate flux observed in the first-pass RO was significantly lower than the second-pass RO. This was apparently attributed to the higher feed–solute concentration in the first pass with DS 3.7 g/L than in the second pass with DS 0.18 g/L. The higher DS levels in the feed solution should result in a higher osmotic pressure and subsequently, the lower net differential pressure between the feed side and the permeate side, and finally, the smaller permeate flux. Furthermore, the higher DS in the first-pass feed side relative to that in the second-pass RO process would bring a severer concentration polarization, which is known to play an important role in suppressing the permeate flux. Concerning the impact of operating temperature on the permeate flux, it was clear that the temperature effect observed in the second-pass was more evident than in the first-pass. It is possible in the first-pass for the higher osmotic pressure and the consequent concentration polarization played a large role in governing water permeation and partly masked the temperature effect, while in the second-pass, the effect of operating temperature was shown to be very evident. In a recent report on RO processing of dairy industry effluents which had some proximity with the UF-treated anaerobically digested wastewater, fouling of RO membranes reduced the permeate flux by 15% within the first 5 h and by 60% after 40 h of operation [25]. The evolution of permeate flux with a long-term operation over days and weeks is required in further study.

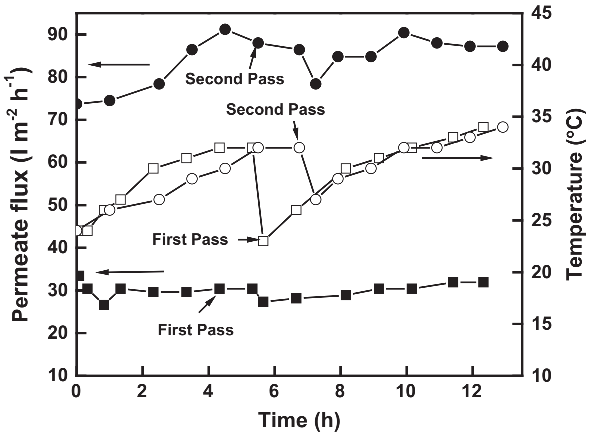

Fig. 2. The evolution of permeate flux with time for the two-pass RO system and the corresponding variation of operating temperature.

3.2. Ionic rejection

Table 2 presents the concentrations of ions containing in the feed, concentrate, and permeate solutions for the two-pass RO runs. UF-treated anaerobically digested wastewater initially contained 840 mg/L of NH_4^+-N. After the first-pass RO processing, ammonia concentration in the produced permeate was reduced to 192 mg/L of NH_4^+-N and subsequently dropped to only 63 mg/L of NH_4^+-N after the second-pass RO. The RO treatment showed a considerably higher performance for potassium removal than that of ammonia. Potassium concentration in the first-pass RO permeate was observed to be 71 mg/L. After a two-pass RO processing, the resulted water contained only 4 mg/L of residual K^+ in comparison with the initial value of 1,000 mg/L in the feed wastewater. The Na^+ concentration in the feed solution was 370 mg/L, which was significantly lower than those of initial values measured with ammonia and potassium. The RO performance for sodium was equally as high as potassium; Na^+ concentration in the second-pass RO permeate was observed to be only 1.3 mg/L. The removal of chloride by RO processing was also determined. The RO processing showed an appreciably high Cl^- rejection, yielding a product liquid with no measurable chloride after the two-pass RO treatment. It was pointed out that the first-pass RO permeate, meaning the feed of second-pass RO, was somewhat diluted by the water remaining in the two pre-filter bags.

Table 2 also presents the ionic rejections of NH_4^+, K^+, Na^+, and Cl^- for the two-pass RO runs. It was illustrated that the rejections of potassium and sodium were significantly higher than ammonia. Specifically, both first- and second-pass RO afforded rejections for sodium and potassium well above 90%, whereas for ammonia, they were only 77 and 54%, respectively. The overall rejections of both Na^+ and K^+ by the two-pass RO were greater than 99%, whereas in case of ammonia, it was approximately 88%. The significantly lower rejection of ammonia compared with the former two ions can be explained by the NH_4^+/NH_3 equilibrium in the solution. RO membranes are known to easily reject the charged ion, such as K^+ and Na^+. However, for the case of ammonia, there are two species NH_4^+ and NH_3 in the water. The zero-charged NH_3, which becomes a more prominent species in aqueous system when pH reaches pKa = 9.1 can easily diffuse through the membrane into the permeate side

Table 2
Ionic concentrations containing in the feed, concentrate and permeate solutions, and ionic rejection by two-pass RO processing (Unit: mg/L)

Ions	Stage	Feed	Concentrate	Permeate	Rejection (%)
NH_4^+-N	First pass	836 ± 41.8	1842 + 37/−30	192 + 8.49/−12.5	77.1
	Second pass	137 ± 6.8	373 + 20/−8	63 + 4.29/−4.68	54.2
K^+	First pass	1,004 ± 50	3,148 + 92/−133	71 + 9.9/−13.8	92.9
	Second pass	43.8 ± 2	241 + 5/−9	4 + 0.1/−0.12	91
Na^+	First pass	366 ± 18	1,163 + 35/−62	23.8 + 2.9/−4.0	93.5
	Second pass	17.2 ± 1	84.7 + 2.3/−4.1	1.3 + 0.03/−0.04	92.5
Cl^-	First pass	705 ± 35	1900 + 55/−101	46.3 + 3.7/−5.6	93.4
	Second pass	31.6 ± 2.0	157 + 3.6/−6.7	Undetected	100

due to its small atomic radius and zero charge. Particularly in this study, the pH value for the UF-treated anaerobically digested wastewater was 8.5 and after the first-pass, the pH increased to 9.2 so that molecular form of ammonia became more prominent. Hence, the prominent NH_3 species in higher pH solution probably induced a lower rejection of ammonia compared with ions, such as potassium and sodium. As for the anionic chloride, it showed a slightly higher rejection than the cation ions, including potassium and sodium. This trend became particularly evident in the second-pass RO, where a complete rejection of Cl^- was observed. The relatively higher rejection for monovalent anions, such as Cl^- compared with the monovalent cations, such as K^+ and Na^+ was also noted in the previous literatures [26]. This can probably be explained by the static-electric repulsion between the RO membrane surface and the anionic ions, given both are identically negatively-charged. Concerning the divalent (Ca^{2+}, Mg^{2+}, and SO_4^{2-}) and trivalent (PO_4^{3-}) ions, their contents in the tested wastewater were much lower than those of K^+ and Na^+. It was observed that their complete rejection occurred (data not shown).

Here, the behaviors of inorganic ions, especially ammonia were primarily addressed due to their significance in influencing further applicability of the product water for the reuse in anaerobic digestion process. For example, it is established that high concentration of ammonia containing in the bioreactor has a critical role in suppressing the activity of microorganisms for the transformation of organic acids to hydrogen product [4,10]. As a result, there are substantial fractions of dissolved organic acids, such as acetic acid, oxalic acid, lactic acid, and butyric acid remained in the UF-treated ADMW. Instead, in this study, the behaviors of these dissolved organic compounds were not determined. By the use of RO post-treatment, Li and his co-workers [27] recently explored the rejections of six organic compounds typically presenting in the fermentation broth, which had some similarities with the wastewater characteristics in the present study. Based on their report, the rejections of organic acids by thin-film composite polyamide RO membrane, which was chemically the same as the membrane material used in this study were mostly up to 90%, yielding an overall rejection of total organic carbon 95.5%. Therefore, a higher rejection of organic acids in UF-treated ADMW was expected.

3.3. Rejection of alkalinity and change in pH

The water alkalinities (Fig. 3) were reduced from 2,870 to 580 and 180 mg/L of $CaCO_3$ (from 57.4 to 11.5 and 3.6 mmol/L of OH^-) by the first- and second-pass RO runs, correspondingly yielding the alkalinity rejections of 79.9 and 69%, respectively. According to the thermodynamic equilibrium constant of HCO_3^-/CO_3^{2-} ($K_2 = 4.8 \times 10^{-11}$) and NH_3/NH_4^+ ($K_b = 1.8 \times 10^{-5}$), it was estimated that the species of HCO_3^- and NH_4^+ most largely dominates in the solution at a feed water pH of 8.5. As shown in Fig. 4, compared with the feed water pH of 8.5, the solution

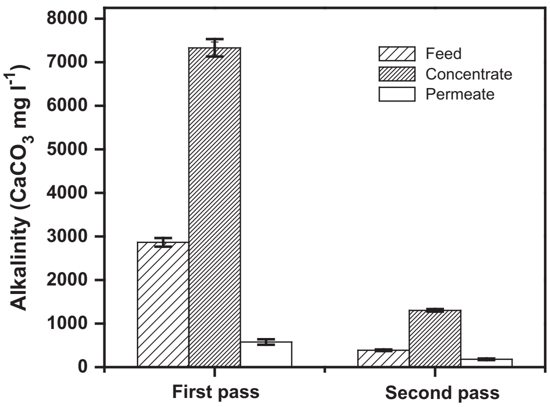

Fig. 3. The change of alkalinity by the two-pass RO systems.

pH in the first-pass permeate and second-pass permeate were increased to 9.5 and 10.3, respectively. Therefore, the amount of NH_3 retained in the first- and second-pass permeate was calculated as 8.6 and 4.1 mmol/L, accounting for 75 and 100% of residual alkalinity, respectively, retained in the two-pass permeate. Therefore, the rejection of alkalinity in the current study mostly reflected the rejection of total ammonium which appeared to be significantly lower than the rejection of other monovalent and divalent cations.

The significant increase in permeate pH (Fig. 4) could be explained in two respects. Firstly, as illustrated in above subsection, ammonia with the form of NH_3 permeated much more easily through the membranes than the other cations, such as K^+ and Na^+; the accumulation of molecular NH_3 (a relatively strong alkali) in the permeate side relative to other cations would result in the increase in pH as a consequence of NH_3 hydrolysis, which is expressed as equation $NH_3 + H_2O = NH_4^+ + OH^-$.

3.4. The improvement in water quality after the two-pass RO

Fig. 5 shows the images of water specimens in different stages of membrane treatments. From left to right, the samples referred to initially ADMW, UF-concentrate, UF-permeate, RO-concentrate, and RO-permeate, respectively. ADMW was black in color with high turbidity of 21,700 ntu. After the UF-treatment, the product water appeared to be completely transparent with brown color and turbidity reduction upto only 5 ntu. Subsequently, after the RO treatment, the resulted water became rather clear with reduction of DS from 3,750 mg/L in the UF-permeate to $225\,\mathrm{mg}^{-1}$ by the first-pass RO and to 45 mg/L by the second-pass RO (Fig. 6). The overall rejections of DS were estimated to be 94% with the first-pass RO and 98.8% after the two-pass RO. Comparable with the results achieved in this study, the RO treatment of dairy cow urine, which resembled the IMUS wastewater characteristics, yielded a product water with DS concentrations of 130 mg/L [28]. Pieters et al. [29] investigated the purification of MF-treated raw pig manure by the use of a polyamide flat-sheet RO module yielding an almost entirely complete rejection of DS. However, a poor RO performance with less than 50% of DS rejection was observed when the raw pig manure with 2.0% total solids was pretreated by electrodialysis [30]. The difference in water quality of RO permeate reported in numerous literatures should be attributable to a number of factors, such as characteristics of targeted wastewater, the pretreatment methods, selected membrane properties, and RO operating parameters.

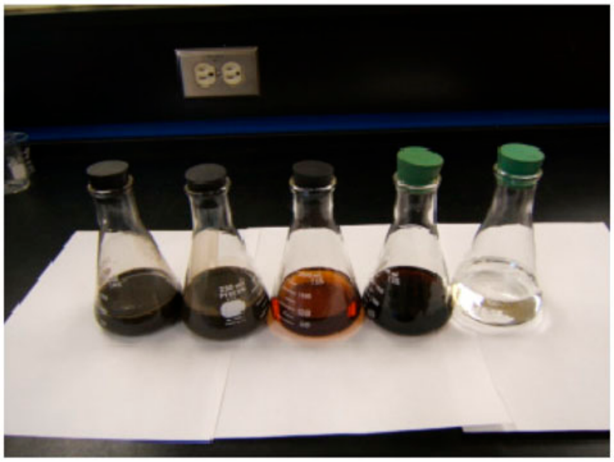

Fig. 5. Picture of water samples in different stages of membrane treatments (Solutions in flasks from the left to right, UF feed, UF concentrate, UF permeate, RO concentrate, and RO permeate).

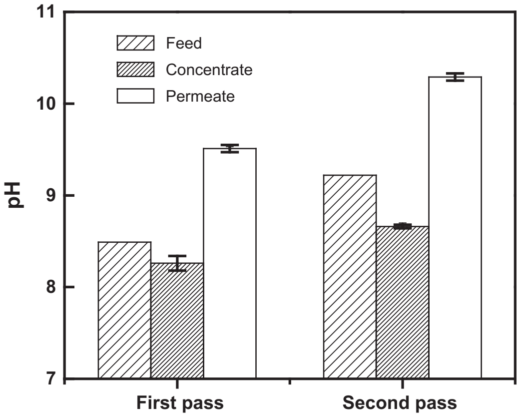

Fig. 4. The change of pH by the two-pass RO systems.

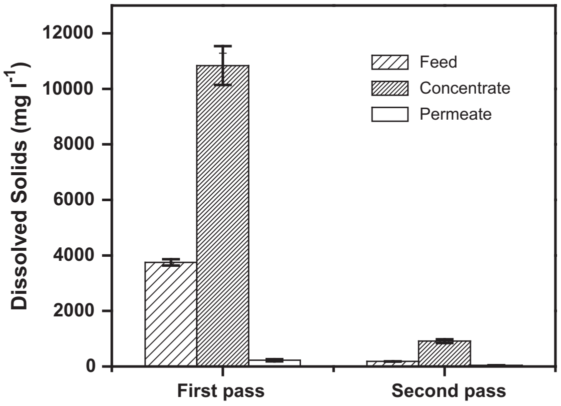

Fig. 6. The decline in concentration of DS by the two-pass RO systems.

According to the results reported above, the DS removal became almost entirely completed and the ammonia concentration was further reduced to tens mg/L after the two-pass RO, yielding the product water with properties favorable for the reuse in the anaerobic fermentation reactor. Furthermore, a higher rejection of ammonia is expected if the second-pass RO processing is conducted at the optimal pH (6–7), given the pH adjustment of the first-pass RO permeate, not like the raw wastewater, is readily available because of the low alkalinities. Ammonia retaining in the concentrate of first- and second-pass amounted to 1840 and 370 mg/L of NH_4^+-N indicating ammonia losses of 20 and 10%, respectively, probably due to volatilization during RO processing. A similar phenomenon was observed in the RO treatment of dairy cow urine wastewater [28]. High ammonia concentrations in the concentrate coupled with increasing solution temperature probably promoted the formation and volatilization of NH_3. ST was employed in our IMUS system for the advanced ammonia concentration and recovery, which could produce a liquid product with 10% ammonia for the commercial use in agriculture [21]. Besides a 3.1 g/L of K^+ cumulating in the first-pass RO concentrate, which is plant nutrient compatible with soil properties, there were substantial proportions of COD and salt remaining in the RO retentate which would be the issue for the post-treatment consideration.

3.5. Chemical cleaning of RO membranes

Fig. 7 compared the performances of three clean alternatives. It was noted here that the clean No. 1 was performed after the membranes were left for half-year without any cleaning following the course of one-month operation, while the clean nos. 2 and 3 were conducted after 13 h first-pass RO processing and one week of immersing the membrane in the wastewater. When checked with tap-water flush, the water flux of the used membranes substantially declined compared with the original membrane and would be restored neither with long-time water flush nor completely recovered by the acidic (using HCl, pH 3) and alkaline (using NaOH, pH 10) immersion. The clean No. 1 was conducted according to the manual guide by successively soaking the membrane modules overnight in the acid A-11 at pH 3 and base A-22 at pH 10. To minimize down time and water consumption, the clean Nos. 2 and 3 were conducted with appropriate modifications by successively circulating 10 L of A-11 and A-22 for 30 min with operating pressure $70\,\psi$. As seen in Fig. 7, the water flux was completely recovered by three cleaning alternatives indicating the feasibility of flush method for the membrane flux restoration. Independent of intensities of membrane fouling, the water flux at 15°C was enhanced to $53–59\,\mathrm{L\,h^{-1}\,m^{-2}}$ after the acid A-11 treatment, and subsequently increased to $68–71\,\mathrm{L\,h^{-1}\,m^{-2}}$ by the base A-22 conditioning. In the three cleaning options, the overall contributions to water flux restoration were 60–72% from the acid A-11, and 28–40% from the alkali A-22, respectively.

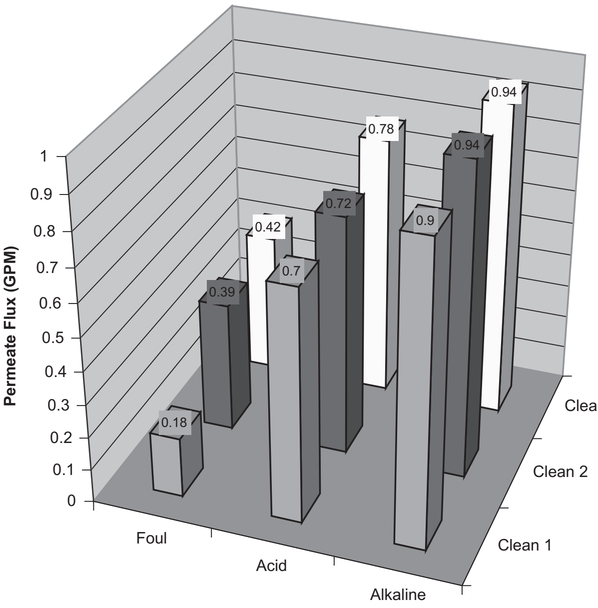

Fig. 7. Restoration of permeate flux by chemical cleaning.

The general mechanism of water flux restoration by acid flush is that it dissolves the salt precipitations, which deposit on the membrane surface during the RO processing. The further improvement in water flux deduced by alkali cleaning is probably because of the washing-out of the accumulated organic solutes. For the RO treatment of ADMW, it appears that the membrane fouling is firstly induced by salt precipitations, and secondly by the accumulation of organic solutes on the membrane surface. However, the pH adjustment in the first-pass RO to control salt precipitations is not likely to be practically feasible and cost-effective due to appreciable pH buffering capability of wastewater characteristics.

4. Conclusions

The feasibility of the two-pass RO processing for the purification of UF-treated ADMW was investigated in this study. Ammonia rejection by RO processing was especially addressed in this study, which was significantly lower compared with sodium and potassium, probably attributable to the high rate of NH_3 permeation across the membranes. Probably as a result of high diffusion of NH_3 across the RO membrane, the permeate pH was considerably increased compared with feed pH. After the two-pass RO treatment, the DS removal became almost entirely completed and ammonia concentration was further reduced to tens of mg/L, yielding product water with properties suitable for the reuse in the anaerobic fermentation reactor. The permeate flux was successfully restored by the successive flush of specific acid and base. No significant permeate flux deterioration was noticeable for either the first- or second-pass RO over the operation period of 13 h. However, the evolution of permeate flux over long-term RO processing is required in the further study. Long-term RO operation will be required in the updated studies in which the concerns should be addressed, such as the identification of predominant mechanism of membrane fouling, cleaning frequency, post-treatment of RO concentrate, minimization of ammonia loss, and economic evaluation.

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