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Importance of microbial adaptation for concentrate management in wastewater reuse process

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Abstract

Wastewater reuse has gained attention as an alternative and sustainable water resource. Reverse osmosis has been widely applied for wastewater reuse; however, generation of concentrate stream is the main drawback. Concentrate stream contains high concentrations of contaminants, and therefore, it should be properly treated prior to being discharged into a water body. Several technologies have been suggested for concentrate management, but the most common option is returning this stream to a wastewater treatment plant where a wastewater reuse plant is located. In this study, we investigated the feasibility of concentrate management by returning the concentrate to a wastewater treatment facility as a part of influent. The characteristics of the concentrate were extensively monitored, and it was verified that it contained high concentrations of salt and hardly biodegradable organics, which impede their application in biological wastewater treatment processes. The effect of seeding sludge was investigated using two different types of seeding sludge, adapted and unadapted. The adapted sludge taken at the wastewater treatment plant located at the wastewater reuse facility showed much better performance in terms of organic and nutrients removal. Moreover, the performance was recovered by a few days of additional adaptation time. However, the seeding sludge taken from another wastewater treatment plant (unadapted) showed poor performance due to different influent characteristics, especially salt concentration. Therefore, it could be concluded that the microbial adaptation step is very important for effective concentrate treatment when it is being returned to a wastewater plant as influent.

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Keywords

Reverse osmosis concentrate, concentrate management, microbial adaptation, sequencing batch reactor, dissolved organic matter

Introduction

Water recycling refers to the reuse of treated wastewater for beneficial purposes such as agricultural and landscape irrigation, industrial processes, toilet flushing, and replenishing ground water basins. With growing water scarcity worldwide, reclaimed wastewater is an increasingly attractive option for meeting water demand.¹ The most widely applied technology for wastewater reuse as a quaternary treatment is combined membrane processes such as microfiltration together with reverse osmosis (RO).² This technology employs semipermeable membranes that allow separation of a solution into two streams: RO permeate (ROP), containing the purified water that passes through the membrane, and RO concentrate (ROC), the portion that contains salts and retained compounds and therefore requires a suitable and environmentally friendly management option.³

The characteristics of the waste stream, ROC, depend on the quality of the feed water, the quality of the produced water (recovery varies from 35% to 85%), the pretreatment method (added chemicals), and cleaning procedures used.^{4–6} Then constituent concentrations in the ROC are found to be double or higher than that in feed water.⁴ A comparison of the various pharmaceuticals found in ROC revealed an average concentration factor of 3- to 4-fold that of the municipal effluent.⁷ Moreover, many chemicals were added during the wastewater reuse process as shown in Figure 1, and hence, ROC differs from the feed water or secondary effluent not only with regard to the concentration of contaminants but also in terms of the character of the organic and inorganic pollutants by virtue of the chemicals used prior to the RO treatment.^{2,8,9}

Traditionally, the disposal of concentrate has been accomplished by direct discharge to surface water, sewer disposal, evaporation ponds, deep well injection, and zero liquid discharge.^{10,11} Each disposal method has respective limitations. Furthermore, not all these disposal methods are suitable for the concentrate arising from municipal wastewater treatment. Direct discharge and sewer disposal are widely used disposal options, not only for municipal wastewater ROC⁸ but also for that generated from desalination plants.¹² A survey conducted in 2004 suggested that about 30% of desalination plants discharge the membrane concentrate to wastewater systems.¹³

More recently, several intensive reviews regarding management of ROC from wastewater treatment plants (WWTPs) were published.^{14,15} The ROC from WWTPs presents less salinity than ROC from desalination plants although larger amounts of organic matter, including persistent micropollutants, are contained. It was also reported that ROC could be more heavily contaminated than its feed water.¹⁶ Therefore, specific treatment technologies for reducing the pollutants load have been researched. From the traditional treatment technologies such as coagulation and activated carbon adsorption^{15,17} to advanced oxidation processes including ozonation, Fenton process, photocatalysis, sonolysis, and electrochemical oxidation have been reported.¹⁴ On the other hand, resource recovery from ROC has also



Figure 1. Schematic diagram of wastewater reuse process. The ROC is assumed to return as WWTP influent.

been investigated in the aim of zero-liquid discharge. The recovery of nutrients as well as salts has been investigated using evaporation, crystallization and electrodialysis.^{18,19} Moreover, bioenergy production using ROC has been also recently introduced. ROC was applied to cultivate microalgae for further bioenergy recovery while biogas (methane) production was tested for ROC from tannery wastewater.^{20–23} However, most of these technologies are still at the laboratory or pilot level, not ready to be applied in full-scale operation. The sewer discharge is still accepted because of economic and technical issues as described.

In order to achieve successful sewer disposal for ROC from wastewater, it should be ensured that there are no negative impacts on the treatment process performance and on the quality of the final effluent and biosolids for sewer disposal. Generally, ROC contains a high concentration of salt, which can bring ion imbalance-triggered toxicity to aquatic flora and fauna.^{8,24} Therefore, sewer disposal is mostly only suitable for small plants discharging into large capacity sewage treatment facilities due to the detrimental effects of the high total dissolved solid (TDS) concentration of ROC on the biological treatment process, as some inhibition may occur by high salt concentration.

In a previous study, we conducted research on the microbial acclimation strategy for saline wastewater.^{25,26} It was shown that the negative effect of salt could be minimized by step-wise acclimation of microbes without using any special halophilic bacteria. The microbial acclimation brought changes of microbial communities simultaneously. This finding is consistent with other studies dealing with saline wastewater treatment.^{24,27} Moreover,

physical characteristics of sludge floc were significantly altered by high saline wastewater, which can have negative impact on further membrane operation for wastewater reuse.²⁵

In this study, we investigated the feasibility of concentrate management by returning the concentrate to wastewater treatment facility as a part of influent. The general characteristics of ROC as well as its organic matter composition were identified. The negative impact of ROC on the biological wastewater treatment process was evaluated using real ROC obtained from a wastewater reuse pilot plant. The effect of seeding sludge was investigated using two different seeding sludges, adapted and unadapted. An in-depth study of microbial adaptation was also performed using varied salt concentration to confirm the importance of microbial adaption for effective concentration treatment.

Materials and methods

Wastewater reuse pilot plant

The wastewater reuse pilot plant was located in 'Y' WWTP in the Ulsan industrial complex. This area is one of the largest industrized areas in Korea. Industrial wastewater in the Ulsan industrial complex and domestic wastewater from Ulsan city were combined and treated in the WWTP. An advanced biological nutrient removal process is adapted and the total capacity is 250,000 m³/d.

The overall schematic diagram of the wastewater reuse process is presented in Figure 1. The main wastewater reclamation process is a combination of ultrafiltration (UF) followed by RO. The pilot plant had a total production capacity of $72 \text{ m}^3/\text{d}$. Specific operation conditions were applied as $4 \text{ mg Al}_2\text{O}_3/\text{L}$ for coagulation, 1.2 m/d for UF, and recovery of 75% for RO based on previous short-term test results.²⁸ Applied membranes for UF and RO were AQUAFLEX 40 (Pentair, 40 m^2 , $0.02 \mu\text{m}$) and TML10D (Toray, 7 m^2 , low fouling and high tolerance membrane module), respectively. The specific operation conditions are listed in Table 1.

Sequencing batch reactors for biological wastewater treatment containing RO concentrate

In this study, we investigated the effect of concentrate return on the sewage treatment plant by establishing three sequencing batch reactors (SBRs). Actually, the advanced nutrients removal process was applied in the WWTP, therefore, SBR is not only process for simulating overall WWTP. However, we conducted previous study on the effect of high saline concentrate on sludge floc. As a result, the sludge floc have been altered by addition of

Membrane module	Specifications
Membrane area: 40 m ²	Coagulation (Inline-mixer) \rightarrow UF
Pore size: 0.02 µm	Recovery rate 90% (constant flow control)
	Flux $1.2 \text{ m}^3/\text{m}^2 \text{ d}$
Diameter: 4 inch	Low fouling model
Membrane area: 7 m ² Salt rejection: 99.8%	Recovery rate 75% (constant flow control) Vessel $(2:1)$. Membrane module/vessel = 6
	Membrane module Membrane area: 40 m ² Pore size: 0.02 μm Diameter: 4 inch Membrane area: 7 m ² Salt rejection: 99.8%

Table I. Specific operation conditions for wastewater reuse pilot plant.

sludge floc. In this point of view, we chose an SBR as a model process to simulate biological wastewater treatment as well as its sludge settling ability. The aim of this study is to provide general knowledge about concentrate management, which can be applied in various WWTP having different treatment process train, so we believed the identical treatment process is not required.

The volume of the reactor was 3 L and 1 cycle consisted of feeding (30 min), aeration (180 min), settling (15 min), and decanting (15 min). The influent was then introduced again and the reaction was repeatedly performed. The initial mixed liquor suspended solid (MLSS) concentration, hydraulic retention time (HRT), and solid retention time (SRT) were set as 3000 mg/L, 8 h, and 30 d, respectively, similar to the operating conditions of the WWTP.

The specific conditions for three SBRs are presented in Table 2. In the case of SBR 1, activated sludge from 'D' WWTP was inoculated, while activated sludge from 'Y' WWTP was fed to SBR 2 and 3 in order to evaluate the effect of sludge adaptation on concentrate treatment. The activated sludge from 'Y' WWTP was already well adapted to the corresponding wastewater, and thus it was expected that the treatment efficiency would be higher than the activated sludge from 'D' WWTP, which treats domestic wastewater only. The influent TDS concentration showed a significant difference depending on regional condition of WWTPs such as 3.51 and 0.46 g/L for 'Y' WWTP and 'D' WWTP, respectively. The treatment processes applied in both of WWTPs were similar, which were operated by A2O process configuration with additional bio-carriers. Specifically, DeNiPho process was adapted to 'Y' WWTP and NPR process was applied to 'D' WWTP. Their treatment trains were basically modifications of A2O process with additional bio-carrier in the reactor in order to increase biomass loading.

In addition, only the influent of sewage treatment plant was supplied to SBR 3, while the ROC was mixed with the influent of WWTP for SBR 1 and 2 to simulate the case for ROC return. In order to determine the dilution factor of the ROC, the material balance, as shown in Figure 2, was constructed. As a result, the dilution ratio of about 6.5:1 (raw wastewater: ROC) was selected for SBR operation.

Analysis

Influent and effluent were collected every day over one month of operation, the treatment efficiency was evaluated by monitoring pH, MLSS concentration, mixed liquor volatile suspended solid (MLVSS) concentration, chemical oxygen demand (COD), dissolved organic carbon (DOC), ammonia (NH₃-N), and TDS concentration. COD, MLSS, and MLVSS were determined based on the Standard Methods.²⁹ The DOC was determined by a TOC analyzer (Apollo 9000, Teledyne Tekmar, USA) after filtration of the samples with 0.45 µm syringe filters. Ammonia was analyzed by the titrimetric method after distillation with a

	SBR I	SBR 2	SBR 3
Volume (L)	3	3	3
Seeding sludge	D (unacclimated)	Y (acclimated)	Y (acclimated)
Influent	Wastewater (Y) + concentrate	Wastewater (Y) + concentrate	Wastewater (Y)
HRT (h)	8	8	8

Table 2. Operation conditions for sequencing batch reactors for concentrate management.



Figure 2. Mass balance of wastewater reuse process assuming $100,000 \text{ m}^3/\text{d}$ production of reclaimed water.

Table 3. Water quality analysis for raw wastewater, RO concentrate, and mixture of wastewater and RO concentrate.

	Wastewater (Y)	Concentrate	Wastewater (Y) + concentrate (6.5:1)
pН	$\textbf{7.73} \pm \textbf{0.17}$	$\textbf{7.08} \pm \textbf{0.20}$	$\textbf{7.70} \pm \textbf{0.17}$
SS (mg/L)	89.4 ± 14.1	_	$\textbf{87.9} \pm \textbf{15.9}$
COD (mg/L)	152.7 \pm 26.4	$\textbf{270.5} \pm \textbf{32.3}$	$\textbf{162.8} \pm \textbf{32.3}$
NH ₄ ⁺ -N (mg/L)	34.4 ± 3.5	$\textbf{42.5} \pm \textbf{4.3}$	$\textbf{35.9} \pm \textbf{6.0}$
TDS (mg/L)	$\textbf{3,510} \pm \textbf{240}$	$10{,}530\pm620$	$\textbf{4,200} \pm \textbf{370}$

distilling unit (KJELTEC 1026, FOSS, Denmark). pH and TDS were monitored by a portable multi-parameter meter (Orion Star A325, ThermoFisher, USA). In order to characterize the dissolved organic matter (DOM) in the WWTP influent as well as the ROC, the excitation–emission matrix (EEM) was obtained using a spectrofluorometer (RF5301PC, Shimadzu) and UV-visible spectra were collected using a Shimadzu UV-2401 PC in 1 cm quartz cuvettes. All analyses were conducted as duplicate.

Results and discussion

Characteristics of concentrate stream from wastewater reuse pilot plant

The water quality of raw wastewater and a mixture of raw wastewater and ROC are presented in Table 3. The water quality was not significantly changed by the addition of concentrate at a 6.5:1 of ratio. The suspended solid concentration was slightly decreased while COD and ammonia concentration was slightly increased, although not significantly. This is due to the characteristics of ROC, which contains a very low concentration of suspended solids due to UF pretreatment and a high concentration of organics and ammonia rejected by the RO membrane. More interestingly, the average TDS concentration was increased by around 20% by the addition of concentrate.

The characteristics of DOM in ROC were extensively monitored by the EEM, as presented in Figure 3. Chen et al. operationally quantified EEM spectra by delineating the EEM signals into five regions and calculating the integrated volume under each region to



Figure 3. Excitation–emission diagrams for (a) secondary effluent, (b) RO permeate, and (c) RO concentrate.



Figure 4. Humification index and SUVA₂₅₄ for water samples. WW eff: secondary effluent; ROP: RO permeate; ROC: RO concentrate.

characterize the DOM.³⁰ We have used the regions characterized by previous studies.^{30,31} Figure 3(a) to (c) shows the EEM for the WWTP effluent, ROP, and ROC, respectively. The WWTP effluent showed peaks in the region of 240–260 nm/350–450 nm (excitation/emission), which corresponds to aromatic protein groups. Also, there is a minor peak at the region of 320–350 nm/410–440 nm corresponding to humic acid-like compounds. In the case of ROP, the overall intensity was significantly decreased, indicating a low concentration of DOM in ROP. There is a peak area in the short wavelength of excitation and emission region, which indicates the presence of aromatic proteins and soluble microbial products. On the other hand, very high intensity of peak at 320–370 nm/410–470 nm was observed in the case of ROC. This area corresponds to the humic acid-like compounds.

The EEM matrices were further analyzed by the humification index (HIX). HIX was calculated as the ratio of the emission spectrum (excited at 255 nm) integral over the spectral range of 434–480 nm, to the integral of the emission spectrum over the spectral range of 330–346 nm (excited at the same wavelengths).³² It describes the diagenetic state of the DOM. High HIX values are characterized by high molecular weight, aromatic humic acid. Figure 4 presents the HIX for several water samples, secondary effluent, ROP, and ROC. HIX was 1.632 for secondary effluent. ROC has a slightly higher HIX, 1.975. In previous studies, a very low HIX (0.22–0.83) was reported for raw wastewater, but HIX was

increased through biological wastewater treatment processes.³³ The obtained results indicated that the aromatic degree of DOM would increase gradually with microbial degradation, and therefore, we can conclude that the secondary effluent used in this study mainly contains hardly biodegradable humic-acid like organic matter, which is the residual content from biological wastewater treatment processes. On the other hand, a significantly lower HIX, 0.214, was obtained for ROP. The low HIX value indicates a higher portion of protein-like components, which have small molecular weight compared to humic-acid like substances.

A similar trend could be found in the analysis of the SUVA₂₅₄ value, which is the ratio of the decadal absorbance at 254 nm to DOC (Figure 4). Typically, SUVA₂₅₄ ranges from 1.2 to 2.6 L/mg·m for secondary effluent.³⁴ The SUVA₂₅₄ value for the secondary effluent in this study, 1.632 L/mg·m, was also within the reported range. This value was slightly increased in ROC while a significant decrease was observed in ROP. Because SUVA₂₅₄ is a measure of the aromaticity of DOM, we could conclude that aromatic organic matter was retained by the RO process.

These results clearly showed the removal characteristics of organic matter in the RO process. Most of the organic matter was retained by the RO membrane, which is consistent with the high concentration of COD in ROC. However, a small portion of organic matter in the group of aromatic protein and soluble microbial products was also detected in ROP. These groups of organics have lower molecular weight compared to humic acid groups, and therefore, they could penetrate the RO membrane. From this result, we can conclude that ROC contains a high concentration of organic matter, mainly humic acid-like substances having high molecular weights that are generally hardly biodegradable. This result agrees well with previous studies reporting biodegradability of ROC. Most of the organic content present in ROC is known to be bio-refractory, and biological processes are generally considered to be ineffective for its treatment.^{2,17} Therefore, it is necessary to investigate the negative impact of ROC addition as an influent of biological processes.

Effect of seeding sludge on SBR operation

In order to investigate the effect of microbial adaptation on ROC management, two types of seeding sludge from different WWTPs were used. SBR 1 was seeded by activated sludge taken from 'D' WWTP, which treats domestic wastewater only, while the activated sludge taken from 'Y' WWTP was inoculated to SBR 2. It was hypothesized that the activated sludge taken from 'Y' WWTP would be well adapted for specific wastewater, and therefore, it would show better treatment efficiency.

Figure 5 presents the overall operation performance including COD, ammonia, and SS removal as well as MLSS concentration during 1 month of operation. In the case of COD, ammonia, and SS removal rates, SBR 2 was higher than SBR 1, which is considered to be due to the high adaptability of activated sludge taken from 'Y' WWTP used in SBR 2. Specifically, the COD removal rate was stabilized at 50% after 3 weeks of operation, while ammonia removal was stabilized at 85% in 10 days of operation. The SS removal rate was stabilized at 50% from initial operation. On the other hand, a very low removal rate was observed for SBR 1. After 3 days of operation, the COD, ammonia, and SS removal rates traises of influent including high salt concentration caused by ROC. The removal rate was then restored with respect to time because of microbial adaptation. Similar recovery was



Figure 5. SBR operation performance for concentrate management. (a) COD removal efficiency (%), (b) ammonia removal efficiency (%), (c) SS removal efficiency (%), and (d) MLSS concentration. Note that a mixture of raw wastewater and concentrate was the influent for both SBR I and 2. SBR I was inoculated with unacclimated activated sludge while SBR 2 was inoculated with acclimated sludge.

reported in our previous study using different salt concentrations in membrane bioreactor operation.²⁵ Among three water quality indexes monitored in this study, ammonia removal was the most sensitive. According to Moussa et al., ammonia oxidizers are more sensitive to salt stress than heterotrophs removing organic matter.³⁵ This indicates that freshwater nitrifiers are very sensitive to high salinity, and once they are affected or washed out, considerable time will be required to reestablish them due to their slow growth rates. Therefore, the longer recovery period for ammonia removal than that of COD removal could be caused by the high sensitivity of the ammonia oxidizer to salinity.

Figure 5(d) presents the MLSS concentration in SBRs. SBR 2 showed a more positive trend of increase, which indicates the growth of activated sludge with respect to time. In summary, the adapted activated sludge taken from the same WWTP was able to adapt very well to the addition of concentrate as influent, while the unadapted sludge took much longer time to acclimate. This shows the possibility to return the ROC as an influent if there is a proper adaptation strategy.

Effect of concentrate return on SBR operation

A similar experimental approach was applied to investigate the effect of ROC return on its management. SBR 2 and 3 were inoculated by the same seeding sludge taken from 'Y' WWTP as the acclimated sludge. SBR 2 was fed with a mixture of raw wastewater and ROC, while SBR 3 was fed with raw wastewater only. As shown in Figure 6, it was clear that the addition of ROC into the influent had a negative impact on the bioreactor operation. For example, the ammonia removal rate declined significantly during initial reactor operation. The COD removal rate was also less than 20% during the first week.

However, the low removal efficiency in the initial phase was rapidly recovered and the efficiency reached a similar value to that of the reactor fed by raw wastewater only. It took around 10 days of operation until the overall treatment efficiency was fully recovered. This result clearly showed that ROC return to the WWTP could be considered as an option for proper ROC management with a proper microbial adaptation strategy. The high concentration of MLSS in SBR 2 was due to the accumulation of salt adsorbed in microbial floc.



Figure 6. SBR operation performance for concentrate management. (a) COD removal efficiency (%), (b) ammonia removal efficiency (%), (c) SS removal efficiency (%), and (d) MLSS concentration. Note that both SBR 2 and SBR 3 were inoculated with acclimated activated sludge. SBR 2 was fed by a mixture of raw wastewater and concentrate while SBR 2 was fed by raw wastewater only.

Conclusions

Concentrate management is one of the most important considerations for practical application of wastewater reuse. Returning the concentrate to a WWTP can be the simplest approach, but negative impact on microbial processes should be carefully investigated because the concentrate contains high concentrations of TDS and hardly biodegradable DOMs, which impedes degradation in biological processes. In this study, microbial adaptation was evaluated for stable operation of a biological wastewater treatment process. Acclimated sludge taken from the same WWTP did not suffer a significant negative impact by the addition of concentrate. There was initial inhibition caused by the concentrate, but the overall performance could be recovered within 10 days. On the other hand, unacclimated sludge was not effective when it was exposed to concentrate, which clearly indicates the importance of microbial adaptation when treating ROC. Therefore, combined treatment of concentrate in a WWTP can be considered as a management option if microbial adaptation is carefully taken into account.

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