Reliability and efficiency of an advanced tertiary treatment process for wastewater reclamation
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ABSTRACT
A reliability study for the reclamation of wastewater treatment plant effluent using continuous sand filtration-multimedia filtration (CSF-MMF) combined with ultrafiltration (UF) and reverse osmosis (RO) has been conducted. The objectives of the research are two-fold: (1) effluent of CSF-MMF can be used for surface water supplementation and (2) permeate of UF-RO can be applied as greenhouse irrigation water. The removal efficiencies for nutrients and electric conductivity (EC) as well as some operational parameters of the pilot plant were investigated. The concentration of T-N, chemical oxygen demand (COD) and turbidity in the filtrate of CSF-MMF with external C-course methanol dosage and FeCl₃ dosage could be kept at less than 2.2 mg/L, 35 mg/L and 0.9 NTU respectively. Average EC exceeded the required surface water standard by 10% and it was difficult to meet the low surface water standard for T-P (below 0.15 mg/L). The EC of RO permeate was below 20 μS/cm, which was much lower than the standard for greenhouse irrigation. With frequent back flushes, cleaning in place (CIP) and enhanced cleaning the UF could be operated with a constant permeability of 100 L/(m²·h·bar). An appropriate CIP resulted in a recovery of 47–52% of the RO. The protective cartridge filter prior to the RO should be replaced every 2 weeks.

Key words | continuous sand filtration (CSF), multimedia filtration (MMF), reverse osmosis (RO), ultrafiltration (UF), waste water reclamation

INTRODUCTION
Increasing scarcity of freshwater resources and growing environmental awareness give rise to the use of reclaimed wastewater as an additional source of water supply on a worldwide scale, especially in areas where the climate is (semi) arid and/or the population and economic growth is fast (Hochstrat et al. 2006; Yang & Abbaspour 2007). Municipal sewage is promising as an alternative water resource and can satisfy non-potable water requirements such as streams in recreational parks, toilet flushing, irrigation and so on (Hidaka et al. 2003). Advanced treatment at existing wastewater treatment plants (WWTPs) is expected to provide a more feasible solution for wastewater reclamation compared to optimizing operation and upgrading of existing conventional biological nutrient removal steps.

It is indispensable to develop a cost-effective, compact and easy to maintain treatment scheme for sewage reclamation, which is capable of the removal of both nutrients and ions to some extent according to the required quality for reuse. In order to produce reliable and reusable water from WWTP effluent for European Water Framework Directive (WFD) and/or greenhouse irrigation water, an advanced effluent treatment scheme on a pilot scale was developed. This pilot consists of two steps: (1) continuous sand filtration (CSF) followed by multimedia filtration...
(MMF) and (2) a dual membrane filtration (ultrafiltration followed by reverse osmosis, UF-RO). The first step focuses on nutrient removal and the second step on ions rejection.

CSF is an innovative sand filtration technique that removes suspended solids (SS) physically through sand filtration and performs nitrogen removal by the nitrification and denitrification bacteria enriched in the biofilm developed on the sand particles (Sin et al. 2008; Xu et al. 2012). CSF has been successfully applied to tertiary denitrification in both industrial and municipal WWTPs. A stable denitrification efficiency of more than 83% could be reached for longer periods with appropriate influent dissolved oxygen (DO) and NO₃-N, methanol dosage and airlift velocity.

Although the phosphorus concentration of the secondary effluent is already low, it still exceeds the surface water standards. Precipitation with Fe³⁺ combined with MMF is regarded as a simple and effective method to remove phosphorus (as orthophosphate). MMF with sand and anthracite was found to be the most effective in aspects of pressure drop and breakthrough (Xie et al. 2005). Besides precipitation of phosphate, coagulation and flocculation of fine suspended material will occur, improving the overall SS removal of the continuous sand filtration-multimedia filtration (CSF-MMF) step. In the pilot study, the combination of CSF-MMF is introduced to reclaim water suitable for surface water supplementation.

The combination of UF followed by RO has become a viable technology for effluent reclamation due to its good performance in high rejections of conductivity, viruses and turbidity (Wilf & Alt 2000; Abdel-Jawad et al. 2002; Qin et al. 2006b, 2010; Zhao et al. 2010). Three factories with a total capacity of 92,000 m³/d began supplying NEWater to wafer fabrication plants and other industries for non-potable reuse in Singapore. The polymeric RO membrane could tolerate organics from the industrial wastewater and performed >96% salt rejection (Qin et al. 2006a). However, membrane fouling is still a major technical hurdle that needs to be addressed to enhance a cost-effective operation of RO processes for wastewater reclamation. There are still considerable uncertainties about the long term performance of a dual membrane process regarding membrane fouling and final greenhouse irrigation water quality.

The objectives of this pilot study are to investigate the reliability and efficiency of CSF-MMF followed by UF-RO to reclaim effluent of WWTP for surface water supplementation and greenhouse irrigation water.

**MATERIALS AND METHODS**

**Description of pilot process**

The schematic diagram of the pilot process is shown in Figure 1. Effluent of WWTP is pumped to a high level buffer tank to supply water to the pilot. The effluent flows by gravity to the CSF and the flow can be adjusted.

**CSF**

The characteristics and main relevant operational parameters of the pilot CSF are given in Table 1. Influent is fed into the filter via a feed pipe and distribution arms in the lower section, and it then flows upwards through the downwards moving sand bed, reaching the effluent outlet at the top. The intensive contact between water and sand particles induces biofilm growth on the particles. From the bottom part, the sand with biofilm and water is pumped upwards by an airlift pump through a central pipeline. At the top of the pipeline the air escapes and the dirty water is discharged. At the bottom of the washer the sand is released to the filter bed. In this way, the sand particles are re-circulated continuously and filtration remains in service without interruption. Methanol is added into the inflow for denitrification (Xu et al. 2012). The effluent of CSF is stored in a buffer tank. The operation of the CSF is controlled by a programmable logic controller (PLC).

Since the effluents of WWTP are usually very low in organic carbon, an external carbon source is needed to accomplish the denitrification. Methanol is most commonly used as an external carbon source which serves as the electron donor and facilitates the denitrification process (Timmerman & van haute 1983; Hamlin et al. 2008). The methanol dosage is calculated according to Equation (1) (Timmerman & van haute 1983; Koch & Siegrist 1997; Foglar & Briški 2003).

\[
[\text{CH}_3\text{OH}] = 2.47[\text{NO}_3^-\cdot\text{N}] + 0.87[\text{DO}]
\]  

(1)

[NO₃⁻·N] and [DO] represent the influent nitrate and DO concentrations (in mg/L) respectively.
MMF

Effluent of the CSF is pumped to the MMF and the coagulant FeCl₃ is added simultaneously. Precipitation, coagulation and flocculation occur in the supernatant of the MMF and in the upper part of the filter bed. The filter bed of the MMF consists of two layers, an upper layer of anthracite and a bottom layer of sand. The filtrate is collected by collection pipes at the bottom. The MMF is operated discontinuously and backwashed by air and water for solid removal when the head loss is over a certain limit. The filtrate of the MMF is stored in a buffer tank and is served as backwash water and UF feed. The backwash water is discharged through a drain pipe located in the middle-top of the MMF. For control of operation and backwash a PLC is installed. The characteristics and main relevant operational parameters are given in Table 2.

Table 1 | Characteristics and operational parameters of the pilot CSF

| Description           | Unit    | Value  |
|-----------------------|---------|--------|
| Cross-sectional area  | m²      | 0.7    |
| Sand bed height       | m       | 2      |
| Sand particle diameter | mm    | 1.4–2.0|
| Flow rate             | m³/h    | 9–10   |
| Hydraulic loading rate | m/h    | 13–15  |
| Wash water            | %       | 5–10   |
| Sand velocity         | mm/min  | 10–20  |
| Methanol dosage       | Kg COD/kg NO₃-N | 6–7.5 |

UF

The filtrate of the MMF is pumped to the UF by a feed pump. The UF unit is applied as pre-treatment prior to the RO to remove fine SS, coagulated colloidal materials and bacteria (Qin et al. 2010). The UF is operated in the dead-end mode. The UF unit contains a Capillary multi-bore 67 module (Innovative Membrane Technologies B.V.,
The Netherlands), which is a polyethersulfone hollow fiber membrane with a molecular weight cut-off of 100–150 kDa. The total membrane area is 50 m². Membrane fouling is controlled by a back flush after every 625 L of filtrate production, a chemical enhanced back flush with sodium hypochlorite solution (12.5% NaClO) after every five back flushes, and a Cleaning-In-Place (CIP) with a sodium hydroxide solution (2% NaOH) and citric acidic solution (50% C₆H₈O₇·H₂O) when transmembrane pressure (TMP) exceeds 1 bar. The operation of the UF is controlled by a PLC.

**RO**

To upgrade the water quality after UF, monovalent ions like Na⁺ and Cl⁻ and other compounds have to be removed. The RO unit consists of three XLE-4040 Extra Low Energy spiral wound modules (Dow Chemical Company, The Netherlands), which are made of polyamide thin film composite. The RO unit is designed to operate at a high feed water velocity maintained by a re-circulation pump. Fouling and scaling of the RO are controlled by a CIP system for chemical cleaning with a sodium hydroxide solution (33% NaOH) and citric acidic solution (50% C₆H₈O₇·H₂O) when transmembrane pressure (TMP) exceeds 1 bar. The operation of the UF is controlled by a PLC.

### Results and Discussion

**Characteristics of the raw water**

The effluent of WWTP served as pilot feed water. The quality is presented in Table 3. The values in Table 3 for Na⁺, Mg²⁺, Cl⁻, SO₄²⁻ were from documentary records of WWTP. These values indicate that COD, NH₄⁺-N, NO₃⁻-N, T-N, PO₄³⁻-P, T-P and turbidity fluctuated significantly.

## Sample Analysis

The flow rates of influent and effluent of the CSF and MMF were measured by flow meters. COD, NH₄⁺-N, NO₃⁻-N, T-N, PO₄³⁻-P and T-P were analyzed using Hach Lange cuvette tests. T, pH were measured by handheld meters (pH 315i, WTW company). Samples of the feed and permeate of the UF and RO were analyzed for temperature (T), pH, electric conductivity (EC) and turbidity, which were monitored with on-line instruments equipped in the UF and RO units continuously.

### Pilot Setup

The CSF and MMF were operated to check for leakage and that components were working properly after being placed on 5th February and 9th February respectively. After 25th February, methanol (5%) and ferric chloride solution (40% FeCl₃) were dosed into the inflow of the CSF and MMF, respectively. The flow of CSF and MMF were controlled at 9 and 5 m³/h, respectively. After three weeks a removal of NO₃⁻-N in the CSF was observed, which indicated the growth of denitrifying bacteria in the CSF.

The UF and RO units were in operation from 10th March and 3rd April, respectively.

### Table 2

| Description                        | Unit | Value |
|------------------------------------|------|-------|
| Cross-sectional area               | m²   | 0.5   |
| Bed height of anthracite           | m    | 0.8   |
| Anthracite particle diameter       | mm   | 1.4–2.0 |
| Bed height of sand                 | m    | 0.4   |
| Sand particle diameter             | mm   | 0.7–1.3 |
| Supernatant level                  | m    | 2.0   |
| Flow rate range                    | m³/h | 5     |
| Hydraulic loading rate             | m/h  | 10    |
| Backwash velocity                  | m/h  | 12–25 |
| Backwash water                     | %    | 3–8%  |
| Backwash air                       | Nm/h | 60    |
| FeCl₃ dosage                       | mol Fe/mol P | 3–5  |
Target water quality

The objectives of the pilot are to produce surface water with CSF-MMF and greenhouse irrigation water with the dual membrane process. Some of the general maximum tolerable risk (MTR) standards for surface water in the Netherlands are summarized in Table 4. The main limits of the MTR for surface waters in relation to effluent reclamation are T-N below 2.2 mg/L and T-P below 0.15 mg/L. Greenhouse irrigation water requires EC below 500 μS/cm.

| Parameter | Unit | Value |
|-----------|------|-------|
| pH        |      | 6.5–9 |
| COD       | mg/L | 45    |
| NH₃-N     | mg/L | 0.02  |
| T-N       | mg/L | 2.2   |
| T-P       | mg/L | 0.15  |
| SS        | mg/L | 15    |
| Turbidity | FTE  | 0.9   |
| EC        | μS/cm| 900   |
| Cl⁻       | mg/L | 200   |
| SO₄²⁻     | mg/L | 100   |

*Data of Harnaschpolder WWTP effluent.

NO₃⁻-N removal in CSF and methanol dosage

After the CSF was placed and operated well, methanol (5%) was dosed into the inflow to cultivate a biofilm around the sand particles including denitrifying bacteria and heterotrophic bacteria. The flow rate and dirty water rate were controlled at 9 and 0.7 m³/h, respectively. From 15th March more than 50% NO₃⁻-N removal was observed in the CSF, which meant the denitrification in the CSF had been accomplished gradually. The NO₃⁻-N removal rate fluctuated from 14.3 to 96% and the average NO₃⁻-N removal rate reached over 60.9% in the four-month study period. The average effluent NO₃⁻-N was kept below 1.0 mg/L, as shown in Figure 2. Pure methanol dosages were 14.4 and 28.8 mg/L before 23rd April and after 24th April respectively. It also indicates that the CSF had a higher and more stable NO₃⁻-N removal rate when the influent NO₃⁻-N is over 4 mg/L. In some commercial CSF it was found that a high influent NO₃⁻-N concentration (above 10 mg/L) may be the reason for high removal rates of more than 85–95% NO₃⁻-N removal.

The DO of the effluent from the clarifiers ranged from 3.6 to 5.5 mg/L. According to Table 3, the average NO₃⁻-N concentration was 4 mg/L. In accordance with Equation (1), the theoretical dosage (TD) of pure methanol is calculated as follows:

\[ [\text{CH}_3\text{OH}] = 2.47 \times 4.0 + 0.87 \times 5.0 = 14.4 \text{ mg/L} \]

In the pilot research, NO₃⁻-N removal rates at different methanol dosages are shown in Figure 3.

The methanol dosage was adjusted at three levels, 1.0, 2.0 and 2.6 TD. Figure 3 shows that NO₃⁻-N removal rates improved slightly with increasing methanol dosage. Though no definite increase of NO₃⁻-N removal was observed when the methanol dosage was doubled from 1.0 to 2.0 TD, NO₃⁻-N removal rates and the effluent NO₃⁻-N values were both more stable when increasing the methanol dosage. A similar phenomena was observed where NO₃⁻-N removal remained constant in real wastewater treatment when increasing methanol/NO₃⁻-N ratios above 3.5 (Foglar & Briški 2003). As shown in Figure 3, at a methanol dosage of 1.0 TD the NO₃⁻-N removal rate fluctuated dramatically, even if the influent NO₃⁻-N was at a relatively
high and stable level. It seemed that a high methanol dosage could enhance the stability of the NO₃⁻-N removal in the pilot. High methanol dosage may eliminate the influence on denitrification of environmental conditions, such as influent DO, temperature and so on.

**PO₄³⁻-P removal and FeCl₃ dosage**

Usually phosphorus in the clarifier effluents is mostly soluble and presents as orthophosphate (PO₄³⁻-P) (Dueñas et al. 2003). Consequently, tertiary treatment of secondary municipal effluents to remove PO₄³⁻-P has become increasingly necessary to meet environmental regulations worldwide (Wei et al. 2008).

PO₄³⁻-P removal was studied in CSF and MMF. Figure 4 shows PO₄³⁻-P removal with and without FeCl₃ dosage (5 mol Fe/mol PO₄³⁻-P) in four months of pilot research.

From 7th March to 12th April, FeCl₃ dosage of 5 mol Fe/mol PO₄³⁻-P was applied to the influent of the MMF. PO₄³⁻-P concentration in the MMF effluent was as low as 0-0.26 mg/L (only one sample of eight was above 0.15 mg/L). The average MMF effluent PO₄³⁻-P was below 0.06 mg/L and the PO₄³⁻-P removal rate reached 73.8%. The average orthophosphate concentration in the CSF effluent was low at <0.10 mg/L when the influent PO₄³⁻-P was low (0.3 mg/L). A similar phenomenon could be observed after 21st July. Orthophosphate removal in the CSF was due to uptake by the biofilm bacteria.

From 14th April to 20th July FeCl₃ dosage was cancelled because of low orthophosphate concentrations in the effluent of WWTP. MMF effluent PO₄³⁻-P could be as low as 0.1 mg/L when the WWTP effluent PO₄³⁻-P was below 0.3 mg/L. However, when PO₄³⁻-P exceeded 0.4 mg/L the PO₄³⁻-P removal efficiency was very poor.

At PO₄³⁻-P in the WWTP effluent lower than 0.3 mg/L, the removal by biological uptake in the CSF and coagulation/flocculation in the MMF is just enough to bring total phosphate below the standard (0.15 mg/L). According to Table 3 approximately 50% of the total phosphorus in the effluent is in the form of orthophosphate.
This means that with a complete removal of orthophosphate the remaining total phosphorus concentration is still twice as high as the required standard when WWTP effluent PO$_4^{3-}$-P exceeding 0.3 mg/L.

**Permeability of UF unit**

The permeability of the UF unit during the study period is shown in **Figure 5**. During April, membrane fouling was programmed by a back flush after every 625 L of filtrate production, a chemical enhanced back flush with sodium hypochlorite solution (12.5% NaClO, CIP caustic) after every five back flushes. Manual caustic, acidic or caustic and acidic was applied frequently because of an unstable UF process. In May, the second running month, only automatic water backflush and caustic CIP was used for permeability recovery. The relatively high frequency of the caustic CIP means that this cleaning was not effective for the permeability recovery. On 14th and 17th June, a combined caustic/acidic CIP was carried out twice, which was followed by a caustic CIP 7 days later. After 24th June, the permeability of UF could be kept at 100 L/(m$^2$·h·bar) for more than one month at a recovery rate of 60–80% only with programmed back flush. Before 21st June the UF
influent temperature was 17–19 °C and COD was above 40 mg/L. After the end of June, UF influent temperature was above 21 °C and COD was 32–35 mg/L. These factors may be the reasons for UF running well in June and July. Low temperature and high COD may be the reason for unstable running before June. The performance of the UF unit demonstrated that if the chemical enhanced back flush and CIP were applied well, the UF unit can run successfully in a treatment scheme for reclamation of effluent of WWTP.

**Performance of RO unit**

Figure 6 shows the post cartridge pressure and TMP of the RO unit during the study period. From May to July, the RO performed very well with one cartridge replacement nearly every 2 weeks and a CIP at 47–52% recovery rate. The post cartridge pressure was maintained at 2.5–3 bar. When TMP reached 1 bar, CIP ran automatically.

**EC and pH of dual membrane**

Figure 7 shows the EC of UF feed, UF permeate and RO permeate during the study period. The EC of the UF feed ranged from 807 to 1,106 μS/cm. UF has no effect on the EC, which is logical because UF does not remove ions. The EC of the RO permeate was very low at 9–17 μS/cm.

Average values for the pH of raw water, CSF effluent, MMF effluent, UF permeate and RO permeate are shown in Figure 8. The pH of CSF effluent increased slightly due to the formation of hydroxyl ions during denitrification. The pH of RO permeate was 5.5–5.9, which was 1.1–1.5 units lower than the pH of feed water. This agrees with the 1.0–1.3 unit pH drop of RO permeate for reclamation of secondary treated sewage effluent (Qin et al. 2006a). RO can remove ionic substances efficiently but cannot remove gases. There is a dynamic equilibrium among CO2, HCO3− and CO32− in water. When HCO3− and CO32− are removed, CO2 in water reduces pH. This situation is quite common in RO processes (Walker et al. 1994) and pH of 5.5–5.9 in greenhouse irrigation water is not harmful for crops (van Rotterdam-Los et al. 2008).

**Targets accessibility**

Filtration (CSF and MMF) and dual membrane filtration (UF-RO) in the pilot study are aimed at producing surface water and greenhouse irrigation water, respectively. The range and average values of some parameters of raw water, MMF effluent and RO permeate are summarized in
Table 5 together with the standards for surface water and greenhouse irrigation water, respectively.

Table 5 shows that pH, turbidity, and COD of MMF filtrate meet the surface water quality standards. T-N ranged from 1.1 to 3.5 mg/L and the average value is below the required 2.2 mg/L (two out of nine samples are above 2.2 mg/L). However, T-P varied from 0.1 to 0.5 mg/L and the average value is above the standard of 0.15 mg/L. Because only 50% of the phosphate is present as orthophosphate, it is not possible to meet the low surface water standard for T-P with only iron precipitation. The EC of the CSF-MMF filtrate varied from 680 to 1,270 μS/cm. The average value of 998 μS/cm exceeded the standards by approximately 10%. Turbidity changed from 0.4 to 1.9 mg/L and the average value is below the required 0.9 mg/L (four out of 39 samples are above 0.9 mg/L).

The EC of the RO permeate varied from 7 to 15 μS/cm with an average of 10.5 μS/cm, which was well below the required value for greenhouse irrigation water.

| Parameter   | Unit | MMF effluent | Standard for surface water | RO permeate | Standard for greenhouse irrigation water |
|-------------|------|--------------|-----------------------------|-------------|------------------------------------------|
| pH          |      | 7.0–7.7      | 7.2                         | 5.5–5.8     | /                                        |
| T-N         | mg/L | 1.1–3.5      | 2.0                         | <2.2        | /                                        |
| T-P         | mg/L | 0.1–0.5      | 0.3                         | <0.15       | /                                        |
| Turbidity   | NTU  | 0.4–1.9      | 0.8                         | <0.9        | /                                        |
| COD         | mg/L | <35          | /                           | <45         | /                                        |
| EC          | μS/cm| 680–1,270    | 998                         | 7–15        | <500                                     |
CONCLUSIONS

(1) CSF can be successfully applied for denitrification of WWTP effluent. The NO$_3$-N removal rates were between 14.5 and 95% and the average removal rate was 60.9% with a proper methanol dosage, which was in agreement with the denitrification efficiency of CSF (Scherrenberg et al. 2008; Xu et al. 2012). The NO$_3$-N removal rate fluctuated widely because of the varying nitrate concentration of the CSF influent.

(2) Filtrate pH, COD, T-N and turbidity of CSF followed by MMF can meet surface water standards. Average EC exceeded the required standard by approximately 10%, because filtration cannot remove ions. Approximate 50% of phosphate in the WWTP effluent is present as orthophosphate and it is difficult to meet the surface water standard for T-P (Scherrenberg et al. 2012).

(3) Dual membrane filtration (UF-RO) performed successfully in the production of greenhouse irrigation water (Qin et al. 2006a). The EC of RO permeate is well below the required value. The UF unit can be used as a pre-treatment for RO in reclamation of effluents from WWTPs. Fouling of the UF membranes can be controlled with frequent back flushes and chemical enhanced back flush cleaning. The protective cartridge prior to the RO unit should be replaced nearly every 2 weeks and the RO can have a 47–52% recovery rate with an appropriate CIP.

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