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Pretreatment in membrane processes for ultra-pure demineralized water production from secondary effluent

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ABSTRACT

Reverse osmosis pretreatment methods for membrane processes were investigated in steam production from secondary-treated municipal effluent (SE). The performance of combined full-scale biological aerated filter (BAF), coagulation, sand filtration and ultrafiltration (UF) processes were assessed. Results show that higher nitrification and denitrification performance were achieved via the CN/DN (nitrification and denitrification) BAF process, when the hydraulic retention time (HRT) was 1 d, with a C/N of 4. About 96.1%, 98.3%, 71.4% of the total nitrogen (TN), ammonia nitrogen (NH_4^-–N) and phosphate were removed, respectively. Total suspended solids and volatile suspended solids can be removed efficiently using coagulation and sand filtration processes. Monovalent cation elements (K^+, Na^+) and anions (Cl^- and SO_4^{2-}) were present at higher concentrations during the whole process. Silicate, calcium and magnesium were co-precipitated to some extent during the coagulation stage, with hardness further removed by the sand filter and UF stages. The level of dissolved organic carbon decreased sharply in sand filter and UF processes. Excitation–emission-matrix spectra, UV_{254} and SUVA results confirmed that a significant proportion of aromatic organics (containing C=C or C=O groups) can be destroyed by a combination of BAF and coagulation processes, with UF further reducing any residual aromatic organics following sand filtration. This study provides useful information for the production of reclaimed water from secondary effluent under zero liquid discharge conditions.

Keywords: Pretreatment; Reclaimed water; Secondary effluent

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1. Introduction

Reclaimed water from secondary-treated municipal effluent (SE) in municipal wastewater treatment plants has been recognized as a potentially valuable alternative global water resource [1]. In comparison with the levels of residual constituents in industrial effluent or sea water, SE contains significantly fewer constituents (less than 1%), including nutrients (phosphate and ammonia), organic matter and heavy metals. In industrial scenarios such as power production plants, for SE to be reused for steam production or processing water, ultra-pure demineralized water with a conductivity of less than 1 mS/cm must be produced.

In steam production systems, the water constituents become concentrated, with the elevated mineral concentrations and high water temperatures enhancing severe mineral deposition (scaling), corrosion and biofouling [2]. Therefore, effective and intensive control of mineral deposition is essential when using SE as the sole source of water in a recirculating cooling system [3,4].

To achieve a high level of purification of wastewater, membrane processes such as ultrafiltration (UF) followed by reverse osmosis (RO) or ion exchange, have commonly been adopted as tertiary treatments. The use of tertiary treatment allows

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effective reuse of secondary effluents by removing residual constituents, such as organic or inorganic micropollutants and hardness [5,6]. However, these membrane technologies can only remove a portion of dissolved organic matter (DOM) due to the limited retention capacity of membranes. In addition, membrane permeability is reduced as a result of membrane fouling by high concentrations of DOM in wastewater.

Therefore, pretreatments must be combined with membrane filtration processes to reduce the pollutant load, and the assessment of different pretreatment processes in isolation and in combination are of great importance for achieving a high level of contaminant removal efficiency.

Some studies have previously reported the effectiveness of pretreatments in membrane filtration, with flocculation and adsorption identified as the most convenient pretreatment methods for UF–RO membrane filtration. Guo et al. [7] observed a significant reduction in flux decline when flocculation and adsorption were applied as pretreatments in crossflow microfiltration. Abdessemed et al. [8] found that using aluminum sulphate and ferric chloride as coagulants, flocculation–adsorption processes could remove 86% of chemical oxygen demand (COD) from domestic wastewater, along with a considerable reduction in turbidity. Guo et al. [9] reported that pretreatment with flocculation and adsorption could effectively remove phosphorus and organic substances. Kim et al. [10] investigated the influence of particle characteristics and contaminant loading, on coagulation in membrane filtration during wastewater treatment for reuse [10]. Another long-term study performed using a submerged membrane adsorption process, indicated that powdered activated carbon adsorption helped to significantly reduce membrane fouling [11,12].

As a high-performance, cost-effective and chemical-free approach, biological aerated filters (BAFs) have also been used as a pretreatment method for membrane filtration processes [13]. By using bio-carriers to add additional substrate surface area for the attachment of microorganisms, BAF has been proven to improve the removal of nutrients and DOM (composed of humic, carbohydrates and proteins substances, etc.), the main substances associated with membrane fouling.

Most studies on the production of demineralized water from SE have focused on the effects of specific pretreatment unit or technology on UF–RO processes, in terms of membrane fouling and permeate flux, such as in coagulation and activated carbon adsorption. Few long-term, comprehensive studies have been performed, to evaluate the transformations of various pollutants within combined water treatment processes, especially in biological processes (i.e., BAF) followed by UF–RO. To the best of our knowledge, this is the first study to describe a practical biological process application (i.e., BAF) for the production of demineralized water from SE in power plants.

Additionally, analysis of variation in composition was performed, including natural organic matter, ammonia, heavy metals and so on, during each stage of the treatment process. This data provide additional insights into process selection, mechanical comprehension and the optimum range of operation parameters in the pretreatment process. The objectives of this research were to (1) investigate the effects of several combined pretreatments prior to RO membrane processes, for the treatment of SE from a domestic wastewater treatment plant; (2) evaluate the degradation/conversion characteristics of various constituents, especially bio-refractory organics, during each separate treatment stage in an integrated industrial water treatment plant. These stages include BAF, coagulation (with $FeCl_3$ as coagulant), sand filtration, UF, RO and ion exchange.

2. Materials and methods

2.1. Treatment process

The power plant receives SE from the city of Zhanjiang, Guangdong (South China), which operates an anaerobic-anoxic-oxic process at a flow rate of 340 t/h. The characteristics of the secondary-treated municipal wastewater are outlined in Table 1.

Table 1
Characteristics of the influent secondary effluent system

Parameter	Range	Parameter	Range
COD (mg/L)	20–55	Ca^{2+} (mg/L)	22.54–44.75
BOD (mg/L)	11–15	Mg^{2+} (mg/L)	7.82–31.95
TN (mg/L)	20–45	K^+ (mg/L)	12.05–29.12
NO_3^- (mg/L)	<0.1–20	Na^+ (mg/L)	66.58–298.16
NO_2^-(mg/L)	<0.004–22.539	Fe^{3+} (mg/L)	0.08–1.78
NH_3–N (mg/L)	10–20	Fe^{2+} (mg/L)	0.01–0.65
TOC (mg/L)	9–13	Total Fe (mg/L)	0.12–1.88
UV_{254} (cm^{-1})	0.16–0.22	F^-(mg/L)	0.14–0.92
Al^{3+} (mg/L)	<0.02–20.00	Free CO_2	8.84–36.15
Ba^{2+} (mg/L)	<0.02–0.04	Hardness (total)	111.46–285.54
Sr^{2+} (mg/L)	<0.06	Carbonate hardness	40.07–128.73
Cu^{2+}(mg/L)	0.11–0.41	Non-carbonate hardness	35.62–202.33
Mn (mg/L)	0.03–0.25	SO_4^{2-} (mg/L)	46.36–128.7
SiO_2 (mg/L)	5.96–16.08	HCO_3^- (mg/L)	48.86–156.98
$TSiO_2$ (mg/L)	8.71–23.28	Cl^- (mg/L)	112.68–526.55
H_2SiO_3 (mg/L)	7.75–20.90		

A full-scale treatment process was assessed for the production of demineralized water, consisting of CN/DN (nitrification/denitrification) BAF, pre-oxidation, coagulation with $FeCl_3$ as the coagulant, sedimentation, sand filtration, disinfection and UF–RO in series. CN/DN BAF was used to remove nutrients and achieve preliminary oxidation, followed by chemical oxidation using chlorine (NaClO), prior to coagulation. CN/DN BAF processes are comprised of aerobic and anoxic stages, with a corresponding hydraulic retention time of 1 h, respectively. Dissolved oxygen (DO) concentrations in the aerobic stage were maintained at 3–5 mg/L, while anoxic tanks contained low speed mixers to keep the mixed liquid suspended solids in suspension.

Coagulation with 10 mg/L iron salt ($FeCl_3$) was used prior to sedimentation, destabilizing and removing most of the suspended solids and colloids. After coagulation, the effluent was filtered through a sand filter bed operated in up-flow mode, then disinfected using NaClO. Finally, the treated water was subjected to UF and RO membrane filtration, with the RO effluent finally treated by cation and anion resins operated in series. This system successfully achieved the required level of purity for demineralized ultra-pure water production (Table 1). The waste water generated from the backwashing (cleaning) of membranes and sand filters, as well as the regeneration of ion exchangers, was also recycled to the inlet of BAF, eventually obtaining zero liquid discharge.

Water samples were collected from each stage to evaluate the performances of individual units of operation. A schematic description of the treatment processes is shown in Fig. 1.

2.2. Analytical procedures

2.2.1. Analysis methods

Analysis methods for effluent characterization, such as COD, total nitrogen (TN), total phosphorus (TP) and so on, followed the standard methods [14]. Ammonia concentration of the samples was obtained according to the Chinese NEPA standard method [15].

DOC values of water samples were established following filtering through a 0.45 μm membrane (Tianjing Jinteng Experiment Equipment Co., Ltd., China), after every individual treatment unit, then measured with the TOC analyzer (Elementar Co., Germany). UV_{254} of the samples were determined using a DR5000 UV/VIS spectrophotometer (HACH Co., USA). $SUVA_{254}$ was established as the UV_{254} divided by COD. Turbidity values of water samples were analyzed using a turbidity meter (HACH-2100P, USA). Conductivity and pH were measured using a multi-parameter Hanna HI 255 instrument (UK). The pH of every water sample remained within the limits of 7.6 (±0.5).

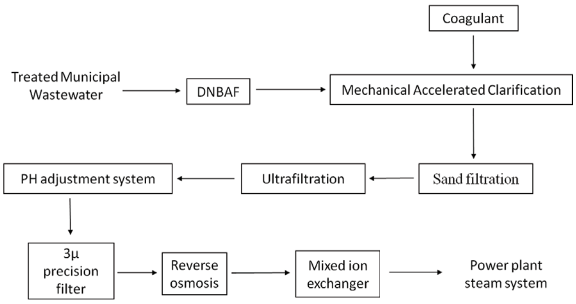

Fig. 1. Secondary-treated municipal wastewater treatment process.

Metal concentrations were determined by inductively coupled plasma mass spectroscopy (7500CX ICP-MS, Agilent, USA) and atomic absorption spectroscopy (PinAAcle 900T, PerkinElmer, USA). Anion species were analyzed by ion chromatography (Dionex IonPacTM AS9-HC). DO and pH were detected by DO meter (Hach-LDO™, USA) and pH meter (Hach-sens ION$^+$ pH 1, USA), respectively.

2.2.2. EEM analysis

Spectroscopic techniques can provide information on the source and composition of DOM present in a system [16]. Hence, three-dimensional EEM fluorescence spectroscopy was used to characterize the behavior of DOM compounds throughout the whole system, with UV-Vis absorbance and fluorescence spectroscopy.

Three-dimensional excitation–emission-matrix spectra (3D EEM) of samples were obtained using a Hitachi F-7000 fluorescence spectrometer (Hitachi Inc., Japan), equipped with a 150 W Xenon lamp at a PMT voltage of 400 V (Ex = 5 nm and Em = 5 nm for grating slit, respectively). Emission spectra were scanned from 280 to 550 nm at 2 nm intervals, while excitation spectra were scanned from 200 to 450 nm at 5 nm intervals. EEM analysis was conducted at a scan rate of 1,200 nm/min and peak Ex/Em pairs were analyzed using Hitachi FL software [17]. All samples were analyzed in duplicate.

3. Results and discussion

The degradation characteristics of secondary effluent treated from domestic wastewater by the combined processes were studied for a 3-month period.

3.1. Nitrogen and phosphate removal performance

As we know, nutrients (nitrogen and phosphorus) induce the growth of bacteria, enhancing corrosion and scaling problems in biofilter systems. Phosphate can precipitate with divalent and trivalent cations, while ammonia is a strong complexing agent for copper and iron. Therefore, the common metal materials used in steam systems, are at significant risk of serious corrosion and scaling problems. Therefore, it is necessary to remove these nutrients before recycled effluents are applied to steam production.

Figs. 2 and 3 show the variation in nitrogen, phosphate and COD concentrations in the influent and effluent, throughout the experimental period. As shown, nutrients and organic compounds were both effectively reduced in the CN/DN BAF biological process, under aerobic and anaerobic conditions in series. Ammonia and total nitrogen were the dominant species to be biologically degraded, with removal efficiencies of 99.7% and 97.5%, respectively, while nitrite concentrations were constantly very low. Moreover, phosphate was also removed by the CN/DN BAF process, with

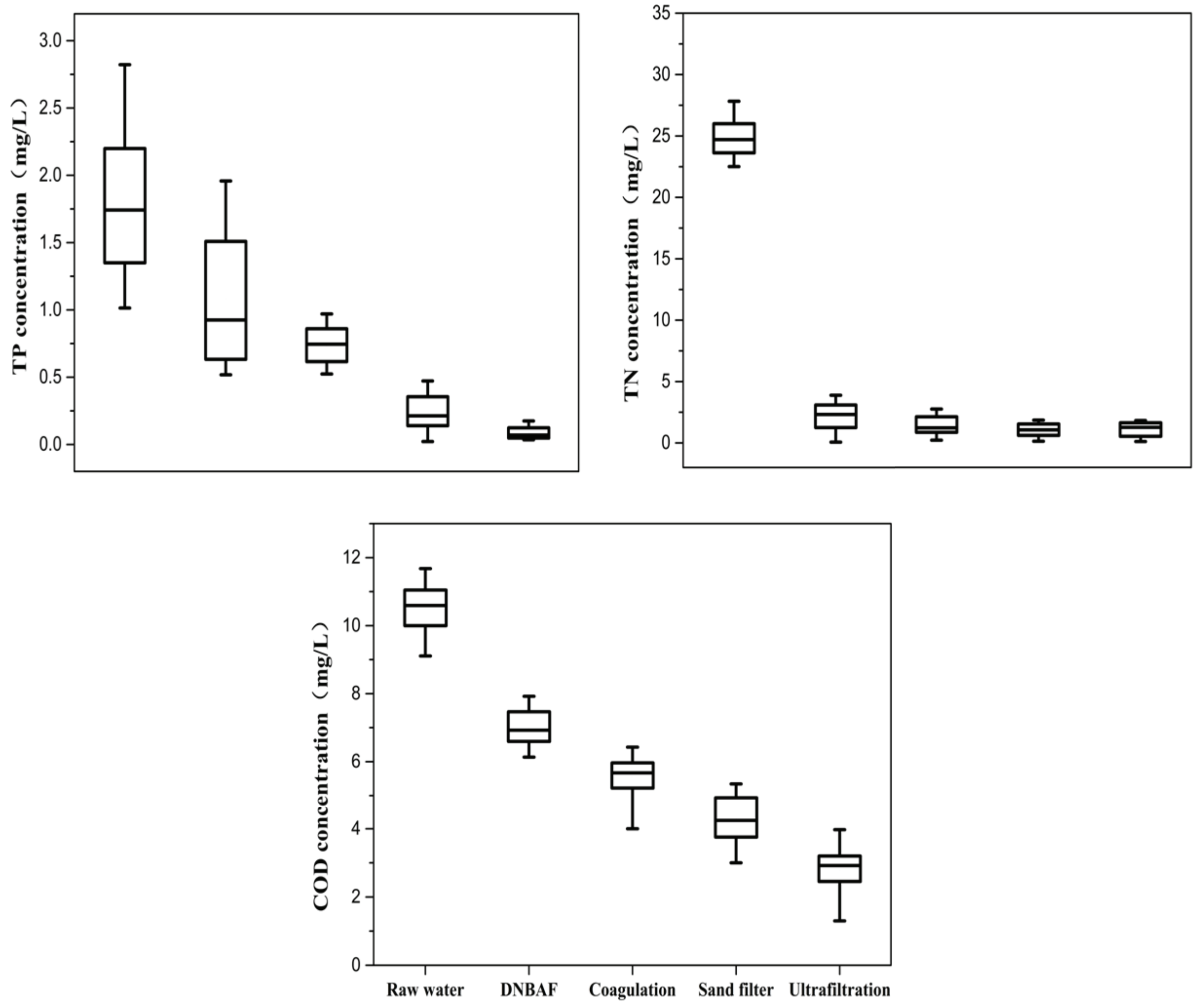

Fig. 2. TN, TP and COD concentrations of each stage.

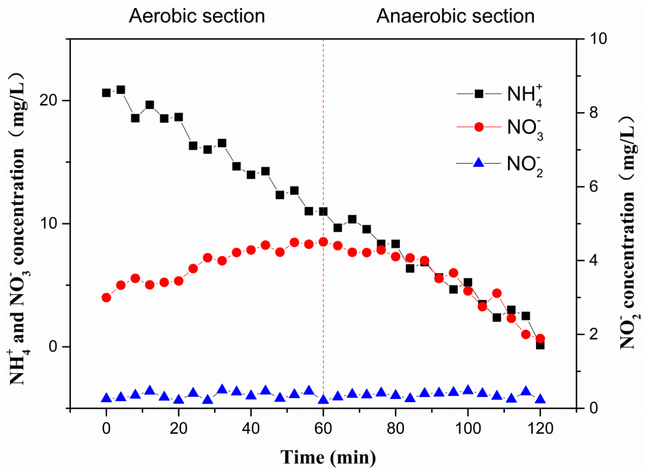

Fig. 3. Nitrogen concentrations of each stage.

approximately 71.4% reduction observed during the BAF process and a further reduction to 89% following coagulation and UF processes. The average effluent concentration of TN, nitrate nitrogen (NO_3^-–N), ammonia nitrogen (NH_4^+–N) and TP were 1.005–1.235 mg/L, 0.521–0.723 mg/L, 0.056–0.153 mg/L and 0.043–0.175 mg/L, respectively, which were significantly lower than the corresponding influent levels.

To further elucidate the COD and nutrient removal mechanisms of the CN/DN BAF system, additional CN/DN experiments were performed in batch flow mode. These experiments evaluated the transformation of COD, different forms of nitrogen and phosphorus, during the aerobic and anoxic phases.

In the first aerobic phase, ammonia was nearly completely nitrified to nitrate, and phosphorus and organic substance were also removed to some extent, accounting for a 63.41%, 71.4% and 39.3% reduction in ammonia, phosphorus and total COD, respectively, while nitrate remained stable at approximately 2 mg/L.

In the subsequent anoxic phase, denitrification occurred, with nitrate denitrified to nitrite with the consumption of COD, resulting in the accumulation of NO_2^-–N during the first 30 min. In the following 30-min period, nitrite was gradually eliminated. The simultaneous consumption of COD and elimination of nitrogen indicates that the denitrifying bacteria present were heterotrophic.

3.2. Changes of TSS and VSS

Fig. 4 shows the residual concentration and relative removal efficiencies for total suspended solids (TSS) and volatile suspended solids (VSS) in each process. In contrast with their relatively low removal efficiencies in BAF alone, significant reduction of TSS and VSS were observed following coagulation, sand filtration and UF processes. In particular, in sand filter and UF processes, more than 91.7% of TSS and 53.6% of VSS can be removed by coagulation and sand filtration processes, respectively.

Coagulation processes removed TSS more efficiently than VSS, while sand filtration removed VSS more efficiently. A possible explanation of this phenomenon is that flocs and colloids formed from organic matter were small and compact, therefore could not be removed efficiently by coagulation as compared with sand filtration. It is of note, that VSS did not change obviously during the BAF process, potentially due to microbial growth and metabolism of BAF products, which will be discussed in the following section.

3.3. Metal elements

Six cation metal elements (K, Na, Ca, Mg and Fe) were measured by ICP-AES in each stage of water treatment processes, as shown in Table 1. A high concentration of dissolved monovalent cation elements (K: 12.05–29.12 mg/L; Na: 66.58–298.16 mg/L) were present throughout the whole experimental period, remaining almost constant during BAF, coagulation, sand filter and UF processes. Calcium and magnesium cations were co-precipitated during the coagulation stage, with further removal by the following sand filtration and UF stages. The specific situation is shown as Fig. 5.

It is of note, that the concentrations of Fe^{2+} and Fe^{3+} increased by 22.03% and 66.67%, due to the addition of $FeCl_3$ as a coagulant and the formation of dissolved metal complexes as part of the coagulation process. As expected, most of Fe^{2+} (20%) and Fe^{3+} (30%) species were removed by sand filtration and UF stages as shown in Fig. 6, with these results supported in agreement with a study by Chaturvedi and Dave [18].

3.4. Non-metal elements

The concentration of inorganic ions (Cl^-, SO_4^{2-}, HCO_3^-, F^-, B^-) were also measured during the whole experimental period. As shown in Table 1, Cl^- and SO_4^{2-} were the main non-metal components identified in SE, in the range of 112.68–526.55 mg/L and 46.36–128.7 mg/L, respectively. Fig. 7 shows levels of Cl^- and SO_4^{2-} remained stable during the BAF, coagulation, sand filtration and UF processes. Bicarbonate (HCO_3^-) concentrations were also very high, although they were reduced by around 25% during the clarification process.

3.5. Hardness and silicates

Hardness-causing ions and silicate concentrations are also main components in mineral deposition (scaling). Silicates form adherent deposits by precipitating anions (HCO_3^-, CO_3^{2-}) bound to divalent and trivalent cations (Ca^{2+}, Mg^{2+}), onto the surfaces of turbine nozzles and blades. In addition, silicates are strong complexing agents for copper and iron, which will eventually result in reduced turbine capacity, altering steam velocities and causing reduction in pressure.

Both hardness-causing ions and silicate concentrations are persistent and difficult to reduce using BAF. During coagulation, calcium and magnesium are co-precipitated with PAC flocs, with silica components partly removed by absorption or entrapment into larger PAC flocs. Only around 30% of hardness and 30% of silicate removal were obtained during the clarification process as shown in Fig. 8, due to the low efficiency of coagulation processes for hardness-causing ions and silicate concentrations reduction. It is of note that the remaining silicate fraction was further reduced during the following UF and RO process.

3.6. Transformation of DOC and SUVA

DOM (composed of carbohydrates, proteins, humic substances, etc.) often causes membrane fouling and is considered one of the most essential substances for effective treatment system management. DOM removal by the different treatment processes compared in the present study was measured in terms of DOC, UV_{254} and SUVA, with findings shown in Table 2.

Although DOC concentrations did not show any obvious change during BAF processes, the concentrations decreased sharply in the following sand filtration and UF processes. It is noted that the DOC removal efficiency for the four stages were 10.3%, 7%, 32% and 42%, respectively, potentially due to the microorganisms and metabolites discharged from the BAF process.

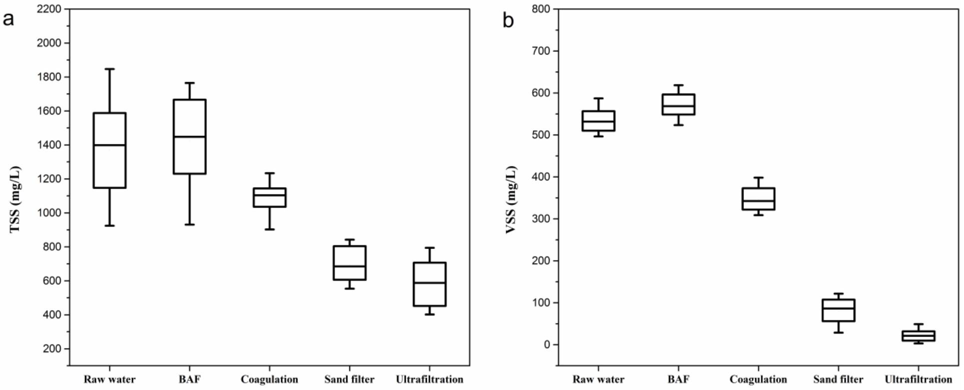

Fig. 4. Residual concentration and relative removal efficiencies for (a) TSS and (b) VSS.

The UV_{254} value often represents organic matter containing C=O and C=C structures, such as phenols, polyaromatic hydrocarbons, aromatic ketones and aromatic aldehydes, among others. SUVA represents the level of aromaticity of DOM. It is found that up to 58.2% UV_{254} removal efficiency was achieved after the coagulation stage, showing that a significant quantity of aromatic organics containing C=C and C=O structural groups, can be removed by combined BAF and coagulation processes. The level of UV_{254} removal was further increased to 78.3% and 89.4% using combined sand filtration and UF processes.

Table 2
Transformation of DOC and SUVA

Stage	DOC		UV_{254}		SUVA (m^{-1} L/mg)
	DOC (mg/L)	Rate (%)	UV_{254} (m^{-1})	Rate (%)	
Raw water	8.42	0	0.195	0	0.023
BAF	7.55	10.3	0.139	28.7	0.018
Coagulation	7.02	6.3	0.082	29.2	0.012
Sand filter	4.77	26.7	0.042	20.5	0.009
Ultrafiltration	2.77	23.8	0.02	11.3	0.007

3.7. Transformation of EEM

The Ex/Em wavelengths of 200–250 nm/280–330 nm and 200–250 nm/330–380 nm, represent simple aromatic proteins, such as tyrosine, tryptophan and phenylalanine; fulvic acid-like Ex/Em is in the range of 200–250 nm/380–550 nm; soluble microbial products (SMPs) Ex/Em are in the range of 250–280 nm/280–380 nm; humic acid Ex/Em is in the range of 250–400 nm/380–550 nm [19,20].

The color bar (on the right side) in Fig. 9 indicates the different fluorescence intensity of DOM. As shown, aromatic proteins such as tryptophan proteins and fulvic acid constituted the main component of the raw water (SE), with Ex/Em wavelengths of 200–250 nm/330–380 nm and 200–250 nm/380–550 nm [21]. Although fluorescence intensity was gradually reduced during the treatment process, the major peak positions in raw water (SE) were almost the same as following coagulation, sand filtration and ultrafiltration. This suggests that aromatic proteins and tryptophan proteins were the dominant recalcitrant substances [22]. Moreover, after the coagulation stage a significant decrease in fluorescence intensity was observed for tryptophan proteins, humic substances and SMP, with approximately 38%, 22% and 29% eliminated, respectively. These results indicate that the combination of BAF and coagulation processes could effectively remove aromatic, humic substances and tryptophan proteins.

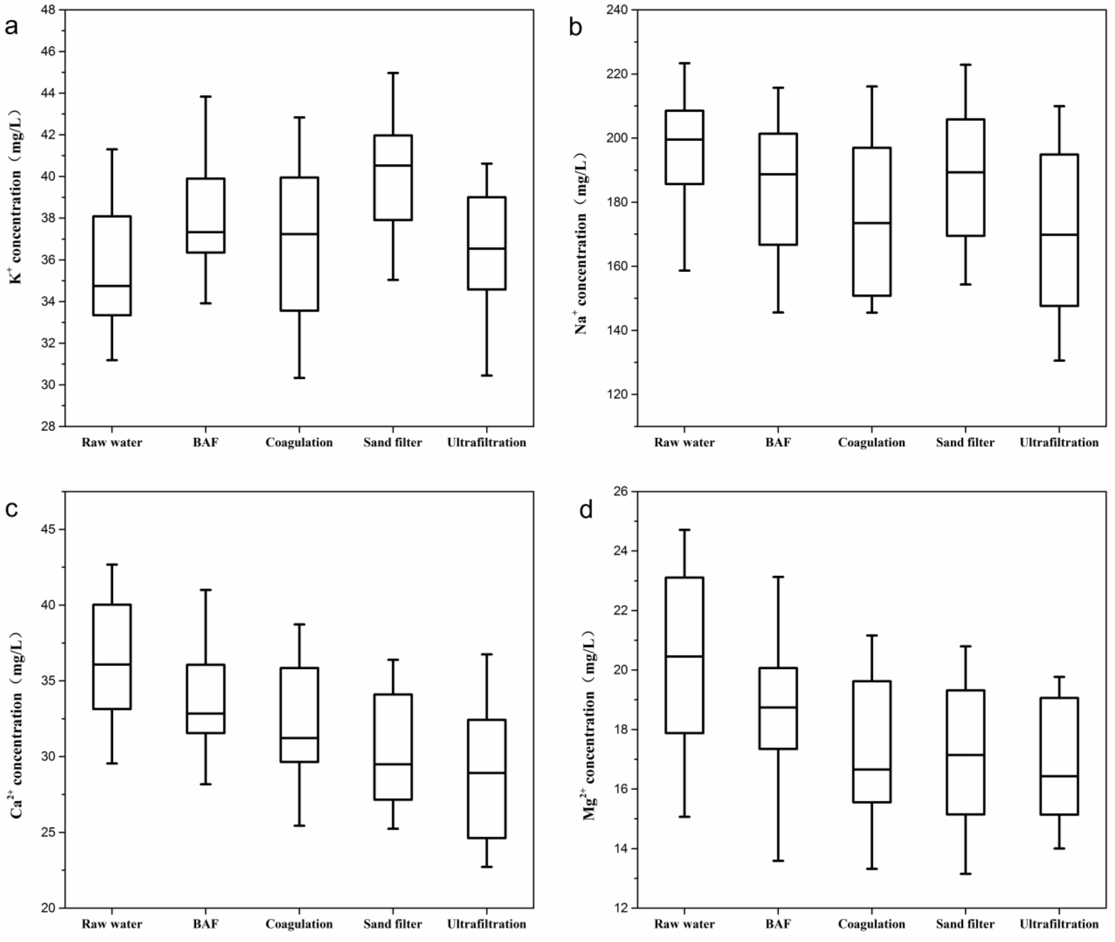

Fig. 5. Metal elements concentrations of each stage. (a) K^+; (b) Na^+; (c) Ca^{2+} and (d) Mg^{2+}.

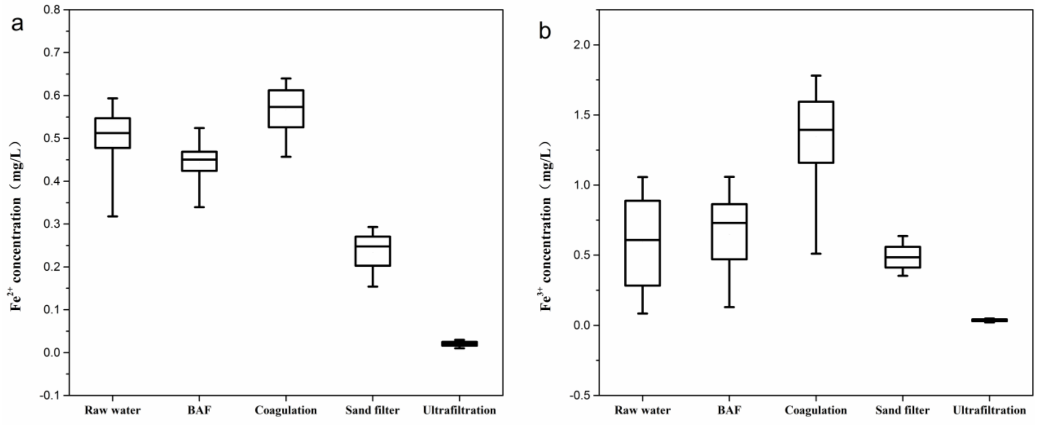

Fig. 6. Metal elements concentrations of each stage. (a) Fe^{2+} and (b) Fe^{3+}.

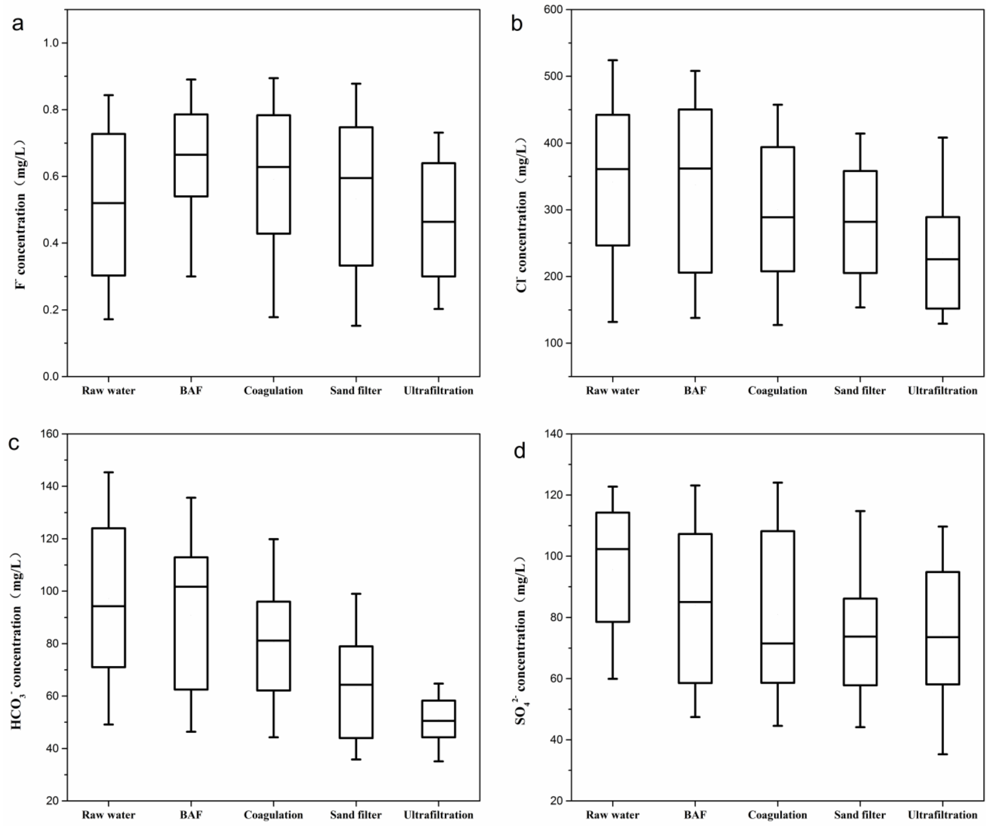

Fig. 7. Non-metal elements concentrations of each stage. (a) F^-; (b) Cl^-; (c) HCO_3^- and (d) SO_4^{2-}.

In comparison, following sand filtration and UF stages, low removal efficiencies (less than 10%) were observed for humic substances and SMPs. This suggests that these DOM were mainly of a low molecular weight, passing sand filtration and UF membranes easily.

4. Conclusions

In this study, a combination of treatment processes was assessed for the production of ultra-pure demineralized water in thermal power stations. BAF, coagulation, sand filtration and UF performances were compared, with the highest nitrification and denitrification performance achieved using CN/DN BAF processes. Hardness-causing ions such as calcium and magnesium, and silicates were co-precipitated to some extent during the coagulation stage, with further removal by the following sand filtration and UF stages. Although BAF could not completely remove DOC, TSS and VSS, the combination of BAF and coagulation significantly reduced concentrations of nutrients and DOM (composed of carbohydrates, proteins, etc.), allowing effective control of membrane fouling.

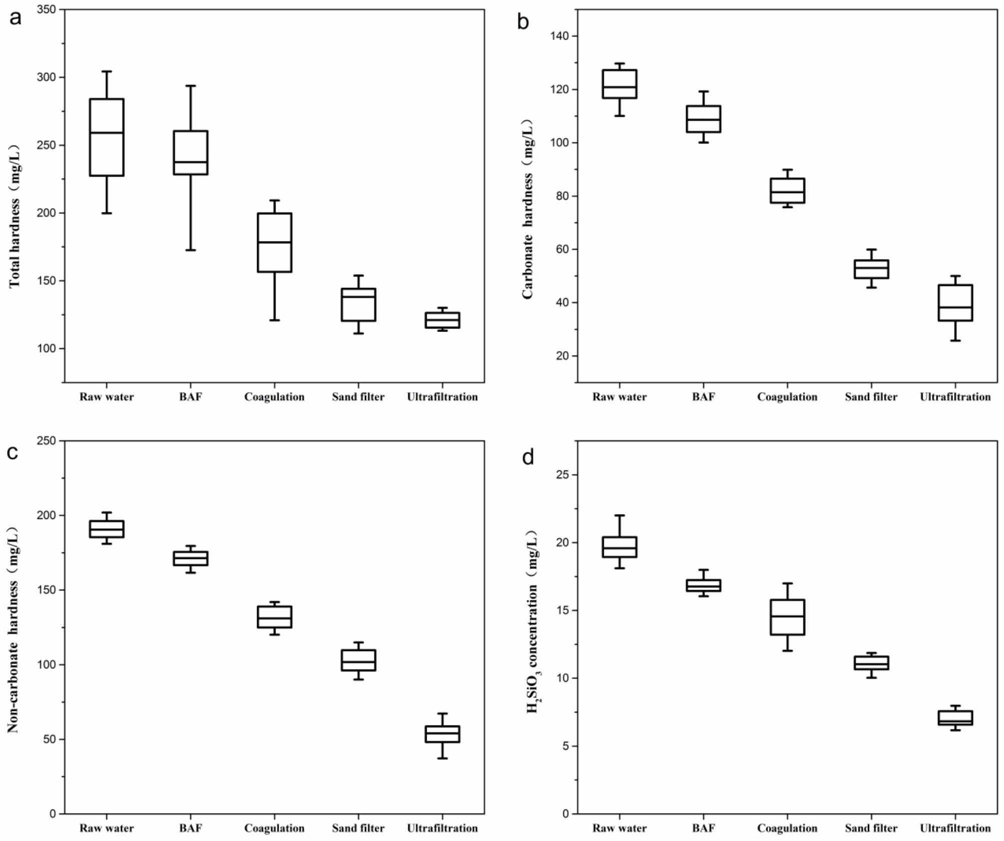

Fig. 8. Hardness and silicates. (a) Total hardness; (b) carbonate hardness; (c) non-carbonate hardness and (d) H_2SiO_3.

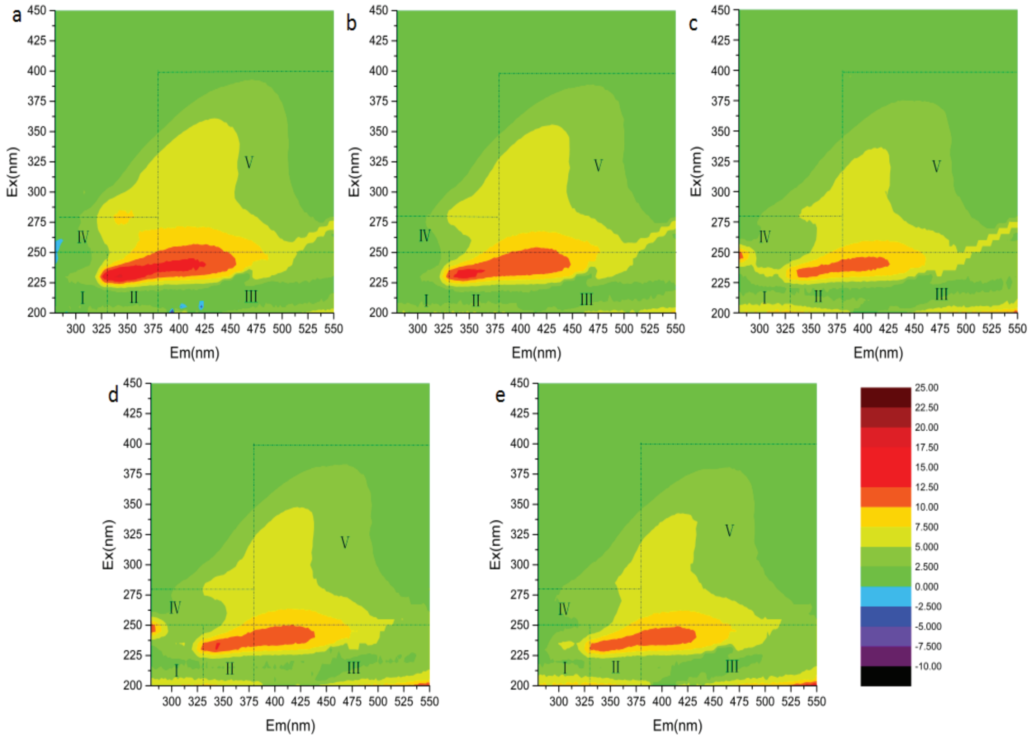

Fig. 9. Three-dimensional fluorescence analysis of (a) raw, (b) BAF, (c) coagulation, (d) sand filtration and (e) ultrafiltration processes.

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