Application of an integrated dissolved ozone flotation process in centralised fracturing wastewater treatment plant

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ABSTRACT

To solve the problems of unstable chemical oxygen demand (COD), turbidity and suspended solid (SS) removal for the electrocatalytic process and unstable operation of the subsequent ultrafiltration membrane–reverse osmosis membrane in a centralised fracturing wastewater treatment plant in Inner Mongolia, the integrated dissolved ozone flotation (DOF) process was proposed to replace the original electrocatalytic process. Multiple processes, such as ozonation, flotation, coagulation and decolourisation, can be achieved in one integrated DOF reactor. The results showed that the removal efficiency of COD, colour, turbidity and SS in the DOF process could reach 25.4, 49.9, 95 and 96%, respectively. Meanwhile, the treatment cost was reduced by 47% (i.e., 1.77 RMB/m^3 for the DOF process) compared with the electrocatalytic process.

Key words | advanced treatment, dissolved ozone flotation, fracturing wastewater, reconstruction project

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HIGHLIGHTS

- Multiple physio-chemical processes can be achieved in one integrated DOF reactor.
- The DOF exhibited higher removal efficiency compared with the electrocatalytic process.
- The DOF has stable removal efficiency during drastical influent quality variation.
- The treatment cost was reduced by 47% compared with the electrocatalytic process.

GRAPHICAL ABSTRACT
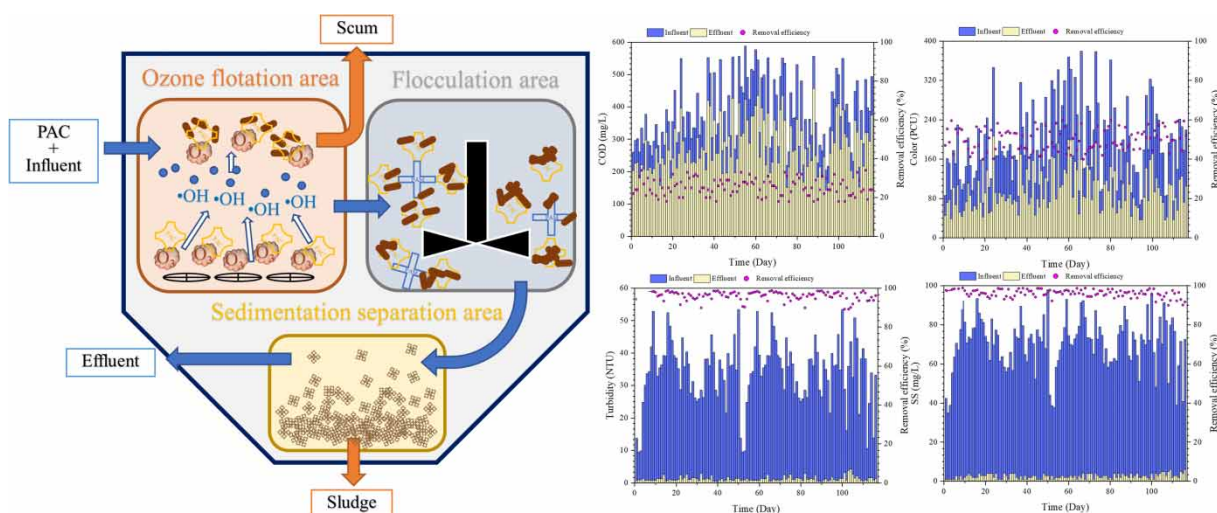
INTRODUCTION

The fracturing wastewater (FWW) comes mainly from the fracturing fluid that flows back to the surface from the well-bore during the fracturing process (Shih *et al.* 2015). Hydraulic fracturing is a technique used to extract oil or natural gas from impermeable host rocks, which involves horizontal drilling into rock formations and injection of high-pressure fracturing fluid (Williams *et al.* 2017; Schultz *et al.* 2020). In the hydraulic fracturing process, up to 24,500 m^3 of water-based fluid can be injected into a single well, and 70% of the fracturing fluid is returned to the surface for harmless disposal (Wang *et al.* 2018a). FWW is a mixture of a variety of compositions, including fracturing fluid additives, as well as geological inorganic and organic substances and their conversion products (Barati & Liang 2014; Luek & Gonsior 2017). FWW has the characteristics of high salt, high organic matter and high viscosity (Yost *et al.* 2016; Stringfellow *et al.* 2017).

In the FWW treatment area, no single treatment process can meet the requirements for stable effluent quality, and two or more combined processes can ensure stable effluent characteristics (Fakhru'l-Razi *et al.* 2009). These independent processes that can be combined include physical, chemical, and biological treatments. Extensive research has shown that: (1) the physical treatment method has the problems of high initial investment and sensitivity to the influent flow; (2) chemical treatment has the problems of high operating cost and disposal of waste sludge; and (3) the biological treatment is sensitive to influent organic matter and salt concentration (Gordalla *et al.* 2013; Olsson *et al.* 2013; Lester *et al.* 2015). Choosing the most suitable FWW treatment technology will ultimately depend on the pollutant characteristics and treatment volume of the single processing unit (Estrada & Bhamidimarri 2016).

A centralised fracturing wastewater treatment plant (CFWTP) is located in Ordos, Inner Mongolia, with an FWW treatment capacity of 5,000 m^3/d. The CFWTP applies microelectrolysis, Fenton oxidation, coagulation and chemical precipitation for the pre-treatment of FWW to initially reduce the chemical oxygen demand (COD) and Ca^{2+} and Mg^{2+} concentrations while increasing the bio-degradability of the effluent (Wang *et al.* 2006; He *et al.* 2015; Zhang 2017). The effluent from the pre-treatment stage enters the biological anaerobic–anoxic–oxic (AAO) process for treatment. This stage further reduces the COD in the water while reducing the concentrations of total nitrogen and total phosphorus (Ding *et al.* 2016; Zhang *et al.* 2016). Because FWW has the characteristics of high salt, high organic matter and high viscosity, the effluent from the

biological treatment cannot meet the reuse water standard (GB/T18920-2002), which can be used as a compound drilling fluid and for miscellaneous municipal purposes (Lira-Barragán *et al.* 2016). If the AAO effluent can be used for miscellaneous municipal purposes, it must pass through an ultrafiltration membrane and a reverse osmosis membrane (Qurie *et al.* 2013). Therefore, advanced treatment is necessary for the AAO effluent to meet the requirements of reclaimed water.

The existing advanced treatment process for this CFWTP is an electrocatalytic system (ECS) and dual-media filtration. However, due to the large number of chloride ions in the raw water during the actual operation, the chloride ions in the water were oxidised to chlorine gas under the action of the electric current, and the chlorine gas further reacted with water to form hypochlorous acid and hydrochloric acid (Zodi *et al.* 2009; Gao *et al.* 2010). This reaction not only causes a waste of electric energy but also generates oxidising substances that need to be neutralised by adding reducing substances; otherwise, the presence of these miscellaneous will cause irreversible pollution in the subsequent processing system (Dixon *et al.* 2013; Jin *et al.* 2019a). At the same time, partially generated chlorine gas emissions to the air would also threaten the health of on-site operators (Clark *et al.* 2013). To solve the above problems and maintain favourable dissolved organic matter removal efficiency, the proposed integrated DOF process was applied in this study (Jin *et al.* 2006). In this process, ozonation and flocculation are carried out simultaneously during the air flotation process, and the flocs are attached to the microbubbles discharged with the scum (Jin *et al.* 2015). Our previous study indicated that the metal coagulants in the integrated DOF process can act as catalysts to promote the generation of hydroxyl radicals (Jin *et al.* 2016).

The DOF process has been applied in many fields such as urban wastewater treatment plants, printing and dyeing wastewater, milk industry wastewater and cosmetic wastewater (Lee *et al.* 2008; Bogacki *et al.* 2017; Wang *et al.* 2018b; Pereira *et al.* 2020). Nevertheless, there are few reports on the application of advanced FWW treatment. Therefore, it is necessary to carry out long-term experimental research on the application of the DOF process to FWW advanced treatment to evaluate the feasibility of the process.

The aim of this study was to investigate the removal performance of the DOF process under long-term operating conditions. In addition, a comparison between the DOF and the original ECS process was conducted to better understand the advantage of the DOF process in terms of removal performance and cost.

MATERIALS AND METHODS

Raw water quality

The DOF reactor was fed with the effluent from the sedimentation tank in a centralised fracturing wastewater treatment plant (CFWTP) in Inner Mongolia, China. The CFWTP mainly treats pre-treated FWW with a biological anaerobic–anoxic–oxic (AAO) treatment process. The main operating parameters of the oxic section are as follows: total hardness = 469.6 ± 187.3 mg/L, total alkalinity = 770.1 ± 386.5 mg/L, total phosphorus = 5.8 ± 1.4 mg/L, total nitrogen = 21.1 ± 6.8 mg/L, SV30 = $48.5 \pm 19.5\%$, SVI = $23.0 \pm 10\%$ and sludge concentration = $19,600 \pm 4,600$ mg/L. The raw water possessed the following characteristics: COD = 396.3 ± 192.7 mg/L, colour = 223.8 ± 155.8 PCU, turbidity = 31.45 ± 22.99 NTU, SS = 67.80 ± 32.72 and pH = 7.89 ± 0.16.

DOF experimental set-up

Figure 1 presents the experimental set-up of the DOF reactor, which contains four main parts: the water inlet system, DOF unit, dosing system and ozone generation system. The DOF unit is composed of two cylindrical reaction vessels, which are independent of each other. A single reactor is divided into three portions, consisting of inner, middle and outer cylinders. The outer cylinder is the ozone flotation area, the middle cylinder is the flocculation area, and the inner cylinder is the sedimentation separation region. The inner diameter of a single reactor column is 3,400 mm, the total height of the main body is 7,000 mm, and the effective volume is 50 m^3.

Table 1 shows the operating parameters of the DOF reactor. Treatment capacity refers to the total influent to the DOF unit. The influent of a single cylindrical reaction
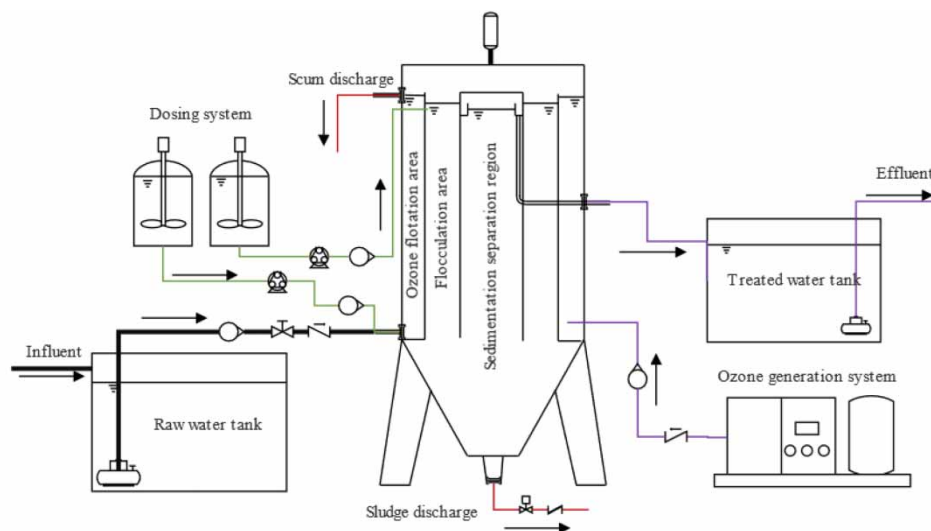
Figure 1 │ Experimental set-up of the DOF reactor.

Table 1 │ DOF process operating parameters

Project	Value
Treatment capacity	50 m³/h
PAC dosage	550 mg/L
PAM dosage	2.5 mg/L
Ozone concentration	35 mg O_3/L$_{liquid}$
Total hydraulic retention time	120 min

vessel is 25 m³/h, and the remaining items refer to the operating parameters of a single cylindrical reaction vessel.

The raw water is pumped into the DOF reactor, mixed with the poly-aluminium chloride (PAC) in the pipeline mixer and enters the outer cylinder area. Ozone, PAC and water pollutants are in full contact and mixed in this region. On the one hand, organic matter reacts under the combined action of ozone and PAC; on the other hand, the microbubbles generated during the aeration process adsorb suspended pollutants to float on the liquid surface in the form of scum (Jin *et al.* 2016). The scum formed after ozone flotation is removed by the slag scraper, then the water flows to the middle cylinder area through the overflow port, and polyacrylamide (PAM) is added to this area. In the middle cylinder area, the stirring device installed on the top of the processing unit performs slow stirring to make the water fully mixed, and the flocs produced by flocculation enter the inner cylinder area with the water flow. In the inner cylinder area, the sludge-water separation efficiency is improved by the inclined plate, and the

clean water is discharged through the outlet weir. The sludge is collected at the bottom of the tank and regularly discharged out of the tank.

Analytical methods

This study measures the COD, colour, turbidity and SS of the water. To avoid the interference caused by the high sodium chloride salinity in the secondary effluent, COD adopts the low-range multiple dilution method in the 'Determination of Water Quality Chemical Oxygen Demand (HJ828-2017)'. Turbidity is measured by a turbidity meter (HACH-2100N, Shanghai, China), and colour and SS are measured by a water quality analyser (HACH-DR900, Qingdao, China). The three-dimensional fluorescence was analysed using a fluorescence spectrophotometer (FP-6500, Jasco, Japan); the three-dimensional fluorescence spectrum was measured at an excitation wavelength of 200–400 nm and an emission wavelength of 280–500 nm.

Data analysis

In this study, two software programmes, origin (OriginPro 2019b) and SPSS (IBM SPSS Statistics 26), were used for data analysis. Between these programmes, Origin is mainly used to analyse the long-term operation data of the DOF-integrated reactor, and the Mann–Whitney tests in SPSS

are used to comparatively analyse the treatment effects between DOF and ECS. The Mann–Whitney test is used to test whether two independent samples originate from the same population, that is, to judge whether the mean of two populations is significantly different (Mikocka-Walus & Andrews 2016; Ye & Ahammed 2020).

For the Mann–Whitney test, the data are recoded according to the process: $1 = $ DOF process and $2 = $ ECS process. The new sample is called 'Group'. The COD, colour, turbidity and SS removal efficiency for processes 1 and 2 were used as dependent variables and analysed together with the independent variable 'Group'. SPSS itself generates the hypothesis. Null hypothesis, H_0: The distribution of COD (or colour, turbidity and SS) removal efficiency is the same across categories of Group. Alternative hypothesis H_A: The distribution of COD (or colour, turbidity and SS) removal efficiency is not the same across categories of Group.

RESULTS AND DISCUSSION

Organic matter removal performance of the DOF process

COD and colour removal performance

The COD variation in the influent and effluent of the DOF reactor is shown in Figure 2(a). The influent COD of the

DOF reactor is 204–588 mg/L, and the treated water COD is 143–457 mg/L. The average COD removal efficiency is 25.4%, and the highest removal efficiency can reach 34%. The influent COD of the DOF reactor varies drastically. An explanation for this variation might be that the raw water of the CFWTP comes from different gas wells as well as different fracturing operation stages. FWW quantity and composition changes significantly in the early stages (e.g., high flow, high COD and low TDS) to the later stages (e.g., low flow, low COD and high TDS from the second week) (Hayes 2009; Slutz et al. 2012). Furthermore, during AAO treatment, a high TDS concentration can hinder biological activity and affect COD effluent, so that the quality of the biotreated water is not stable (Lester et al. 2014).

Another important finding is that the removal efficiency of the DOF reactor remains relatively stable when the influent COD changes. This finding also agrees with our previous observations, which showed that the organic matter removal performance remained constant when the ozone dosage was greater than 0.8 mg/L (Jin et al. 2006; Jin et al. 2015). This consistent phenomenon could be attributed to the interactions between ozone and coagulants. Due to the addition of metal coagulants, ozone reacts with the hydroxyl groups on the surface of the hydrolysed metal coagulants in the aqueous solution through electrostatic adsorption and hydrogen bonding force (Jin et al. 2018). Studies have found that the carboxyl group on the surface of the metal
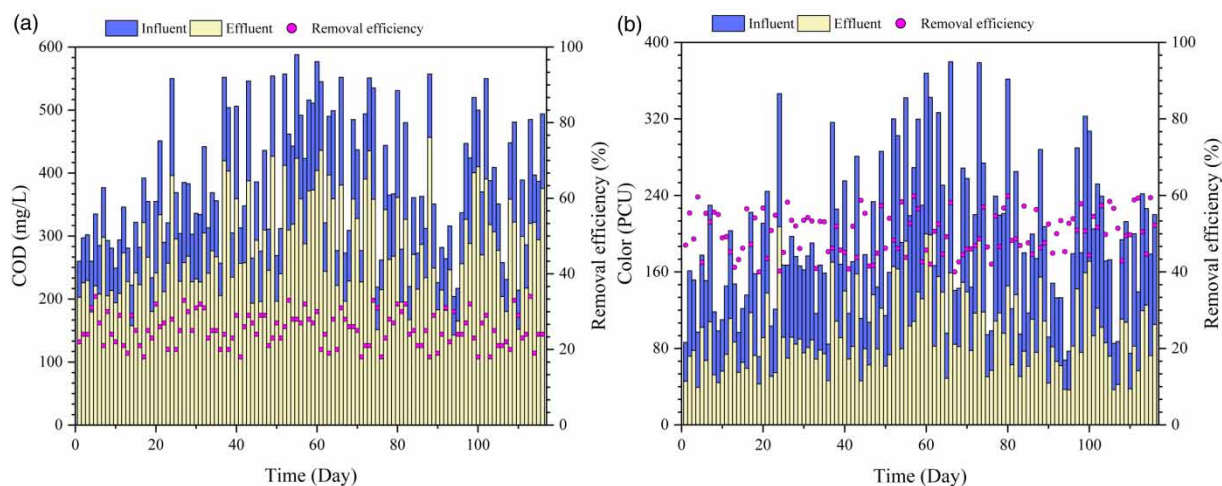

Figure 2 | (a) The removal of COD by the DOF device. (b) The removal of colour by the DOF device.

catalyst will cause the decomposition of ozone to form $\cdot O_2H$, $\cdot O_3H$ and $\cdot O_4H$ and then generate $\cdot OH$ through a chain reaction (Jin *et al.* 2017). In this study, aluminium-based coagulants will form a large number of hydroxyl functional groups on the surface through hydration (Song *et al.* 2019). In addition to the direct reaction of ozone with OH^- in the water to initiate a chain reaction to generate $\cdot OH$, the hydroxyl groups on the surface of the coagulant can also react with ozone to initiate a chain reaction and produce a large amount of $\cdot OH$ (Jin *et al.* 2019b). The simultaneous existence of the above two effects makes the DOF reactor more efficient for organic matter removal.

Figure 2(b) shows the colour removal performance of the DOF process, which shows that the influent colour of the DOF reactor is between 68 and 379 times, with an average of 191 times. The removal of colour by the device is relatively stable. The average removal efficiency is 49.9%, and the highest removal efficiency can reach 59.8%. In

accordance with the present results, previous studies have demonstrated that coagulation and ozonation have decolourising effects on wastewater (Collivignarelli *et al.* 2019). Several reports have shown that coagulation can make the wastewater colour removal efficiency reach more than 40% through three processes of coagulation, flocculation and sedimentation (Malik 2004; Ahmad *et al.* 2006; Zonoozi *et al.* 2008; Erkanlı *et al.* 2017). Ozonation has been reported to be able to destroy chromogenic and auxochrome groups such as azo, carbon–carbon double bonds and benzene rings in water to achieve decolourisation (Zhou *et al.* 2013).

Fluorescence characteristics of dissolved effluent organic matter

Figure 3(a) presents the three-dimensional fluorescence spectrum of dissolved organic matter in the biochemical effluent, i.e., the influent of the DOF process. Figure 3(b)–3(d)
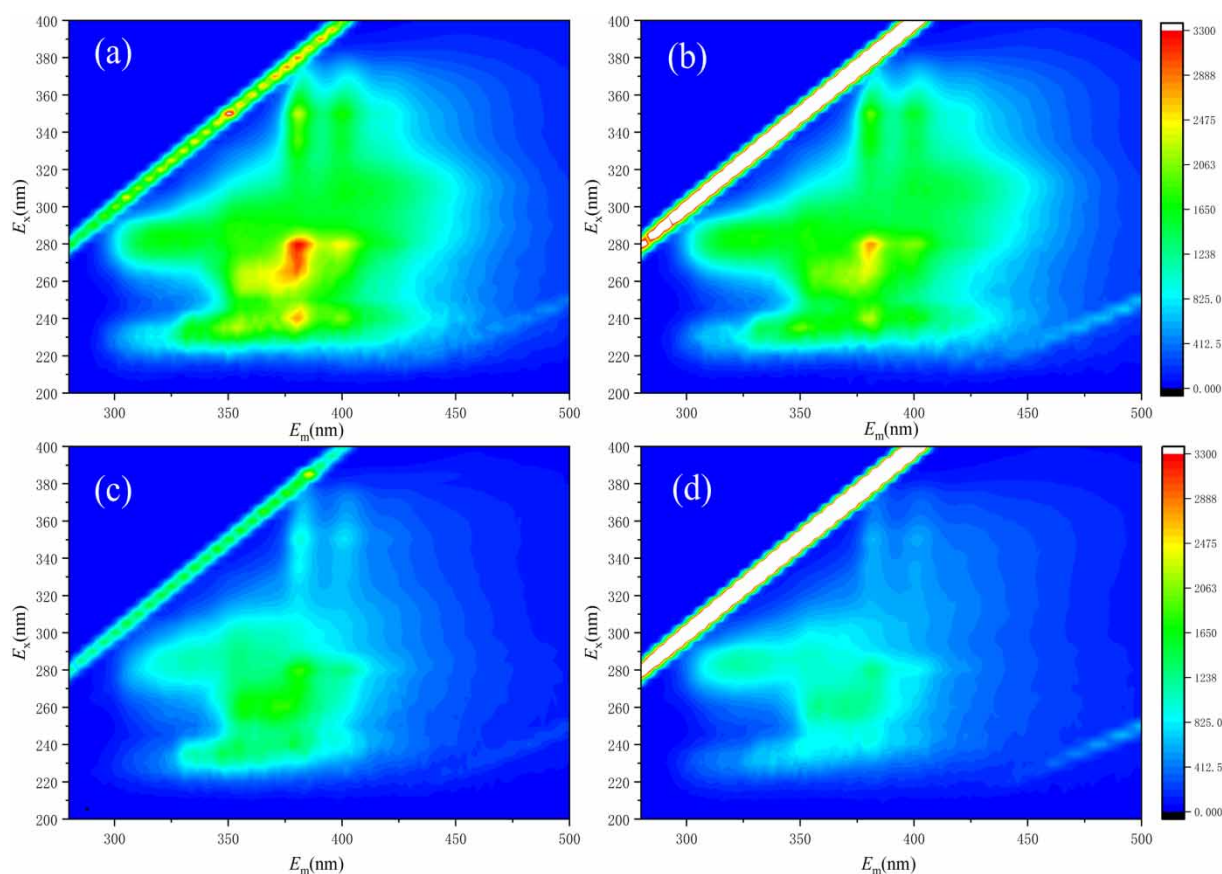

Figure 3 | Three-dimensional fluorescence spectra of (a) raw water and effluent at ozone dosages of (b) 15 mg/L, (c) 25 mg/L and (d) 35 mg/L.

presents the three-dimensional fluorescence spectra of the dissolved organic matter in the effluent of the DOF process at ozone dosages of 15, 25 and 35 mg/L.

There are three main fluorescence peaks in Figure 3(a): the fluorescence peaks of proteins or phenols containing tyrosine aromatic amino acids (emission wavelength 300–340 nm, excitation wavelength 260–290 nm), fulvic acid-like fluorescence peaks of amino acids (emission wavelength 370–440 nm, excitation wavelength 240–270 nm) and fulvic acid-like fluorescence peaks (emission wavelength 370–450 nm, excitation wavelength 310–360 nm) (Komatsu et al. 2008). The soluble organic matter in the biochemical effluent is attributed mainly to proteins and fulvic acid, which is consistent with the results of Wang et al. (2017). Figure 3(b)–3(d) presents the three-dimensional fluorescence spectra of the effluent with the addition of ozone at 15, 25 and 35 mg/L, respectively. Figure 3 shows that with an increasing ozone concentration, the fluorescence intensity of these two types of substances obviously decreased, and the fluorescence peak of fulvic acid substances exhibited a more significant decrease. Ozone can react with functional groups such as unsaturated carbon–carbon double bonds and aromatic rings of these two types of substances to further oxidise large-molecular-weight organics into small-molecular-weight organics (Lucas et al. 2010). As mentioned in the previous research, the molecular weight of organic matter drops from 2,000–6,000 to 2,000–3,000 Da after ozonation (Jin et al. 2015).

Suspended pollutant removal performance in the DOF process

Figure 4(a) shows the turbidity removal performance of the DOF process. Figure 4(a) shows that the turbidity of the influent water fluctuates in the range of 10–54 NTU, and the quality of the influent water changes drastically. The turbidity of the effluent of the device is stable within the range of 0–5 NTU. Nevertheless, the effluent of the DOF process can remain stable regardless of the variation in the raw water quality. The turbidity removal efficiency is above 98%, and the average removal efficiency can reach 95%, indicating that the device exhibited excellent turbidity removal ability and can adapt to the variation in water quality.

Figure 4(b) shows the suspended solid (SS) removal performance of the DOF process. Figure 4(b) shows that the influent SS of the DOF reactor fluctuates in the range of 35–100 mg/L. However, the effluent SS of the DOF reactor is distributed between 1 and 6 mg/L, and in 50% of the water samples during the operation period, SS cannot be detected, indicating the excellent removal performance for SS. The removal efficiency for SS by the DOF reactor fluctuates in the range of 90–99%, and the average removal efficiency is 96%, which can meet the requirements of water reclamation such as drilling fluid preparation.
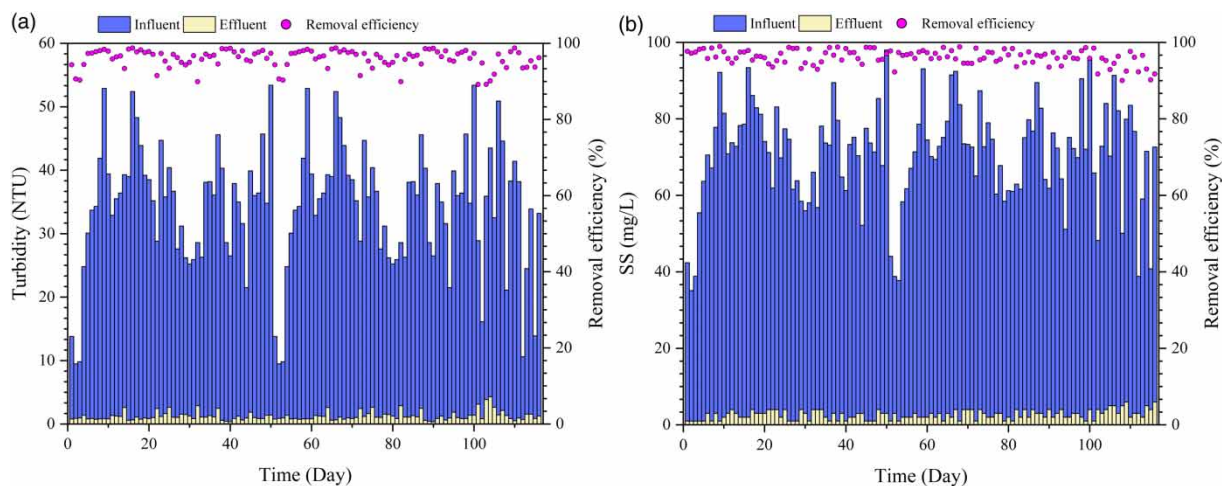

Figure 4 | (a) The removal of turbidity by the DOF device. (b) The removal of SS by the DOF device.

Comparison between the DOF and ECS processes

Removal performance

Table 2 shows the SPSS outputs for the Mann–Whitney tests. Table 2 shows that the significance level is less than 0.05 ($p = 0.000$ for COD, colour, turbidity and SS). Therefore, the test results are statistically significant. The decisions from the hypothesis testing were that the null hypotheses were rejected for all four cases. Therefore, the distribution of the above four detection indicators in the effluent of the DOF and ECS processes is significantly different, showing that the advanced treatment effluent of CFWTP has a significant impact due to changes in the process.

Figure 5 shows the distribution of COD, colour, turbidity and SS removal efficiency in different processes in the Mann–Whitney test. Figure 5(a) shows that the COD removal efficiency distribution of the DOF process is more concentrated, and the removal efficiency of the ECS process is scattered. This inconsistency can be attributed to variable influent quality and electrochemical oxidation characteristics in the ECS process. The removal efficiency has been reported to depend on the water quality of the treated wastewater when the current density is constant (Chen 2004; Anglada et al. 2009). Since equipment can only apply a constant current density during operation, the COD removal efficiency of the ECS process varies greatly. In addition, Figure 5(b) shows that the removal efficiency for colour in the ECS process is also unstable. Figure 5(c) and 5(d) show the distribution

of turbidity and SS removal efficiency in the two processes. Figure 5(c) and 5(d) show that the turbidity and SS removal efficiency of the DOF process is above 90%, while the removal efficiency of these two parameters in the ECS process is distributed between 20 and 80%, showing that the DOF process exhibits a significant advantage in suspended pollutant removal performance. In the ECS process, due to the high conductivity of the influent water and the characteristics of the ECS process itself, the sacrificial electrodes made of iron and aluminium will quickly dissolve (Mollah et al. 2001, 2004). So, consumable metal plates such as iron or aluminium are not used as sacrificial electrodes. Therefore, the ECS process has hardly removal ability for SSs. Therefore, the DOF process replaced the ECS process, and the removal efficiency of all indicators exhibited a significant increase.

Economic evaluation

The present tests were designed to determine the difference in operating costs between the DOF and the ECS processes.

The operating cost of the DOF process applied to the DFWTP secondary effluent (without considering labour costs and equipment depreciation costs) includes the following main parts: flocculant agent (i.e., PAC) cost, polymer coagulant aid (i.e., PAM) cost and power consumption. The operating cost of the original ECS process includes the following main parts: reducing agent (i.e., sodium bisulfite) cost and power consumption. The unit price of PAC solid is 2,000 RMB/t, the unit price

Table 2 | Hypothesis testing summary of the Mann–Whitney test

Null hypothesis	Test	Significance	Decision
The distribution of COD removal efficiency is the same across categories of Groups.	Independent-samples Mann–Whitney U test	0.00	Reject the null hypothesis
The distribution of colour removal efficiency is the same across categories of Groups.	Independent-samples Mann–Whitney U test	0.00	Reject the null hypothesis
The distribution of turbidity removal efficiency is the same across categories of Groups.	Independent-samples Mann–Whitney U test	0.00	Reject the null hypothesis
The distribution of SS removal efficiency is the same across categories of Groups.	Independent-samples Mann–Whitney U test	0.00	Reject the null hypothesis

Notes: Asymptotic significance is displayed. The significance level is 0.050.
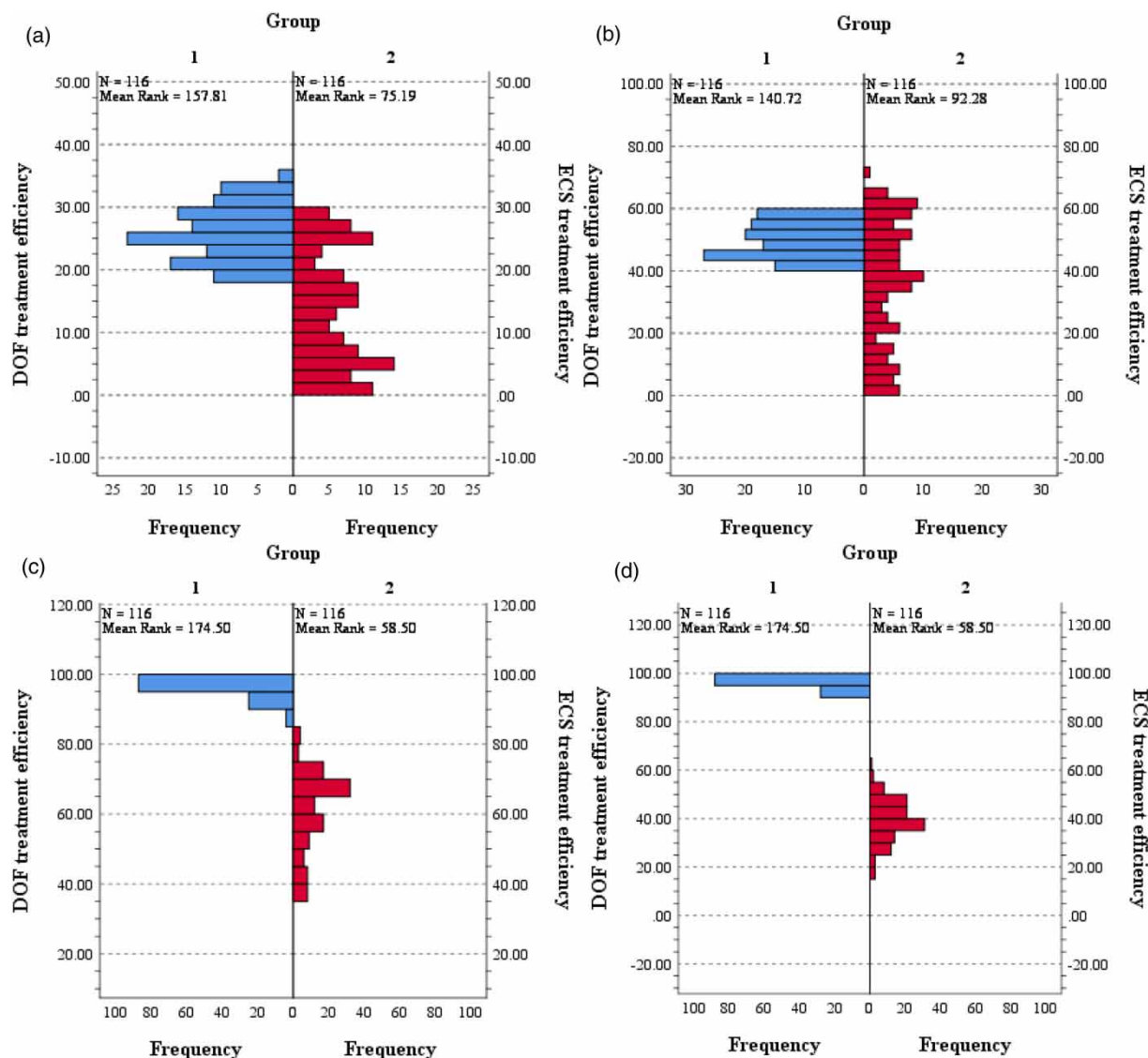
Figure 5 | Independent-samples, Mann–Whitney *U* test of DOF and ECS (a) COD, (b) colour, (c) turbidity and (d) SS removal efficiency.

of PAM solid is 7,900 RMB/t, the unit price of sodium bisulfite solid is 2,400 RMB/t, the electricity price is calculated as 0.45 RMB/kW·h, and the sludge treatment fee is 0.01 RMB/m^3. When the treatment capacity is 50 m^3/h, the corresponding treatment costs for ozone flotation and electrocatalysis are 1.77 and 3.34 RMB/m^3, respectively. The specific calculation results are shown in Table 3.

Table 3 | DOF and ECS process operating cost calculation

Project	PAC dosage (g/m^3)	PAM dosage (g/m^3)	NaHSO$_3$ dosage (g/m^3)	Pharmacy fee (RMB)	Electricity bill (RMB)	Sludge treatment fee (RMB)	Total price (RMB)
DOF	550	2.5	–	1.22	0.55	0.01	1.78
ECS	–	–	780	1.87	1.47	–	3.34

The reduced operating cost of the DOF process comes from the following two aspects. First, compared with the ECS process, the energy consumption of the DOF process exhibited a significant decrease. At the same time, because the DOF process does not produce Cl_2 and HClO, the DOF process will not cause equipment corrosion and personnel health threats. Second, since HClO is not produced, there is no need to add reducing agents before the membrane treatment process is entered. Improvements in the above two aspects led to an improvement in the working environment of workers, removal performance and treatment economy.

CONCLUSIONS

To solve the problems of unstable COD, turbidity and SS removal for electrocatalytic processes with unstable operation of the subsequent ultrafiltration membrane–reverse osmosis membrane in CFWTPs, the study indicated that the DOF process exhibited favourable COD, colour, turbidity and SS removal efficiency, and the removal efficiency reached 25.4, 49.9, 95 and 96%, respectively. Meanwhile, the DOF process effluent can meet the influent requirements of the subsequent dual membrane system. The treatment cost was apparently reduced by 47% (i.e., 1.77 RMB/m^3 for the DOF process) compared with the original ECS process. This work offers valuable insights into the application of the DOF process in the enhanced treatment of the CFWTP secondary effluent.

CONFLICT OF INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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