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ORIGINAL PAPER

Pretreatment of high strength waste emulsions by combined vibratory shear enhanced process with Fenton oxidation

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Abstract Pretreatment of waste emulsions with high organic content by a combined process of vibratory shear enhanced process and Fenton's oxidation prior to biological treatment was investigated. Vibrating membrane had shown good performance in chemical oxygen demand and oil removals and the mitigation of concentration polarization. However, the permeate after filtration processing still contained high content of organics. Thus, additional Fenton oxidation was applied to reduce the organic loading, and improve the biodegradability of the wastewater. The optimal molar ratio of ferrous iron to hydrogen peroxide was 0.05 obtained from the jar-test experiments. Removal of organics was enhanced by increasing hydrogen peroxide dosage, while efficiency of hydrogen peroxide reached maximum of 1.11(w/w) at the hydrogen peroxide dosage of 6.8 g/L. Furthermore, the biological experiments indicated that the high concentration of organics could inhibit microbial activity, which decreased the chemical oxygen demand degradation rates. The adaptive period of the microbe was greatly shortened using Fenton's reagent at the low dosages. The improvement of the biodegradability could be explained by partial mineralization and chemical transformation of parent organic compounds after Fenton oxidation.

Keywords Aerobic batch bioassay · Hydroxyl radical · Industrial wastewater · Integrated process · Ultrafiltration

Introduction

Waste emulsions with high organic content cause serious contamination and rancid odor due to the presence of complex chemicals, biocides, etc., hence, their treatment and final disposal must be handled carefully (Anderson et al. 2003). Traditional separation processes such as gravity, flotation and skimming are not so efficient to remove oils from water since oils have been chemically emulsified (Karakulski et al. 1995). Ultrafiltration (UF) is one of the most promising technologies for oil removal, thanks to the advantages of chemical additive-free and high oil rejection (Karakulski et al. 1995). However, the permeate flux of traditional membrane separation decreases rapidly with time due to membrane fouling and concentration polarization. Therefore, various physicochemical methods have been used to maintain a stable permeate flux, such as adding reactive salt (Belkacem et al. 1995), coagulation pretreatment (Zhong et al. 2003) and dynamic shear enhanced processes, i.e., rotating membranes and vibratory shear enhancing process (VSEP) (Jaffrin 2008). VSEP can efficiently prevent concentration polarization and membrane fouling in the process of emulsions filtration by producing high frequency of shear. Consequently, a high flux can be achieved at low feeding pressure, resulting in

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low energy consumption but high treatment capacity (Al Akoum et al. 2002; Moulai-Mostefa et al. 2007, 2010; Jaffrin 2008).

Besides, it is commonly observed that permeate after UF contains high content of organics mainly including surfactants and biocides, which requires an additional biological treatment for the permeate. Whereas, high concentration of surfactants and biocides could cause a series of physiological responses and dysfunction of microbes and in their cells. For example, once the concentration of surfactant in wastewater exceeds 1,000 mg/L, microbial activity will be inhibited seriously and foaming can occur in aeration tank (Aloui et al. 2009), which can significantly influence the removal efficiency of biological treatment. Thus, a chemical oxidation step is normally required to reduce organic loading and toxicity of wastewater prior to biological processing (Kitis et al. 1999; Pignatello et al. 2006). Fenton oxidation has been proven to be an effective way to reduce organic loading (Lin et al. 1999) and improve biodegradability of industrial wastewater containing biorefractory compounds, such as surfactants (Kitis et al. 1999), dyes (Gharbani et al. 2008) and pharmaceuticals (Deegan et al. 2011). This technology depends on in situ producing a non-selective strong hydroxyl radical which can oxidize various organic matters at ambient condition (Gogate and Pandit 2004).

This study proposed an integrated process of VSEP, Fenton oxidation and biological technology to treat the waste emulsions with high organic content. The objective of this study was to investigate: (1) the effect and performance of VSEP in oil removal and mitigation of concentration polarization; (2) the optimal operating conditions were determined by considering both H_2O_2 efficiency and biodegradability improvement in Fenton process; (3) influence of initial concentration of organic matters and Fenton oxidation on biodegradability of permeate using aerobic batch bioassays; (4) the feasibility and effects of using the integrated process for treating waste emulsion in full-scale application. The laboratory-scale experiment was carried out in School of Resources and Environmental Science, East China Normal University (Shanghai, China), from April to October 2010. The full-scale treatment facility was operated stably for 8 months (June 2011–April 2012) at the Shanghai Chemical Industry Park.

Materials and methods

Waste emulsions

The three oil-in-water (O/W) emulsions used in this study were obtained from an appliance manufacturer and an electronic accessory producer and metal working corporation. The characteristics of the emulsions are presented in Table 3.

Emulsions treatment

Filtration of emulsions by VSEP

The filtration module is depicted in Fig. 1. The shaft supporting the membrane module serves as the function of a torsion spring, which transmits oscillations created by an eccentric driving motor. The displacement amplitude and oscillation frequency of the equipment were fixed at 2 cm and 50 Hz, respectively. The permeate from the membrane filtration passes through the permeate tubing which locates upon the spring under atmospheric pressure. The concentrated stream permeates through the “out” line. The return flow passes through the flow limiter and the control valve allowing the fine adjustment of outlet pressure. A “Y-trap” and bypass valve for adjusting the inlet pressure is equipped at the inlet and outlet of pump, respectively. Meanwhile, the transmembrane pressure (TMP) is estimated as the mean value of the inlet and outlet pressure (Moulai-Mostefa et al. 2010), and the inlet and outlet pressures were kept constant at 344.8 and 125 kPa. Membrane flux is measured in accordance with the volume of fluid within a certain period. Each run is stopped after reaching 80 % permeate recovery. The operating conditions of VSEP are given in Table 2. Membrane is made of hydrophilic polyethersulfone with 150 kDa nominal cut-off, and the effective area is 1.54 m^2. The detailed features of the membrane can be found in Table 1. Molecular weights of Dextran T2000 and T500 are 2,000 and 500 kDa.

Fenton’s oxidation of VSEP permeate

The pH of permeate was adjusted to 3.0 before the Fenton oxidation experiments. 500 mL of permeate was added in an 800-mL beaker. The beaker was placed in thermostatic bath (25 ± 1 °C). Then, appropriate amounts of ferrous sulfate heptahydrate salt ($FeSO_4 \cdot 7H_2O$) were added in the beaker to achieve desired concentration of ferrous ions. Finally, an aliquot of H_2O_2 (30 % (w/v)) was spiked in the solution under vigorous stirring using a magnetic stirrer. The reaction was stopped by raising the pH of the solution to 12, after 3-h stirring, and then the mixture was boiled for 10 min to remove H_2O_2 prior to chemical oxygen demand (COD) analysis. Note that boiling process does not affect the COD determination. The suspension was poured into a graduated cylinder to allow the precipitate settles down until lower than 20 % of the total volume. The supernatant was collected from the cylinder. The COD values of the supernatant and precipitate were determined.

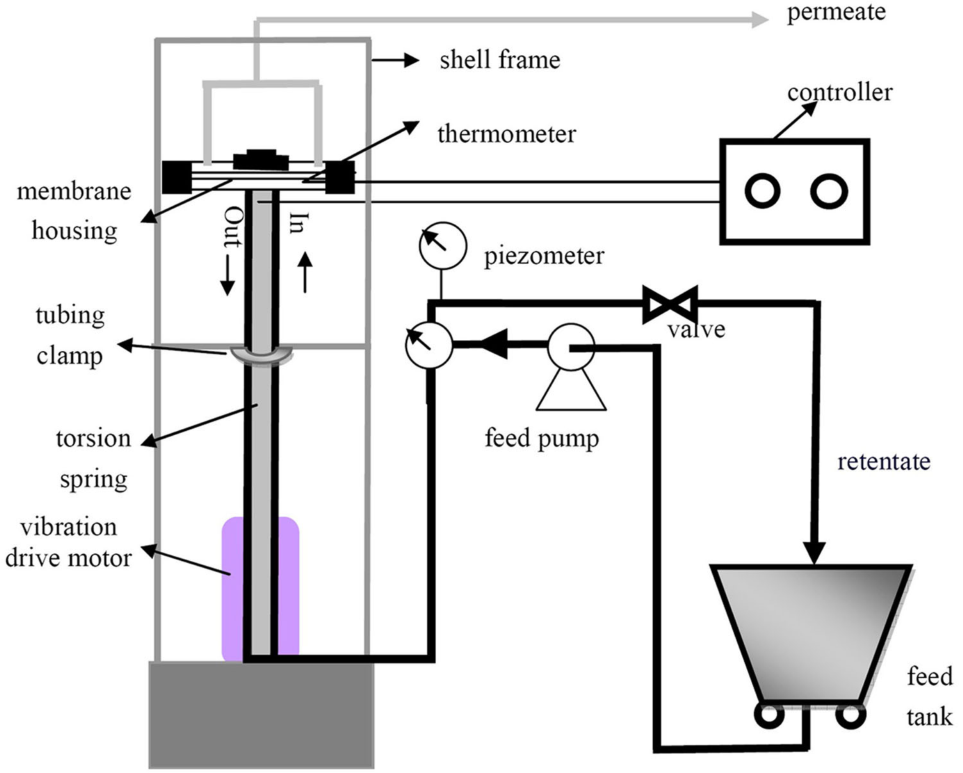

Fig. 1 A schematic presentation of the vibrating membrane equipment

Table 1 Technical data of UF-19 membrane (70 kPa, 20 °C, stirred cell: 700 rpm)

Test solute rejection (%)		Pure water flux (L m^{-2} h^{-1})	
Dextran T2000 (1 %)	Dextran T500 (1 %)	70 kPa	300 kPa
87–92	50–60	300–600	1,200–2,100

Biodegradability assessment

Aerobic batch bioassay was adopted to assess the biodegradability of permeate. This approach has been described in details elsewhere (Adams et al. 1997; Kitis et al. 1999). Briefly, the bioassays were conducted by adding 300 mL of sample into a 500-mL of Erlenmeyer flask. The water sample was incubated with a microbial seed from aeration tank in Quyang wastewater treatment plant, Shanghai), after spiking an aliquot of nutrient solution. In order to examine the influence of concentration of organics on the microbial activity, permeate with COD concentrations of 1,000, 5,000 and 10,000 mg/L were examined. In addition, batch bioassays were also conducted on the permeate with/without Fenton's oxidation at an initial COD concentration of 1,000 mg/L. The supernatant was filtrated through 0.45 μm membrane filter before COD analysis. Sample was considered biodegradable when the measured COD in solution decreased over 70 % after biological treatment.

Water quality analysis

Hydrogen peroxide was measured using iodometric method. Oil concentrations in the feed and permeate solutions were analyzed using gravimetric method after extracting with petroleum ether according to the National Standard of China CJ/T51-2004, while COD was measured using the potassium dichromate method based on the National Standard of China GB 119 14-89. Content of suspended solid (SS) and pH was also determined by gravimetric and electrometric methods, respectively.

Results and discussion

Waste emulsions treatment by VSEP

Effects of emulsions treatment by vibrating membrane

The effects of VSEP on removal of COD, oil, and SS were presented in Table 2. Due to the different sources and composition of water sample, the removal efficiency of COD and oil was different. But apparently, SS was completely rejected. The recovery of permeate in each water sample was more than 80 %. The range of hydrophilic–lipophilic balance (HBL) values of emulsifiers used in O/W emulsion is from 8 to 18. This indicated that the adhesive force between water and surfactant was stronger than that

Table 2 Experimental results of treatment of three waste emulsions by VSEP

Sample	pH	COD (mg/L)		Oil (mg/L)		SS (mg/L)	
		Feed	Permeate	Feed	Permeate	Feed	Permeate
A	7.3	31,100	4,840	2,280	335	2,075	0
B	8.0	117,100	24,820	876	92	5,490	0
C	9.2	235,300	32,600	12,296	54	0	0

between oil and surfactants. Thus, the surfactants and additives with low molecular weight could pass through membrane instead of being rejected along with oil. As a result, the UF permeate still had a high COD level.

Membrane flux

The classical resistance-in-series model can be written in our case as (Belkacem et al. 1995):

$$J = \frac{\Delta P}{\mu(R_{\mathrm{M}} + R_{\mathrm{ads}} + R_{\mathrm{P}})} \quad (1)$$

where J denotes membrane flux, ΔP is transmembrane pressure, μ is permeate viscosity. R_{M}, R_{ads} and R_{P} represent intrinsic membrane resistance, adsorption resistance of solute molecules and concentration polarization resistance, respectively. As shown in Eq. 1, the decrease of permeate flux was mainly ascribed to the formation and accumulation of oil and O/W gel layers on the membrane surface. Furthermore, adsorption of surfactants resulted in reduction of surface porosity of membrane (Lin and Lan 1998; Marchese et al. 2000; Benito et al. 2001; Janknecht et al. 2004).

Figure 2 demonstrated the changes of membrane flux and temperature of the samples A and C during filtration. Because of the frictional heating contributed by high-speed shear force of vibrating membrane, the increase of the temperature of each sample was almost linear to time. The flux of the sample A stably maintained in the range of 47–52 L m^{-2} h^{-1}, while the flux of the sample C increased significantly from 23 to 37 L m^{-2} h^{-1} during filtration processing. These results demonstrated that the VSEP could effectively reduce the concentration polarization of the membrane via high shear rates. However, Moulai-Mostefa et al. (2007), (2010) reported that the membrane flux decreased with time due to the increase of oil concentration at constant temperature and pressure. These inconsistent findings regarding flux, from our results, could be explained by the increase of the sample temperature. It is well known that the higher temperature can decrease viscosity of water, and consequently, increase the permeability through membrane. The increased temperature could counteract the flux decline caused by the increase of oil concentration. Therefore, the membrane flux can be effectively enhanced by increasing the temperature of wastewater.

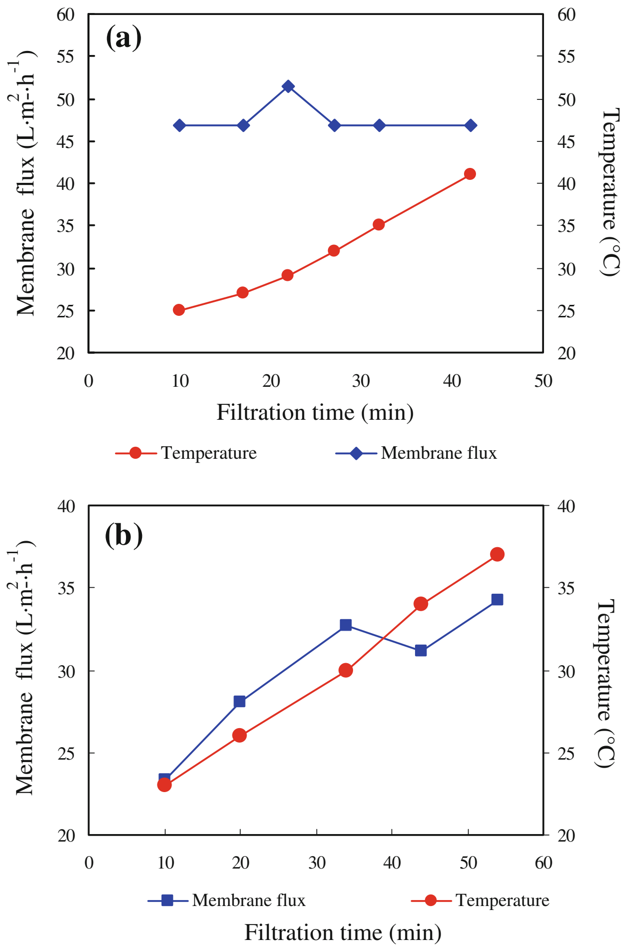

Fig. 2 Changes of membrane flux and temperature of water samples during filtration by VSEP: **a** sample A; **b** sample C

Fenton oxidation

$[Fe^{2+}]/[H_2O_2]$

Permeate was mixed together according to the real wastewater prior to experiment. The COD content in the mixture was 21,975 mg/L. Figure 3a, b illustrated the influence of the ferrous dosage on COD removal at the initial $[H_2O_2]/[COD]$ weight ratios of 1.55 and 0.77, respectively. It was found that the optimum range of ferrous dosage to achieve the maximum organic mineralization was from 1.4 to 2.8 g/L (corresponding molar ratio of ferrous and H_2O_2 was 0.05 ∼ 0.1).

The COD removal rate could be achieved to 61 and 69 % when H_2O_2/COD (w/w) ratio was 0.77 and 1.55, respectively. When the ferrous dosage was less than 1.4 g/L, the decomposition of organics was inhibited. Besides, the residual hydrogen peroxide in the solution could be also observed after 3-h reaction (data not shown). The ferrous ions reacted very rapidly with hydrogen peroxide to produce hydroxyl radicals (reaction 2) which could decompose organics quickly, and then ferric ions were reduced to ferrous ions by hydrogen peroxide (reactions 4 and 5). However, the rate of oxidation in the second stage (ferric system) was much slower than in the first one due to the low generation rate of ferrous from ferric ions (Neyens and Baeyens 2003; Pignatello et al. 2006). The removal of COD slightly decreased as the initial dosage of ferrous was over 2.8 g/L. This was attributed to the competition of reactions 3 and 6, even although this type of competition was negligible at high COD/H_2O_2 ratios (Neyens and Baeyens 2003).

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + \cdot OH + OH^- \quad (2)$$

$$\cdot OH + Fe^{2+} \rightarrow Fe^{3+} + OH^- \quad (3)$$

$$Fe^{3+} + H_2O_2 \leftrightarrow Fe-OOH^{2+} + H^+ \quad (4)$$

$$Fe-OOH^{2+} \rightarrow HO_2\cdot + Fe^{2+} \quad (5)$$

$$RH + \cdot OH \rightarrow H_2O + R\cdot \rightarrow \text{further oxidation} \quad (6)$$

The ferrous ions generated in the above redox reactions could react with hydroxide ions to form ferric hydroxo complexes which acted as coagulant to capture and precipitate with suspended particles (Bautista et al. 2008). Such coagulation/precipitation behavior played an important role in COD removal of leachate, since it had a high concentration of SS that can be easily removed by coagulation (Kang and Hwang 2000). The removal of COD by oxidation and coagulation was also demonstrated in Fig. 3. The removal rate showed similar trends at the H_2O_2/COD weight ratios of 1.51 and 0.77 (Fig. 3a, b). Apparently, the COD removal ratio through ferric hydroxide coagulation was less than 10 % at both H_2O_2/COD weight ratios. The result indicated that it cannot improve the contribution of coagulation in overall organic removal with increasing ferrous dosage, say more than 14 g/L. This could be explained that surfactants used in O/W emulsions were hydrophilic dissolved organics (log K_{OW} values less than 3; Shon et al. 2006). This was in agreement with the result from Bautista et al. (2008), who reported that coagulation–flocculation had a poor effect on the removal of soluble organic compounds such as reactive dyes.

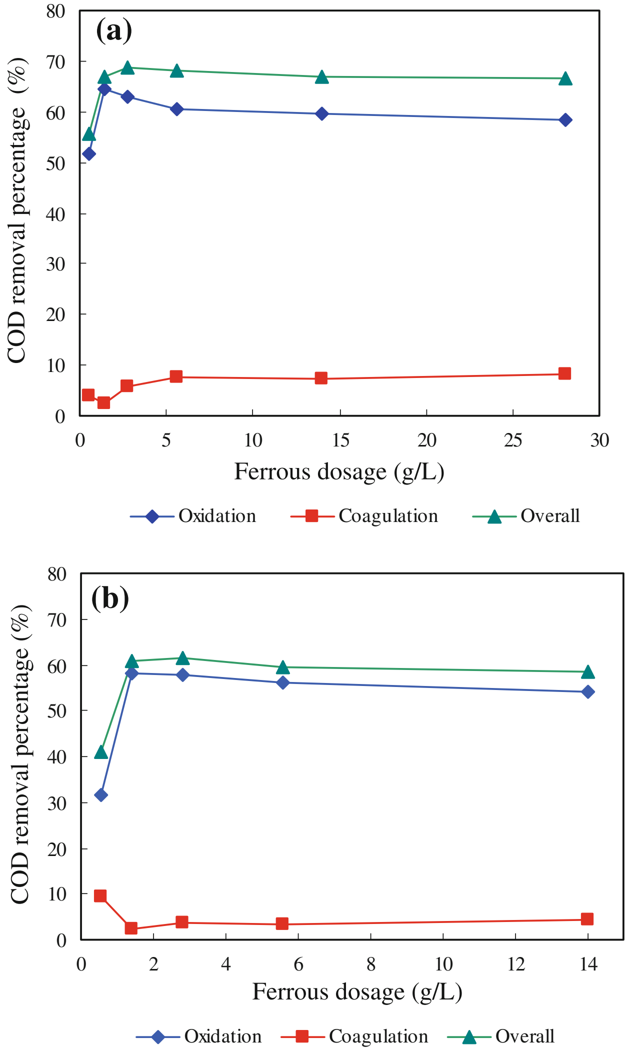

Fig. 3 Influence of ferrous dosage on COD removal ratio by oxidation and coagulation: **a** H_2O_2/COD (w/w) = 1.55; **b** H_2O_2/COD (w/w) = 0.77

The influence of hydrogen peroxide dosage on its efficiency

Figure 4 showed that the efficiency of H_2O_2 reached a maximum value of 1.11 (g COD removed/g H_2O_2) when the dosages of H_2O_2 and ferrous ion were 6.8 and 2.8 g/L, respectively. The H_2O_2 efficiency improved when H_2O_2 dosage was below this value, whereas the removal efficiency drastically decreased at a higher H_2O_2 dosage. In the case of serving low ferrous dosage in the solution, it is rational to assume that ferric ions formed from Fenton reaction might complex with certain organic ligands; consequently, the "passivated" ferric ions could no longer participate in the regeneration reaction of ferrous ions through ferric system. This might weaken the oxidation processing, as well as H_2O_2 efficiency. The causes for the latter were more complex; there might be some organics or partially oxidized intermediates which were resistant to chemical oxidation and could not be effectively removed by Fenton's reagent. On the other hand, ferrous ions were added in the form of sulfate salt. Due to the occurrence of complexation reactions of Fe(III) by SO_4^{2-} (reactions 7 and

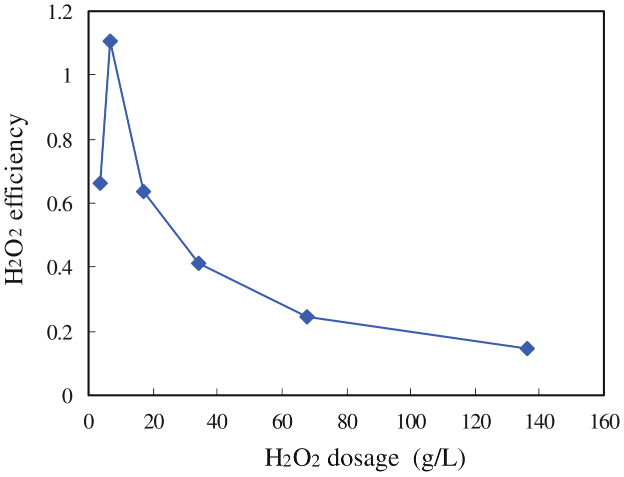

Fig. 4 Effect of hydrogen peroxide dosage on its efficiency (Fe^{2+}/H_2O_2 molar ratio, pH, and reaction time were fixed as approximately 0.05, 3, and 3 h, respectively.)

8), the increase of the SO_4^{2-} concentration could lead to a decrease of the concentration of peroxo complexes, and consequently, a decrease of decomposition rate of H_2O_2 (De Laat et al. 2004). In addition, the sulfate anion in the solution could act as a hydroxyl radical scavenger and react with ·OH according to reactions 9 and 10 (De Laat et al. 2004; Siedlecka and Stepnowski 2007), resulting in a decrease of degradation.

$$Fe^{3+} + SO_4^{2-} \leftrightarrow FeSO_4^{+} \tag{7}$$

$$Fe^{3+} + 2SO_4^{2} \leftrightarrow Fe(SO_4)_2^{-} \tag{8}$$

$$H_2SO_4 + \cdot OH \rightarrow SO_4^{-}\cdot + H^{+} + H_2O \tag{9}$$

$$HSO_4^{-} + \cdot OH \rightarrow SO_4^{-}\cdot + H_2O \tag{10}$$

Influence of initial organic content and Fenton oxidation on biodegradability of wastewater

The performance and efficacy of biological treatment was affected significantly by composition and concentration of the toxic organic matters in wastewaters, especially industrial wastewaters containing high strength organics. Thus, biodegradability of raw permeate was analyzed at three dilutions (1:1, 1:4, and 1:10) to understand the behavior of sludge in contact with wastewater with initial different concentrations. Note that each flask was spiked with an appropriate amount of activated sludge to yield the same initial food-to-microorganisms (F/M) ratio. As shown in Fig. 5, the ultimate COD degradation rates are 75, 59, and 41 % when unoxidized filtrate was also bioassayed at 1,000, 5,000, and 10,000 mg/L as COD, respectively. The high content might be possibly inhibitory to sludge metabolism and resulted in low biodegradation rate. Therefore, chemical pre-oxidation was a required step to eliminate this concentration effect.

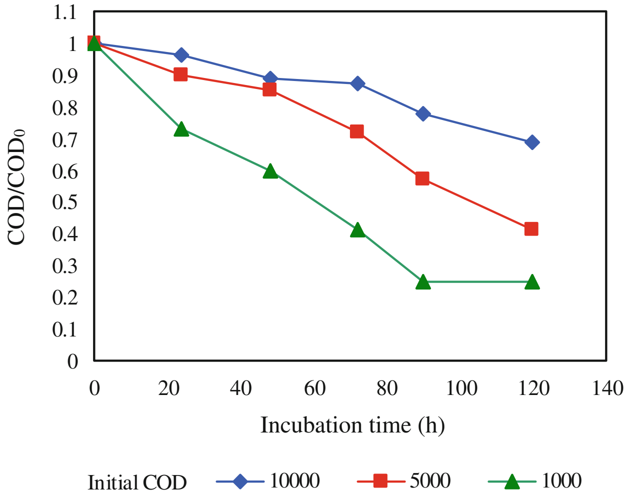

Fig. 5 Influence of initial organic concentration on biodegradability

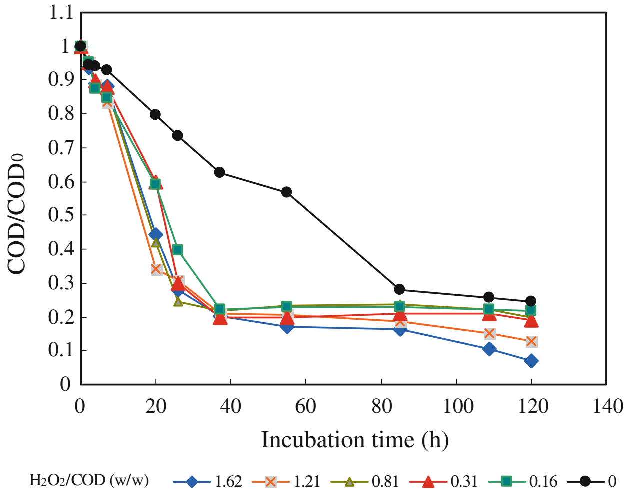

Fig. 6 Influence of H_2O_2/COD (w/w) on biodegradability (Fe^{2+}/H_2O_2 molar ratio, pH, and reaction time were fixed as approximately 0.05, 3, and 3 h, respectively.)

As stated previously, application of Fenton's reagent was an efficient way to reduce organic content of this wastewater through complete mineralization via the generation of hydroxyl radicals, but the effect of Fenton oxidation on degradability of this wastewater was unknown. Therefore, aerobic batch bioassays were also conducted to evaluate the biodegradability of oxidized and unoxidized samples at initial concentrations of 1,000 mg/L as COD. For all samples, 70 % biodegradability was attained after 90 h of biotreatment (Fig. 6). Oxidized samples were biodegradable after only 24 h even at a low dosage of Fenton's reagent, but untreated sample needed much longer adaptation periods (90 h). These results indicated that using Fenton's reagent could accelerate bio-oxidation rate by converting the biorefractory compounds (surfactants and additives) into small concentration of organic intermediates

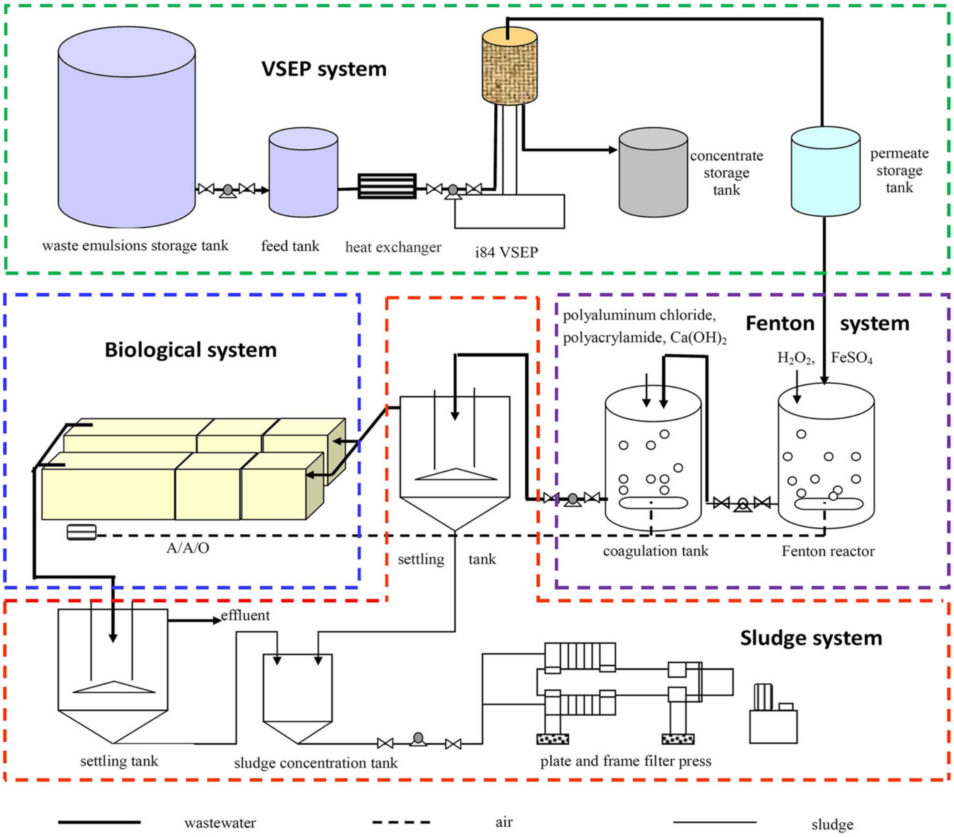

Fig. 7 Full-scale process for treating waste emulsions and other industrial wastewaters

such as carboxylic acids, formic acid or formaldehyde that were more biodegradable. When the dosages of hydrogen peroxide and ferrous iron were 6.8 and 2.8 g/L, ultimate biodegradation rate of wastewater increased up to 81 %. No significant improvement of bio-oxidation rate was observed with the further increase in H_2O_2 dosages. Considering the above results about H_2O_2 efficiency, this dosage could be used to yield sufficient wastewater biodegradability and minimize the hydrogen peroxide consumption.

Full-scale application

This integrated process has been successfully applied to treat various emulsions and other industrial wastewaters, such as passivation liquid, paint wastewater, metal surface treatment liquid and organic solvent wastewater by recombination of several treatment units (coagulation–flocculation, Fenton oxidation, VSEP, anaerobic–anoxic–aerobic treatment) in Shanghai industrial park. The design capacity of wastewater

Table 3 Operating conditions and COD removal efficiency of each treatment unit

Stage	Operating conditions				Effects
VSEP	DA (cm)	P_{in} (kPa)	P_{out} (kPa)	T (°C)	COD (mg/L)[a]
	2	414	207–310	60–80	11,000 ± 2,000
Fenton oxidation	Initial pH	H_2O_2 dose (v/v)	Ferrous dose (g/L)	Reaction time (h)	COD (mg/L)
	3–3.5	1 %	1.4	3	6,000 ± 2,000
Biological system[b]	DO (mg/L)	HRT (day)	SRT (day)	Volume loading (kg COD /m^3 day)	COD (mg/L)
Anaerobic	0–0.2	0.5	>300	12 ± 4	2,000 ± 500
Anoxic	0.4–0.6	0.5	>300		
Aerobic	2.5–3	2	25–30	1 ± 0.25	400 ± 100

HRT hydraulic retention time, *SRT* sludge retention time, *DA* displacement amplitude, *DO* dissolved oxygen, *T* temperature

[a] Inlet COD content of VSEP was 130,000 ± 30,000 mg/L

[b] Influent pH, TN and TP were 7–8, 295 ± 50 mg/L and 1.3 ± 0.5 mg/L

plant is 100 m^3/day, and entire flow diagram is depicted in Fig. 7. The anaerobic/anoxic/aerobic process (A^2/O) was used for biological treatment. Each biochemical pond was filled with composite carriers to enhance the ability against shock loading. Operating parameters and the contribution of each stage to COD removal were given in Table 3. The final effluent quality could reach the discharge standard for municipal sewerage system in Shanghai (DB31/445-2009).

Conclusion

In this study, a combination of VSEP and Fenton's oxidation was used to reduce organic loading and improve biodegradability for subsequent biological treatment of waste emulsions. More than 85 % of oil could be removed for all three emulsions by VSEP, while COD removal efficiency varied widely. The flux was found to be very stable with time due to a high shear rate of VSEP and the increase in water temperature caused by frictional heating. However, high concentration of organic contaminants still remained in UF permeate, hence a further treatment was required to meet the wastewater discharge standards. Since the microbial activity was inhibited by the high concentrations of organics, resulting in a slow biodegradation rate, and the biodegradability was significantly enhanced by Fenton oxidation. The optimum molar ratio of ferrous and H_2O_2 was approximately 0.05. COD degradation rate did not linearly relate with the dosage of H_2O_2, and efficiency of H_2O_2 ascended firstly and decreased later with an increase in H_2O_2 dosage. The contribution of coagulation on the removal of organic matters in permeate was rather limited in Fenton process. The improvement of biodegradability was attributable to both formation of more biodegradable oxidized byproducts and reduction of organic loading in wastewater.

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