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Treatment of the petroleum refinery wastewater using combined electrochemical methods

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ABSTRACT

This study explores the performance of electrochemical and combined oxidation methods for the treatment of petroleum refinery wastewater (PRW). The electrocoagulation operating parameters studied includes reaction time and applied voltage. *In situ* hydrogen peroxide treatments at different dosages and hybrid electrochemical systems (ECEO, ECS_{25}, ECS_{33}, ECEO + US_{33}) have been studied. The efficacy of the treatment system has been evaluated in terms of reduction of phenols, COD, TOC, oil, and grease. The most effective method for PRW treatment is ECEO + US_{33} where the maximum % removal of phenols, TOC, COD, oil, and grease are reported to be 98, 92, 92, and 92%, respectively. The results demonstrate the technical feasibility of using hybrid electrochemical process as a possible and reliable methods for the treatment of PRW.

Keywords: Refinery; Electrocoagulation; Electrooxidation; Peroxidation; Ultrasonication

1. Introduction

Petrochemical industries cause considerable water pollution by discharging their effluents into the surrounding aquatic environment. Large amount of wastewater is produced from petrochemical manufacturing processes like distillation, cracking, treating, and reforming. Petroleum refinery industrial processes occupied the first place in releasing high volumes of oily wastewater compared to any other industrial processes. The average release of the wastewater is 0.4–1.6 times to the volume of the processed crude oil. Consequently, a wide variety of pollutants including refractory organics are usually encountered in petrochemical wastewater [1,2]. The chemical composition of petroleum refinery wastewater (PRW) effluent is very complex and contains several inorganic substances, such as Mg^{2+}, Ca^{2+}, S^{2-}, Cl^{-}, and SO_4^{2-} that upgrade the mineralization of water, emulsified oil, phenols, and sulfides. This wastewater must be treated before being released into the freshwater [3].

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Conventional wastewater treatment methods, including gravity separation and skimming, air flotation, coagulation, de-emulsification, and flocculation, have intrinsic disadvantages such as low efficiency, high operation cost, and corrosion and generates a considerable quantity of secondary pollutants (chloride, sulfate in the coagulation–precipitation) and large volumes of sludge or waste which pose serious environmental problems [4,5]. Evaporation of these solutions in ponds is impractical due to their high salt concentration [6]. Even the biological processes are also inefficient in treating PRW, because the partial inhibition of biodegradation is observed. It might be due to the presence of the sulfide which affects the oxygen transfer of the biosystems [7]. Nevertheless, these processes present limitations which can be accomplished by electrochemical methods.

Electrochemical process is advantageous over the conventional coagulation process, in which the reactants are generated *in situ* through the dissolution of a sacrificial anode by applied current with simultaneous evolution of hydrogen at the cathode. This method has been shown as an efficient and promising technique in treating various wastewater contaminants such as reactive dyes [8], azo dyes [9], metal cutting [10], oily bilge water [11], industrial wastewater [12], poultry slaughter house [13], fluoride [14], pulp and paper mill wastewater [15], phosphate, zinc [14], and textile wastewaters [16]. In hybrid electrochemical methods there are combined reaction possibilities such as electrocoagulation (EC), electrooxidation (EO), and electroflotation along with other treat systems (ultrasonication, photocatalysis, ozonation, and other oxidation applications). Electrochemical methods are efficient in removing soluble inorganic solids, suspended solids as well as oil and grease. It has also the advantage of producing a relatively low amount of sludge [5]. The reactions occurring in an electrochemical cell involving aluminum electrodes are as follows:

Reactions at the anodes:

$$\mathrm{Al} \longrightarrow \mathrm{Al^{3+}}_{(\mathrm{aq})} + 3^{\mathrm{e}-} \tag{1}$$

$$\mathrm{Al\text{-}3^{e^-}} = \mathrm{Al^{3+}} \tag{2}$$

Reactions at the cathodes:

$$3\mathrm{H_2O} + 3^{\mathrm{e}-} \longrightarrow 3\mathrm{H_2(g)} + 3\mathrm{OH^-} \tag{3}$$

Al metal ions immediately form hydroxides and/or poly hydroxides that finally transform into $\mathrm{Al(OH)_3}$ ($_\mathrm{S}$), as shown in Eqs. (1) and (2) (electrocoagulation). Hydroxides have strong affinity to capture the pollutants in the waste water, causing more pollutant removal than those conventional methods. Eq. (3) involves electrooxidation phenomena [17,18].

Along with anodic dissolution of the coagulant, hydrogen bubbles are released from the cathode due to water splitting. Gas evolution during electrochemical treatment wastewaters has been termed electroflotation [19]. This process might be responsible for the removal of oil and grease from the oily waters [17,20].

The organic recalcitrant compounds could also be effectively oxidized by oxidizing agents such as hypochlorite and peroxide. Hypochlorite formation might be due to the presence of NaCl which is readily available in the PRW (Eqs. (4)–(6)). This might be responsible for the indirect oxidation of organics in the solution and/or near the anode surface [17,21,22].

$$2\mathrm{Cl^-} \longrightarrow \mathrm{Cl_2} + 2^{\mathrm{e}} \tag{4}$$

$$\mathrm{Cl_2} + \mathrm{H_2O} \longrightarrow \mathrm{HOCl} + \mathrm{H^+} + \mathrm{Cl^-} \tag{5}$$

$$\mathrm{HOCl} \longrightarrow \mathrm{H^+} + \mathrm{ClO^-} \tag{6}$$

Hydrogen peroxide is most often used as chemical oxidant to improve the radical formation to degrade the organic pollutants. Specifically, peroxide is more effective in the removal of sulfide and $\mathrm{H_2S}$ from PRW. This might be due to the conversion of sulfide in sulfate which can be easily removed from solution by electrochemical methods [23].

Sometimes it may become necessary to use two or more methods of treatments, i.e. hybrid processes, to ensure efficient treatment of wastewater. Removal of coloring materials from dye stuffs using electrochemical methods has been reported by Akyol [5], Lin and Peng [24], Pouet and GrasmicK [25], and Mahesh et al. [15]. Combined electrochemical oxidation, coagulation, and activated sludge have been reported by Lin and Peng [24]. EC has also been used in conjunction with filtration to remove silica and suspended solids that tend to foul reverse osmosis membranes [25,26].

However, no attempt has been made so far in the usage of combined technique (*in situ* electrocoagulation–ultrasonication) for the treatment of PRW. Hence, the present paper discusses the treatment of PRW using EC and hybrid techniques.

2. Materials and methods

2.1. Petroleum refinery wastewater

The PRW is provided by refinery processing industry, India. Initial characteristics of the wastewater are represented in Table 1.

2.2. Experimental setup and methodology

The initial characterization of the sample has been carried according to “standard methods for examination of water and wastewater 21th addition-2005, APHA” [27]. In electrocoagulation method, the operational parameters like reaction time (10–40 min) and current voltage (10–30 V) are investigated. The above EC experiments are carried in a 250 mL glass beaker having a working capacity of 200 mL as shown in Fig. 1(A). Al Electrodes are connected to the respective anode and cathode leading to the DC rectifier (AP lab, L 6403(1–84 V, 0–3 A)). The active surface area of the each electrode is 12.6 cm^2 and the inter-electrode distance between the anode and cathode rods is 3.5 cm. Before each run, aluminum electrodes are washed with tap water and then again rinsed with distilled water.

To increase the organic removal from PRW, the peroxi-electrocoagulation, EC followed by electro oxidation (ECEO) and *in situ* EC ultra sonication (US) are studied.

In peroxi-EC experiment, peroxide dosages are varied from 50 to 400 ppm. The *In situ* EC ultrasono-EC (25 kHz (ECS$_{25}$), 33 kHz (ECS$_{33}$)) are carried as shown in Fig. 1(B) where EC reactor is placed in the sonicator bath which is constituted of a 3.3 L ultrasonic bath (Model No. EN-30-US, Enertech Electronics Pvt Ltd, Bombay, India) with a selective- frequency-based electronic ultrasound generator. The ultrasonic bath has a two piezo-ceramic transducers bonded to the bottom of a stainless steel tank with the option of operating at 25 or 33 kHz in a continuous or pulse (5 s on and 1 s off) mode.

Finally, ECEO followed by ultrasonication at 33 kHz (ECEO + US$_{33}$) has been studied. All the experiments are performed at room temperature (27 ± 2˚C). The samples are collected at an interval of 10 min and are analyzed for reduction of phenol, COD, TOC, oil, and grease content.

Table 1
Initial characterization of the effluent sample

S.No	Name of the parameters	Initial (mg L^{-1})
1	pH	7.92
2	EC (microsiemens/cm)	2,084
3	Total dissolved solids (TDS)	1,550
4	Total solids(TS)	2,000
5	Total suspended solids (TSS)	450
6	Phenols	79
7	Biological oxygen demand (BOD)	195
8	Total hardness(TH)	200
9	Chemical oxygen demand (COD)	760
10	Alkalinity as $CaCO_3$	515
11	Calcium hardness as $CaCo_3$	110
12	Magnesium hardness as $CaCo_3$	90
13	Chlorides as Cl^-	600
14	Sulfates as SO_4	116
15	Nitrates as NO_3^-	146
16	Phosphates as PO_4	1.43
17	Sodium as Na	380
18	Potassium as K	22
19	Fluoride as F^-	0.25
20	Turbidity (NTU)	130.4
21	Sulfides SO_3	14.4
22	Oil and grease	150
23	Total organic carbon	620

Note: All the parameters are expressed in mg L^{-1} except pH, EC, and Turbidity.

2.3. Analytical instruments

The instrumental analysis is carried using Double beam Shimadzu UV 2450 UV–visible Spectrophoto meter and TOC-L CPH E 200.

The pollutant removal percentage (%) is calculated as follows:

$$\text{Removal of the pollutant } (\%) = \frac{C_i - C_f}{C_i} \times 100 \tag{7}$$

where, C_i is the initial pollutant concentration (mg L^{-1}) and C_f is the final pollutant concentration (mg L^{-1}).

3. Results and discussion

The initial characterization of the PRW is given in Table 1.

3.1. Factors affecting the performance of electrocoagulation

3.1.1. Effect of reaction time on the performance of electrocoagulation

Operating time is an important parameter for economic applicability of the EC process. In the present study, the time course of EC has been observed at time intervals of 10, 20, 30, and 40 min. During initial stages of the treatment, the % removal efficiency was observed to increase up to 20 min after which the removal rate was observed to be constant. So 20 min has been chosen as the optimal reaction time for EC of the PRW sample. At this reaction time, 84, 85, 84, and 66% of the phenol, COD, TOC, oil, and grease % reduction have been observed (Fig. 2). The decrease in removal rate after 20 min might be due to the

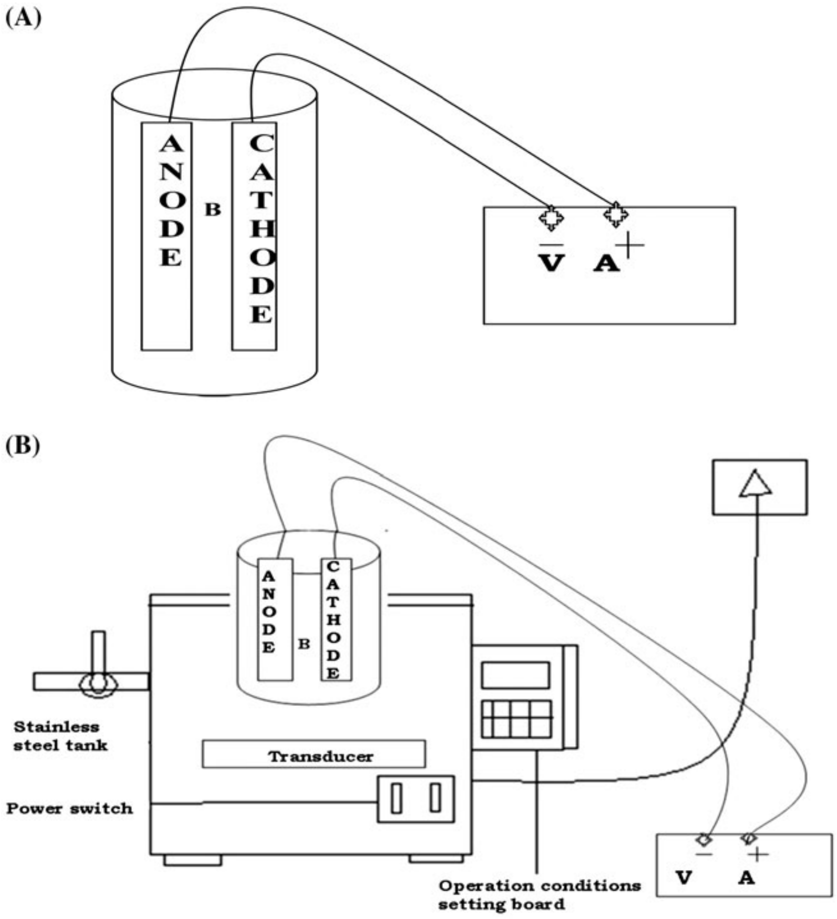

Fig. 1. (A) Systematic design of electrocoagulation setup. (B) Schematic design of sonicator with electrocoagulation.
Note: B-Beaker, V-DC Voltage, and A-DC Ampere.

decreased extent of cathodic reduction (passivation) and formation of monomeric electrocoagulants [5]; this monomeric hydrolysis products includes ($Al(OH)^{2+}$, $Al(OH)_2{}^{+}$, and $Al_2(OH)_2{}^{4+}$) [26,28]. The same results also observed by Mollah et al. [26] and Ahmadi et al. [29] in terms of oil and grease, Akyol in terms of TOC [5] and Ugurlu in case of the paper mill effluent treatment [30]. According to Zhao et al. [31], dissolved substances are difficult to remove only by individual electrocoagulation process even with the reaction time was increased [28,31,32].

3.1.2. Effect of applied voltage on the performance of electrocoagulation

Among the various operating parameters in all the electrochemical processes, the applied voltage is an important factor which strongly influences the performance of electrocoagulation. The effects of applied voltage on the pollutants removal efficiency from PRW have been investigated at voltages of 10, 15, 20, 25, and 30 V, and corresponding current density of 6.3, 9.4, 16.6, 21.3, and 28.4 mA/cm^2 (Fig. 3) had observed, respectively. The maximum % removal (Fig. 4) of the phenol, COD, TOC, oil, and grease has been observed to be 89, 84, 84, and 67%, respectively, at an applied voltage of 20 V. The results showed that the removal efficiency increased with increasing voltage up to 20 V. Further, increase in the voltage leads to a constant or slight decrease in the removal efficiency.

Generally with an increase in the current density, the dissolution of anode and generation of bubbles at cathode increases. This improves the degree of mixing of $Al(OH)_3$ which further enhances floatation ability of the cell, thus, increasing the pollutant removal efficiency [5,33]. However, an excessive increase in the current voltage causes a reduction in the production of the flocs. This had also been previously observed by Adhoum and Monser [34], according to whom this

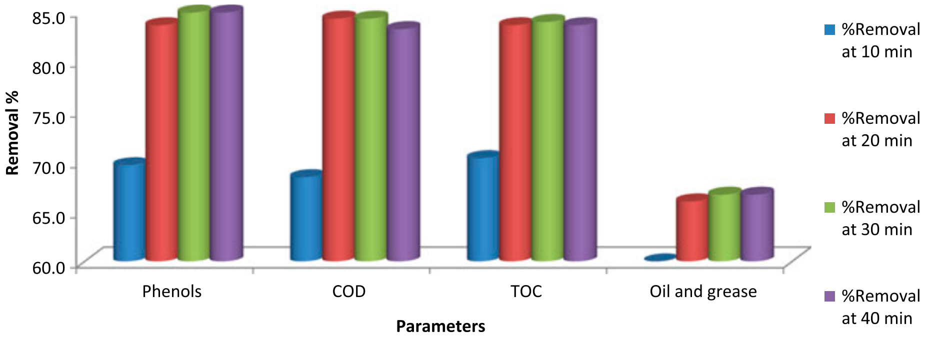

Fig. 2. Effect of electrocoagulation time for the treatment PRW.
Note: Conditions: volume of the sample: 200 mL, electrode: Al/Al, voltage: 10 V, reaction time 10–40 min, surface area of electrode: 25.3 cm^2, and current density: 6.32 mA/cm^2.

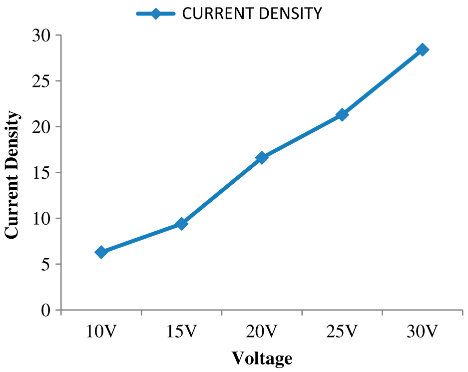

Fig. 3. Applied voltages with respective current density.

might be due the faster generation of gas bubbles, which are responsible for removal of aluminum hydroxide from solution by flotation further leading to a reduction in the probability of collision between the pollutant and coagulant. Thus, despite the high removal efficiency obtained, high current density has been observed to be not beneficial for the maximum use of the flocs [5,17,26].

3.2. Effect of hydrogen peroxide on the performance of electrocoagulation

Peroxide is a strong oxidizing agent and it doesn't pollute the water. The treatment of PRW is performed at peroxi-electrochemical process by varying H_2O_2 dosages (50–400 ppm). It has been observed that from the Fig. 5, with an increase in the addition of H_2O_2, the percentage removal of pollutants has also increased till 300 mg L^{-1} dosage. At this point maximum of % removal of 91.9, 92, 92, and 92% of phenol, COD, TOC, oil, and grease has been achieved. Above which (>300 mg L^{-1}) no significant increase in the % removal of the pollutants has been observed. Increased H_2O_2 dosage increases the $^{\cdot}OH$ radicals generation which further enhances the oxidation ability of treatment process. However, the decressive trend indicated that the over-abundant H_2O_2 could also consume $^{\cdot}OH$ and eliminate the hydroxyl radical generating oxygen (Reactions 8 and 9) [29,30,34]. It is clearly observed from Fig. 5 that maximum removal of phenol is achieved at a dosage of 200 mg L^{-1} of peroxide. This variation between COD and phenol

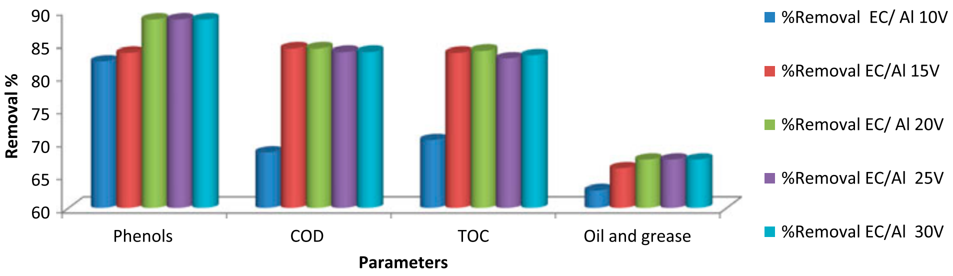

Fig. 4. Effect of applied voltage for the treatment of PRW.
Notes: Conditions: volume of the sample: 200 mL, electrode: Al/Al, voltage: 20 V, reaction time 20 min, surface area of electrode: 25.3 cm^2, current density for 10 V: 6.32 mA/cm^2, current density for 15 V: 11.7 mA/cm^2, current density for 20 V: 16.6 mA/cm^2, current density for 25 V: 21.3 mA/cm^2, and current density for 30 V: 28.4 mA/cm^2.

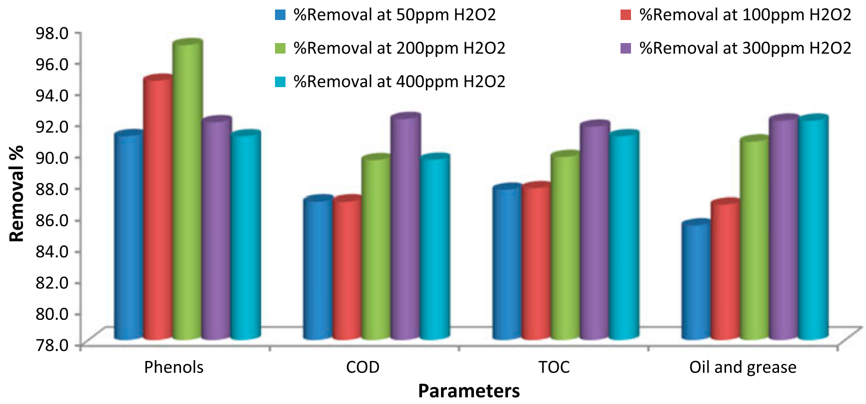

Fig. 5. Effect of *in situ* peroxi-electrocoagulation for the treatment of PRW.
Note: Conditions: volume of the sample: 200 mL, electrode: Al/Al, voltage: 20 V, reaction time 20 min, surface area of electrode: 25.3 cm^2, current density for 20 V: 16.6 mA/cm^2, peroxide concentration: 50–400 ppm.

removal pattern might be due to the presence of complex structure of phenols [35,36].

$$H_2O_2 + {}^{\cdot}OH \longrightarrow HO_2^{\cdot} + H_2O \quad (8)$$

$$2H_2O_2 \rightarrow O_2 + 2H_2O \quad (9)$$

3.3. Comparison between different treatment processes for PRW treatment

The results shown that the % reduction of COD in ECS_{33} is observed to be more, while compared with ECS_{25} treatment process (Fig. 6). This might be due to generation of more OH radicals at higher frequencies [31,32]. Oxidation efficiency of the ECS_{33}, ECEO, and ECEO + US_{33} is observed to be same in case of COD and TOC. The percentage removal of phenol, oil, and grease under different treatment processes is found to be in the order of $ECS_{25} < ECS_{33} <$ ECEO < ECEO + US_{33} which is of 91, 96, 97, and 98% of phenols, and 74, 83, 90, and 92% of oil and grease, respectively.

The pollutant removal by hybrid electrochemical techniques might be due to the direct anodic oxidation and indirect oxidation species (Reactions 4–6). The presence of sufficiently high chloride ion concentrations (concentrations greater than 300 mg L^{-1}) is sufficient for the formation of HOCl at certain pH values and potentials. Typically, chloride is regenerated; however, depending on the parameters of the electrochemical process some chloride can also escape from the solution in the form of gaseous chlorine [17,21,31]. Moreover, phenol and COD removal might also involve electrochemical oxidation and adsorption by electrostatic attraction and physical entrapment [20,34,36–38]. Electrochemical techniques combination with ultrasonication had shown the remarkable increment in % removal of the pollutants from PRW.

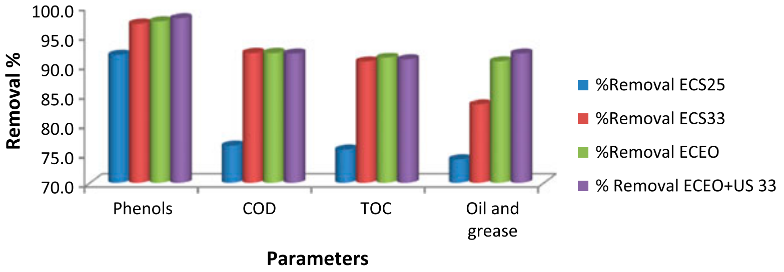

Fig. 6. Electrocoagulation combined with other techniques.
Note: Conditions: ECS_{25}: volume of the sample: 200 mL, electrode: Al/Al, voltage: 20 V, reaction time 20 min, surface area of electrode: 25.3 cm^2, current density for 20 V: 16.6 mA/cm^2, ultrasonication at 25 kHz. ECS_{33}: volume of the sample: 200 mL, electrode: Al/Al, voltage: 20 V, reaction time 20 min, surface area of electrode: 25.3 cm^2, current density for 20 V: 16.6 mA/cm^2, ultrasonication at 33 kHz. ECEO: volume of the sample: 200 mL, electrode: Al/Al (EC), & SS/SS (EO), voltage: 20 V, reaction time 20 min, surface area of electrode: 25.3 cm^2, current density for 20 V: 16.6 mA/cm^2. ECEO + US_{33}: ECEO followed by ultrasonication at 33 kHz.

4. Conclusions

(1) The application of hydrogen peroxide for enhancing the oxidative process of the PRW is observed to be beneficial.
(2) EC alone is not suitable for treatment of the refinery waste waters, because PRW is very complex in composition.
(3) PRW treatment needs collaborative treatment processes like electrochemical methods and/or ultrasonication.
(4) From this study, the maximum % removal of 98, 92, 92, and 92% of phenols, TOC, COD, oil, and grease is observed in ECEO + US_{33} process.

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