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Evaluation and start-up of an electro-Fenton-sequencing batch reactor for dairy wastewater treatment

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ABSTRACT

This study examined the performance of an integrated wastewater (WW) treatment system, namely an electro-Fenton (EF)-sequencing batch reactor (SBR), for dairy industry WW. The EF process was used as the first stage of the SBR. It degrades bio-refractory compounds via advanced oxidation processes, thereby resulting in the formation of simple biodegradable intermediates. Several factors, including the hydraulic retention time (HRT), sludge volume index (SVI), and sludge retention time (SRT) of the EFSBR, were optimized. The effectiveness was studied for 1 y laboratory-scale experiments under stable conditions (HRT of 10 h, SRT of 15 d, mixed liquor suspended solids concentration of 3500 mg/L, and SVI of 89), which revealed 99% chemical oxygen demand, 97% total nitrogen, and 95% total phosphorus removal. Thus, the developed system is economically feasible and superior to other conventional biological treatment systems.

1. Introduction

Discharge of wastewater (WW) containing a high load of organic pollutants is an important environmental issue [45,46]). It causes the growth of algae and aquatic plants in shallow rivers, which affect the esthetic properties of water. A high nutrient load (nitrogen and phosphorus) can cause adverse effects such as reduced concentrations of dissolved oxygen (DO). Among the various food industries, the dairy industry is severely affected by the presence of high loads of organic matter in such effluents [3,24,36,50]. Biological processes that remove nutrients from industrial WW can be performed in several systems, including conventional activated sludge (AS), membrane bioreactors, oxidation ditches, upflow anaerobic sludge blanket reactors, and sequencing batch reactors (SBRs) [7,23]. SBRs use a modified AS process and are operated using a sequence of five discrete phases (filling, reaction, settling, draw, and

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idle). These phases occur in a single reactor basin. During the filling phase, raw WW is mixed with biomass from the SBR, and the reaction (under aerobic conditions) is aided by agitation and aeration [50,51]. In the next phase, stirring is discontinued to allow the sludge to settle (settling phase). During the draw phase, the effluent is discharged. This cycle is then repeated [35]. This approach has many advantages, such as low energy consumption, small space requirement, low construction and operational costs, high flexibility, and high effectiveness compared with other types of biological processes [22,40].

Advanced oxidation processes (AOPs) are chemical processes based on the application of radical species [mainly hydroxyl radicals ($^{\bullet}OH$)] for the degradation of complex organic compounds. The final treatment products are in the form of CO_2, H_2O, and inorganic salts [5,17]). AOPs such as photocatalytic oxidation [16], ozonation (Fernandes et al., 2019, Fernandes et al., 2018), cavitation-based processes [14,15,17], and the Fenton process have demonstrated reasonable performance in the treatment of industrial WW. The Fenton reaction (Fe^{2+}/H_2O_2) is widely used because of its advantages such as ease of operation at ambient temperature and pressure, environmental friendliness, higher efficiency in removing a wide range of organic compounds, and low cost [26,27,30,32]. The $^{\bullet}OH$ generated during the Fenton reaction are highly reactive, and thus can completely degrade organic compounds by hydrogen abstraction. However, this process requires a high consumption of chemicals owing to the acidification of the solution. This causes the generation of high metal sludge [38,39], which is not economically feasible for high pollutant loads. Moreover, the sole use of AOPs such as Fenton or electrochemical oxidation does not provide satisfactory treatment effectiveness (Vahid and Khataee, 2013, Asaithambi et al., 2020).

The electro-Fenton (EF) process [15] is one of the most popular modifications of the classic Fenton process that enhances the oxidation of organic compounds. In the EF process, iron ions are introduced into the liquid phase via a controlled electrochemical reaction. The presence of iron ions in the EF process is an important factor because iron ions are responsible for conducting the catalytic Fenton cycle [11,42], whereas an excess amount of iron ions can cause additional sludge formation. Hybrid processes combining conventional WW treatment (e.g., biological methods) with AOPs are effective and economical methods that allow the degradation of persistent organic pollutants to biodegradable intermediates in the primary step. Biological processes provide the final treatment step resulting in 100% removal of pollutants [21,50,54].

In this study, a novel strategy using the EF process as one of the stages in an SBR (EFSBR) was proposed. The EFSBR process was expected to reduce the treatment cost owing to the low energy consumption and to minimize the shock caused by the high load of pollutants at the inlet of the reactor. It was also expected to ensure effective removal of EF by-products, improve the efficiency with the variety of operation conditions, and reduce the treatment time. However, this approach requires further study and validation with respect to various types of real effluents. The purpose of this study was to design and evaluate the efficiency of the combined EFSBR process with the addition of an extra EF phase for the treatment of dairy WW. To optimize the operation conditions of the EFSBR, the effects of various parameters [i.e., pH, H_2O_2 concentration, current density, hydraulic retention time (HRT), mixed liquor suspended solids (MLSS) concentration, sludge volume index (SVI), sludge retention time (SRT), and reaction time (t_R)] on the overall degradation performance were evaluated.

2. Materials and methods

2.1. Characteristics of dairy industry WW

The AS used in this research was obtained from a municipal WW treatment plant treating dairy industry WW (DIW) in Kerman, Iran and stored at 4 °C until use. Coarse particles were separated using a filter. The system reached steady state after 15 d of operation. The characteristics of the DIW and seed sludge are shown in Table 1.

2.2. EFSBR operation and setup

The EFSBR was made of Plexiglas with a total volume of 10 L, dimensions of 18.8 cm (length) × 18.5 cm (wide) × 30.0 cm (height), which was divided into two sections (EF and biological basins) and six phases (filling, AOP, aeration, settling, draw, and idle) (Fig. 1). The filling phase occurred in the upper part of the EF basin with the filling of WW using a peristaltic pump (DLS MA. ETATRON, Co.

Table 1
Characteristics of the dairy industry wastewater and seed sludge.

Parameter	Dairy wastewater	Seed sludge
Sludge volume index	–	90 mg/L
Mixed liquor suspended solids	–	2000 mg/L
Mixed liquor volatile suspended solids	–	1670 mg/L
Chemical oxygen demand	3225 mg/L	578 mg/L
pH	6.5–8.0	7.0
Dissolved oxygen	1.2 mg/L	3.1 mg/L
Total nitrogen	110.6 mg/L	–
Ammonium nitrogen	47.2 mg/L	–
Total phosphate	37.4 mg/L	–
Phosphate phosphorus	34.8 mg/L	–
Turbidity	1744 NTU	–

Italy). The AOP phase (second phase) was conducted in the EF basin, which contained four iron plates as the cathode and anode (dimensions of 5.0 cm × 5.0 cm × 0.5 cm). The gap between the electrode plates was 5 cm, and a power supply (0–30 V, 0–10 A) was used to adjust the current density. H_2O_2 (30% w/w) solution was added at the beginning of the EF process. The effluent was then passed to the biological basin using a flow head pump. As depicted in Fig. 1, the biological basin consisted of an SBR basin, a feeding system (peristaltic pump), and an aeration system (air pump and four air stones). The aeration system was used to inject air in the form of fine bubbles and to obtain uniform mixing to maintain the DO level at 2 mg/L [18] and decant the system.

All phases in the system were controlled by programmable timers. In the preliminary experimental period, the efficiency of the EF process was evaluated by measuring process parameters such as the H_2O_2 concentration, current density, AOP time (t_A), and pH of the solution. The aeration time in the SBR basin (t_R) was optimized to achieve maximum COD, TN, and TP removal. However, in the EFSBR experiments, according to the results of the preliminary period, some other parameters were kept constant. A compilation of the EFSBR parameters is provided in Table 2. The operating parameters of t_A, HRT, SRT, SVI, and MLSS were optimized to maximize the performance of the EFSBR system.

2.3. Experimental procedure

The reactor was initially inoculated with AS bacteria, i.e., 2.0 cm^3 of the DIW AS. The aeration and reaction phases were then operated in circulation for 15 d until the EFSBR reached steady state. The average time to reach steady state was 3 d–7 d of operation without changes (6% as the criterion) in the system operation conditions with respect to the effluent COD (COD_{eff}) [43]. To control the SRT and MLSS in the EFSBR reactor, a specified MLSS concentration was removed to maintain a stable SRT at the end of every reaction phase of the SBR.

2.4. Analytical methods

The studied parameters included COD, MLSS, mixed liquor volatile suspended solids, TN, NO_3^--N, TP, and PO_4^{3-}-P using the 5220A, 4500D, 2710D, 4500-N C, 4500-NO3-B, 4500-PE, and 4500-PE methods, respectively. These measurements were performed according to the standard methods [1]. Parameters such as the turbidity, temperature, DO, and pH of the solution were monitored online using instruments provided by Hanna Instruments, Italy. Prior to analysis, the collected samples were filtered using a membrane filter (0.45 μm, Whatman). The dissolved iron ion content was measured using the 1,10-phenanthroline method [1]. To maintain the pH in the range from 6.5 to 8.0, sodium bicarbonate was used as a buffer. Evaluation of each operating cycle was performed on the basis of the parameter removal efficiency of TN, TP, and COD. The removal efficiency (R) (%) was calculated according to Eq. (1).

$$R\ (\%) = \left(\frac{[Input] - [Output]}{[Input]}\right) \cdot 100 \tag{1}$$

3. Results and discussion

3.1. SBR performance

To understand the importance of the combined EFSBR process, separate treatment processes were initially conducted for the SBR and EF. The results revealed that the SBR process alone suffered from major limitations for the treatment of WW with a high load of pollutants because it requires high aeration and energy consumption. The SBR process is composed of a series of phases, including

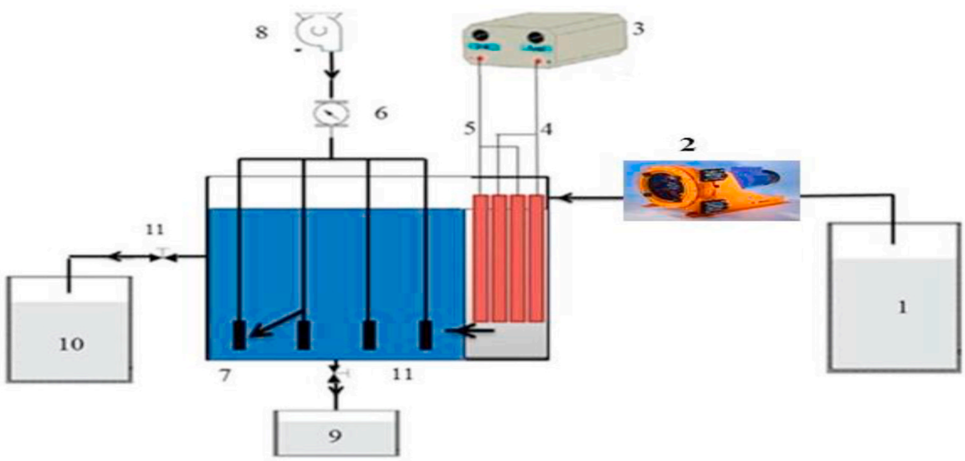

Fig. 1. Schematic diagram of the electro-Fenton-sequencing batch reactor.

Table 2
Operating conditions of the electro-Fenton-sequencing batch reactor system.

Parameters	Range	Parameters	Range
Temperature	28 ± 2	Filling time (t_F)	30 min
pH	6.5–7.5	AOP time (t_A)	45 min
DO	1 ± 0.2 mg/L	Reaction time (t_R)	2–18 h
Organic loading rate	0.245 ± 0.02 [kg COD/(m^3·d)]	Settling time (t_S)	90 min
COD/N/P	100/5/1	Draw time (t_D)	15 min
Food/microorganism ratio	0.06 ± 0.03 [kg BOD/(kg VSS·d)]	Idle time (t_I)	15 min

basin filling, reaction, settling, and drawing, with a defined time. Fig. 2 shows a comparison of the COD removal efficiency from dairy WW for the different systems used in this study. According to similar previous studies, an HRT of 24 h with a filling time (t_F) of 1 h and an anoxic phase of 1 h after the filling phase were reported to be sufficient for dairy WW treatment [19,24,31,35,40,50]. The COD and TN removal efficiencies were 45.7% and 36.7%, respectively, under the optimum conditions.

3.2. Advanced oxidation process performance of the EF process

The removal efficiency of the EF process depends on several important factors such as the pH, H_2O_2 concentration, iron concentration, H_2O_2/Fe ratio, and initial pollutant concentration. The pH value, which affects the formation of $^{\bullet}OH$, is a key parameter in Fenton-based AOPs [4]. Fig. 3a shows that the removal rates of COD, TN, and TP at acidic and neutral pH values were much greater than those at alkaline pH values, which is typical in Fenton-type processes. A maximum removal rate of 78% was obtained at a pH of 5.5. In general, the generation of $^{\bullet}OH$ in the Fenton process mainly occurs in an acidic medium. However, the formation of $(Fe(II)(H_2O))^{2+}$ occurs when the pH is less than 5. It slowly reacts with H_2O_2, thereby resulting in the formation of less reactive oxygen species [9]. Furthermore, it causes a considerable decrease in the iron concentration in the system responsible for H_2O_2 activation, thereby lowering the formation of $^{\bullet}OH$. Moreover, H_2O_2 can attach to the H^+ ions to form $H_3O_2^+$ (Eq. (2)), which reduces the amount of oxidant available for reaction with ferrous ions [12–14].

$$H_2O_2 + H^+ \rightarrow H_3O_2^+ \tag{2}$$

The oxidative potential of $^{\bullet}OH$ decreases as the pH increases. Thus, as the pH increases, the removal efficiency decreases. The EF process mainly follows first-order kinetics; thus, it increases the effectiveness of the process by increasing the amount of oxidants added to the system. Fig. 3a reveals that the best COD removal efficiency of the EF process was obtained at a pH of 4. However, only a minor decrease in the effectiveness was reported in the pH range from 4.5 to 7.0. For pH values above 7, a significant decrease in removal effectiveness was reported. As the pH increased from 4.5 to 7.0, the COD removal efficiency decreased by only 5%. Thus, a pH of 7 was selected for further experiments. Additional costs and operations are needed to neutralize the effluent prior to its discharge into the environment. At an alkaline pH, Fe^{2+} forms $Fe(OH)_3$ precipitates in the system, which affect the catalytic cycle; thus, a sufficient amount of catalyst will not remain in the reaction [20]. Because iron ions tend to form unreactive hydroxoferric complexes at a pH of greater than 7, a neutral pH is considered optimal for the treatment process.

The dose of H_2O_2 plays a vital role in the oxidation process as a source of $^{\bullet}OH$ (Eq. (3)) [44]. The oxidation rate strongly depends on the amount of consumed H_2O_2 [9]. To examine the effect of the H_2O_2 concentration on the efficiency of the EF process, an H_2O_2 concentration of 0.2 mM–2.0 mM was considered for the experiments. As shown in Fig. 3b, by increasing the concentration of H_2O_2 from 0.2 mM to 1.0 mM under optimal conditions (pH of 7, current density of 2 mA/cm^2, and time of 45 min), the COD removal efficiency was increased to 78%. Further enhancement of the oxidant concentration is not effective at obtaining higher degradation,

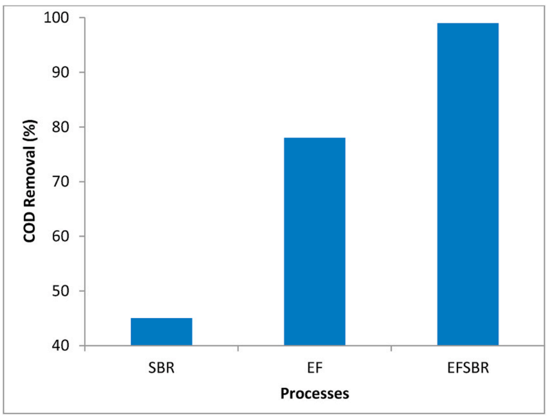

Fig. 2. Removal efficiencies of different dairy wastewater treatment processes.

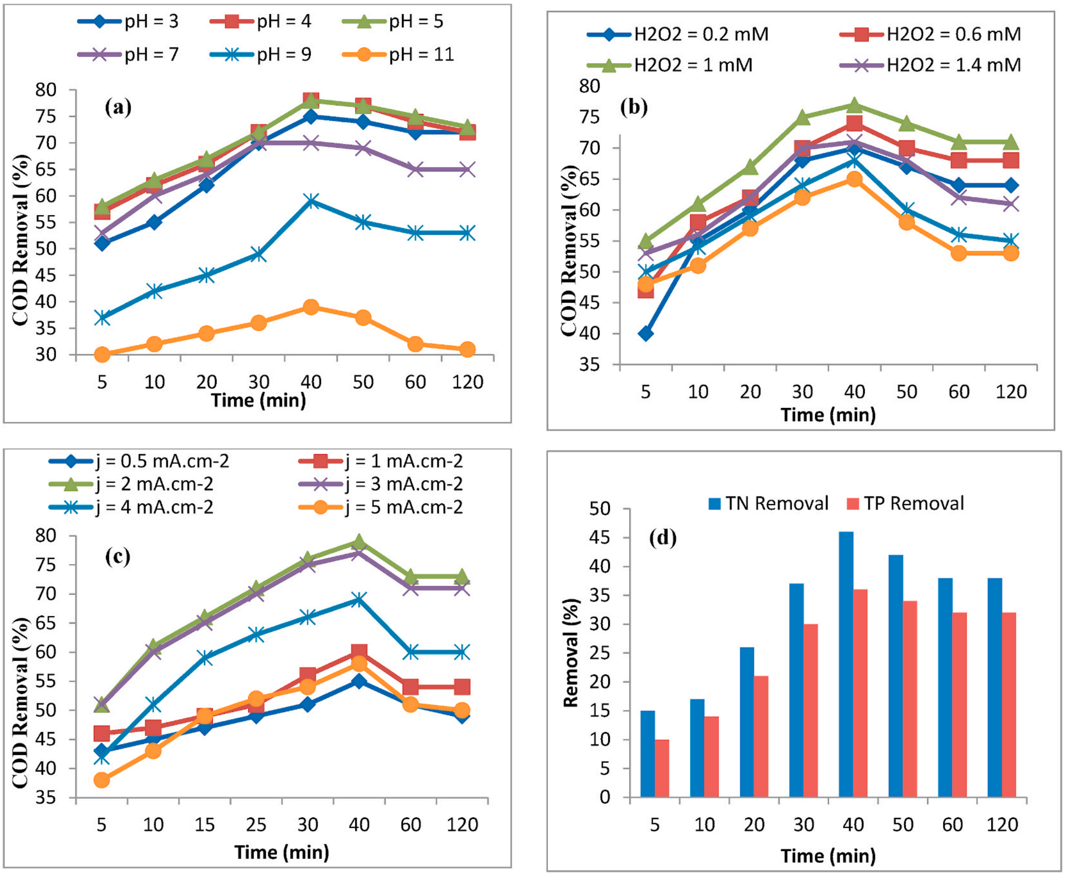

Fig. 3. Optimization of the electro-Fenton process with respect to (a) pH, (b) H2O2 concentration, (c) current density, and (d) total nitrogen (TN) and total phosphorus (TP) removal (pH of 7, H2O2 concentration of 1 mM, current density of 2 mA/cm2, and time of 45 min).

whereas higher concentrations of H_2O_2 can have a scavenging effect with respect to $^{\bullet}OH$ [48]. H_2O_2 can react with $^{\bullet}OH$, thereby causing the formation of less reactive radical species (Eq. (4)) [4,9]. Finally, residual H_2O_2 increases the COD concentration and is harmful to microorganisms that are responsible for degradation in biological processes [10].

$$H_2O_2 \rightarrow {}^{\bullet}OH + {}^{\bullet}OH \tag{3}$$

$$H_2O_2 + {}^{\bullet}OH \rightarrow H_2O + HO_2^{\bullet} \tag{4}$$

Another important parameter in EF is the current density [29]. In this study, the current density was optimized in the range of 0.5–5.0 mA/cm^2 with respect to the removal rate of COD, as presented in Fig. 3c. These experiments revealed that with an increase in current density to 2 mA/cm^2, the COD removal efficiency increased, whereas further increasing the current density had an insignificant effect on the removal efficiency. Because the highest removal rate was obtained at a current density of 2 mA/cm^2, this value was used for further experiments.

The mass of the sacrificed electrode in the EF reaction was 0.25 mg/m^3, which can be calculated from Faraday's electrolysis equation (related to the volume of the effluent in m^3) as follows (Eq. (5)):

$$W = \frac{I \cdot t \cdot M_W}{Z \cdot F} \tag{5}$$

where W is the mass of dissolved iron (g), I is the current intensity (A), t is the reaction time (s), M_W is the molecular weight of iron (M = 55.85 g/mol), Z is the number of electrons involved in the redox reactions ($Z = 2$), and F is Faraday's constant (96.500 C/mol). The sludge formation under optimal conditions was approximately 2 g/m^3 of WW. Thus, the EF process is a technology that minimizes the addition of chemicals and sludge formation. The formed ferrous iron cation (+2) reacted with H_2O_2 to form $^{\bullet}OH$ (Eq. (6)). To exclusively cause the activation of H_2O_2 to form $^{\bullet}OH$, the amount of Fe^{2+} should be "catalytic." An increase in the ferrous ion concentration above the optimal concentration is associated with reduced COD removal [49]. Moreover, excess iron can cause a secondary reaction of the formed Fe^{3+} with H_2O_2 (Eqs. (6) and (7)). Thus, non-optimal iron doses lead to high treatment costs and high sludge production (Mahvi et al., 2011).

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + HO^- + {}^{\bullet}OH \quad (6)$$

$$Fe^{3+} + H_2O_2 \rightarrow Fe\,(OOH)^{2+} + H^+ \quad (7)$$

The release of iron ions from the electrode into the WW requires a specific voltage (Eq. (8)) (Murillo-Sierra et al., 2018).

$$E = \frac{I \cdot v \cdot t}{\Delta COD \cdot V} \quad (8)$$

where E is the energy consumption (kWh/kg COD), I is the operating current intensity (kW), v is the voltage (V), V is the volume of the solution in the EFSBR (L), t is the reaction time (h), and ΔCOD is the COD removal (kg/L). Under optimum conditions, the energy consumption increased from 0.0647 KWh/kg COD to 1.6720 KWh/kg COD and the current density increased from 0.5 mA/cm^2 to 5.0 mA/cm^2. Thus, increasing the current density resulted in an increase in the amount of oxidized iron and enhanced energy consumption. An increase in the current density from 2 mA/cm^2 to 3 mA/cm^2 improved the degradation effectiveness by only 3% for COD, whereas the energy consumption was increased by 20%. Thus, a current density of 2 mA/cm^2 was selected as the optimum value.

To determine the best TN and TP removal efficiencies via the EF process under optimum conditions, experiments were conducted for 5–120 min (Fig. 3d). Fig. 3d indicates that the highest TN and TP removal rates were 46% and 37%, respectively, after 45 min of treatment. In the EF process, the removal efficiencies of TN and TP were lower than the Environmental Protection Agency (EPA) standard for effluent discharge into the environment [49,53,54]. These results can be attributed to acidic conditions (lack of chemical precipitation) and the inability to decompose nitrogen and phosphate compounds into simpler compounds [55].

3.3. Optimization of the EFSBR system

3.3.1. Effect of hydraulic parameters

The abovementioned results clearly indicate that the application of the combined EF and SBR process is favorable in terms of effectiveness. To evaluate t_R, the simultaneous removal of COD, TN, and TP was performed for 2–18 h according to the operating conditions listed in Table 2. The simultaneous removal rates of COD, TN, and TP in the EFSBR system during 6 h of treatment exceeded 95% (Fig. 4a). The removal efficiency for t_R of 6 h and 8 h did not significantly affect the treatment effectiveness. When t_R was greater than 8 h, the changes in the effluent quality and removal efficiency were not significant. Therefore, t_R of 6 h is optimal in terms of COD, TN, and TP removal with respect to the treatment cost. A comparison of the obtained results with those of a conventional SBR system [24] confirmed the significant improvement of the developed process. The EFSBR system can reduce the costs via reduced t_R and provide a higher treatment efficiency. To investigate the effect of the HRT on the COD, TN, and TP removal efficiencies, water samples at the end of each cycle of 9.25 h–26.75 h were analyzed. The data compared in Fig. 4b are presented as the COD removal efficiency as a function of HRT. During the experiment, the COD removal efficiency increased from 91% to 98% when the HRT was prolonged from 9.25 h to 10.00 h. No significant increase in COD removal efficiency was observed after 10 h of HRT. The ability of the system to provide greater removal with an increased HRT ensures the flexibility of the treatment system. Moreover, it is an effective method for nitrification control owing to the limited doubling time of ammonium-oxidizing bacteria in comparison with that of nitrite-oxidizing bacteria. This phenomenon enhances the capability of the bioreactor to receive WW with higher ammonium concentrations [2,7].

3.3.2. Effects of microbiological parameters

Analysis of the SVI is the best-known approach for quantifying and qualifying microalgal biomass and sludge bulk settling properties. This parameter was used to describe the sludge properties in the EFSBR system (Eq. (9)). V in Eq. (9) represents the settled

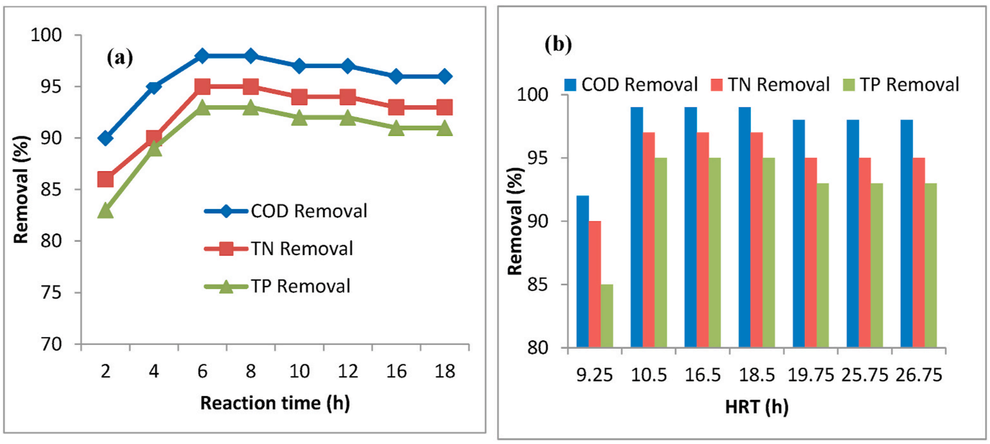

Fig. 4. Effects of the (a) reaction time and (b) hydraulic retention time (HRT) on the chemical oxygen demand (COD), total nitrogen (TN), and total phosphorus (TP) removal efficiencies.

sludge volume (mL/L) obtained in 30 min. SVI values in the range of 100–200 mL/g indicate good settling ability that leads to microalgal aggregates and increased bioflocs. Determination of the optimum MLSS concentration is required to maintain a favorable food/microorganism (F/M) ratio [18].

$$SVI = \frac{1000 \cdot V}{MLSS} \tag{9}$$

Fig. 5a presents the effects of the initial COD and MLSS concentrations on the SVI under the optimum treatment conditions. During the first 15 d, performance fluctuations related to microbial acclimation under the applied experimental conditions were observed. The SVI gradually decreased with the generation of granules and reached 89 mL/g at the end of the cycle. In the EFSBR system, the matured granules were completely settled in 90 min [settling time (t_S) = 90 min]; thus, the supernatant was discharged from the reactor because the effluent was clear. During the initial operation phase in the EFSBR system, the MLSS concentration was less than 1850 mg/L. After the formation of granular sludge, the MLSS concentration increased from 1850 mg/L to 6300 mg/L. During the experiments, as the amount of sludge granules increased, the MLSS concentration decreased [25]. The outgrowth of floc-forming bacteria on the granule sludge caused appropriate settleability and subsequently provided biomass maintenance, as indicated by the increase in the MLSS concentration [19]. The system with a constant MLSS concentration (3500 mg/L), SRT of 15 d, and HRT of 10 h exhibited stable performance as the COD concentration of the influent increased, thereby confirming its applicability in routine operations in which fluctuations in the WW parameters at the inlet of the treatment plant are observed. Furthermore, an increase in the MLSS concentration decreased the SVI; when the MLSS concentration increased from 1800 mg/L to 3500 mg/L, the SVI increased from 46 to 89, respectively. In the EFSBR system, the SVI values were in the range of 80–100 mg/L, thereby indicating that the granular sludge had great settling ability. Fig. 5a indicates that an increase in the initial COD concentration did not significantly affect the SVI. HRT is an efficient operating parameter in biological treatment processes. It can regulate the physiological and microbial acclimation of the bacterial culture, such as the growth phase (F/M ratio), thereby resulting in the stabilization of the system performance. The decrease in the HRT at a constant initial COD concentration (3225 mg/L) led to a decrease in the SVI.

As shown in Fig. 5b, the change in the SRT had a significant effect on the COD removal efficiency. Under the operating conditions, the COD removal efficiency at an SRT of 7 d increased from 75% to 90%, and that at an SRT of 10 d and 15 d was 93% and 98%,

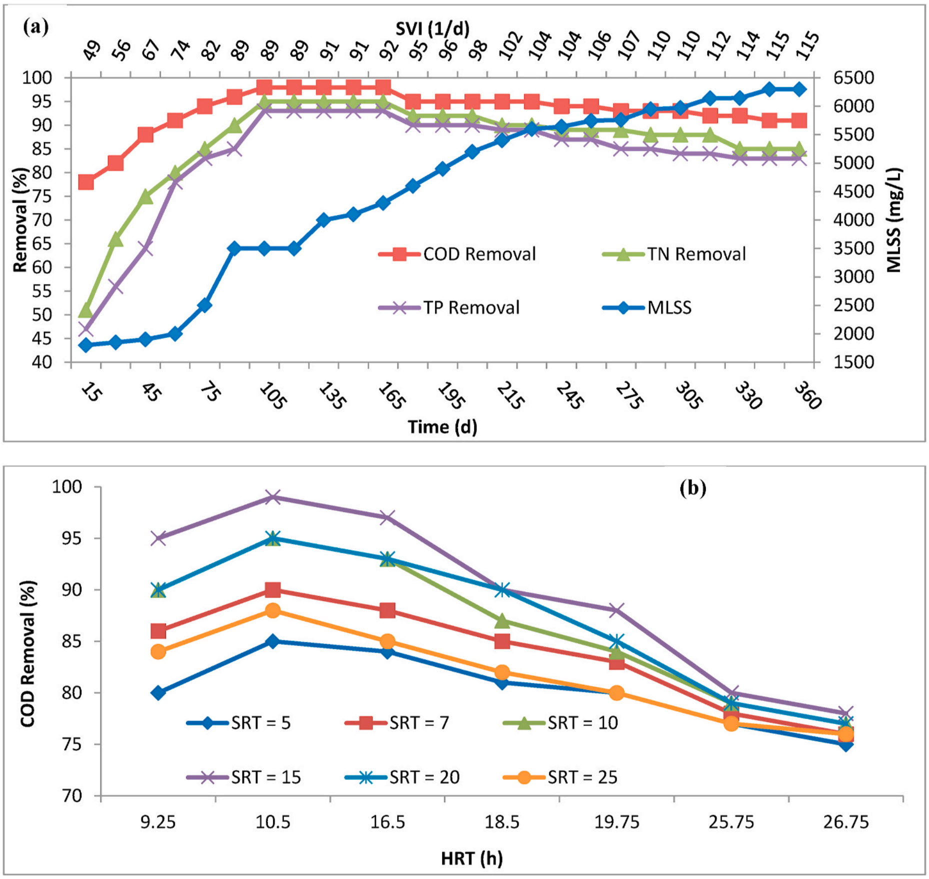

Fig. 5. Effects of the (a) sludge volume index (SVI), mixed liquor suspended solids (MLSS) concentration, and (b) sludge retention time (SRT) on the chemical oxygen demand (COD), total nitrogen (TN), and total phosphorus (TP) removal efficiencies.

respectively. The highest COD removal efficiency was achieved at an SRT of 15 d. Fig. 5b shows that the efficiency of the EFSBR system decreased at an SRT of greater than 20 d owing to the growth of filamentous microorganisms and sludge bulking and at an SRT of less than 15 d owing to the growth of pinpoint flocs. At a lower SRT, floc-forming bacteria showed better growth than filamentous bacteria. The COD removal efficiencies for SRTs of 15 d and 20 d were almost the same. Because it is easier to operate the system at a low SRT, an SRT of 15 d was chosen as the optimum value. Under these conditions (SRT of 15 d), the COD_{eff} decreased from 3225 mg/L to 45 mg/L.

After selecting the optimum conditions of the EFSBR system, its performance was evaluated from a longer time perspective. The nutrient removal mechanism during a 1 y (365 d) study under stable operation conditions was monitored. The average COD, TN, and TP removal efficiencies were 98.4%, 95.2%, and 93.3%, respectively. In this study, the organic loading rate (OLR) was constant [0.245 kg COD/(L·d)]. In addition, the lowest number of adapted bacteria (4.92×10^2 MPN/100 mL) was observed at 0–15 d and the highest number of adapted bacteria (3.10×10^7 MPN/100 mL) was reported on the 95th day.

3.3.3. TN removal mechanism by the EFSBR process

The biological nitrogen removal process involves nitrification processes, i.e., conversion of ammonia into nitrites and nitrates (NH_4^+ to NO_2^- and NO_2^- to NO_3^-), and denitrification processes that transform NO_3 into N_2 [8,21]. In this study, the COD removal efficiency increased in the first 95 d period to 98.45%, which followed the acclimatization of the bacteria to the operation conditions. After 165 d, owing to the increased OLR and the assimilation of nitrogen, which inhibited the bacterial activity (although the bacteria were acclimated to the operating conditions), the change in the removal efficiency was insignificant because nitrification was conducted completely and quickly without accumulation or inhibition during this period. Thus, the population of nitrifying bacteria grew well in the presence of organic matter (COD) [20,22,25].

Because the density of bacteria depends on the SRT of the EFSBR system [31], this parameter was also monitored. As shown in Fig. 5b, the highest density of nitrifying bacteria was observed at an SRT of 15 d; thus, as the SRT decreased, the nitrifying bacteria population decreased. In this study, the removal efficiency of t_R increased because of the extra phase (t_A) before t_R, thereby resulting in decreased competition for organic matter uptake between microorganisms (heterotrophic and nitrifying bacteria). This helped to increase the number of nitrifying bacteria and the removal of ammonia nitrogen [35]. Fig. 5b shows that after the 95th cycle (when the population of denitrifying bacteria was the highest), the NO_3^- concentration in the effluent decreased. In the t_S stage, owing to the presence of residual NO_3^--N in the previous stage (t_R) and the rapid growth of denitrification bacteria in this stage, NO_3^- was transformed into N_2. Therefore, t_S ensured complete nitrogen removal from the effluent. The residual TN concentration in the effluent of the EFSBR was lower than the EPA standard (3 mg/L) [41,43]. According to Eq. (10), the presence of $^{\bullet}OH$ in the biological process increased the DO concentration. The DO concentration in WW increased rapidly and was mainly available for the external respiration of bacteria. The DO concentration is a crucial factor for the efficiency of aerobic WW treatment processes. In addition, the addition of H_2O_2 to the biological reactor can be effective for the generation of peroxidase enzymes by bacteria, which allows the decomposition of organic compounds.

$$HOO^{\bullet} + {}^{\bullet}OH \rightarrow H_2O + O_2 \tag{10}$$

3.3.4. TP removal mechanism by the EFSBR process

This study indicates that the EFSBR is a promising approach for TP reduction (>93%) (Fig. 4b). The phosphorus removal performance was less than 50% in the initial 15 d, but the TP removal efficiency increased to more than 80% on the 50th day. The TP removal efficiency is affected by the competition between phosphorous accumulating organisms (PAOs) and glycogen accumulating organisms (GAOs) because a high C/P ratio favors the growth of PAOs over GAOs and can lead to endogenous denitrification [35,43]. Fig. 4d confirms the presence of denitrifying phosphate accumulating organisms (DPAOs) in the EFSBR system. Conventionally, DPAOs are responsible for the removal of phosphorus in biological processes, which are capable of nitrogen and phosphorus uptake via NO_2^--N and NO_3^--N as electron acceptors. DPAOs can reduce carbon sources (50%), poly hydroxyl alkanets, glycogen, and energy (30%) [43,50]. Throughout the experiments, the pH was maintained at less than 7 to achieve good microorganism growth in the reactor and optimum operating conditions; therefore, phosphorus removal was only slightly attributed to chemical precipitation. Comparison of the SBR and EFSBR systems indicated that the removal efficiency of the SBR system was lower than the standard at a high OLR without pre-treatment, and it could not fulfill the expectations of treatment effectiveness. However, the TN, TP, and COD removal efficiencies in the EFSBR system at lower HRT, SRT, and energy consumption (owing to lower t_R) were 99%, 97%, and 95%, respectively, and it was found that the use of t_A as one of the phases of EFSBR promoted the conversion of complex compounds present in the WW with a high OLR to simple biodegradable organic compounds. In addition, the t_A phase caused NH_4^+-N to be oxidized to NO_3^--N and organic phosphorus to be oxidized orthophosphate, thereby providing the conditions for the simultaneous growth of nitrogen-phosphorus removing bacteria [53]. The performance comparison of the EF (as a pre-treatment unit) and SBR processes alone to the EFSBR process clearly indicated the advantages of the combined approach in terms of most of the studied parameters. The system based on separate processes had higher operating and construction costs (e.g., blower, pump, and land) and a higher SRT, HRT, and t_R. Therefore, in the combined system, the COD:TN ratio was maintained at above 10:1 and enhanced the simultaneous removal of TN and TP [35]. In addition, the EFSBR system operated in fully continuous mode owing to the existence of two separate basins; the first basin included the filling and AOP phases, and the biological basin included the settling and draw phases [52].

3.4. Kinetics of the EFSBR

A kinetic model was developed for COD removal at different initial COD concentrations with an OLR of 0.245 kg COD/(m^3·d) with the operating conditions of the EFSBR system for steady state [51], which is expressed as follows:

$$\frac{X_t}{(S_0 - S)} = \frac{K_s}{k \cdot S} + \frac{1}{k} \tag{11}$$

The model equation (Eq. (11)) was used in the kinetic study, where X_t is the MLSS (mg/L) concentration at t_R (h), S_0 is the initial COD concentration (mg/L), S is the final COD concentration (mg/L), k is the rate constant of substrate degradation per unit mass of microorganisms (h^{-1}), and K_s is a half rate constant (mg/L). The k values of the SBR, EF, and EFSBR processes were 1.20, 3.50, and 4.94 h^{-1}, respectively, and the K_s values for the SBR, EF, and EFSBR processes were 1217.44, 5825.50, and 5984.90 mg/L, respectively. The results were in good agreement with the model for data obtained under the EFSBR process. In addition, the rate constant value calculated from the model indicated that an EFSBR system with a small volume can achieve the desired removal efficiency for dairy WW treatment to reach the discharge standards. Besides the listed advantages of the EFSBR system, on the basis of the rate constant, a synergism (*SK*) of 1.05 for the combined process was calculated (Eq. (12)).

$$SK = k_{EFSBR}/(k_{EF} + k_{SBR}) \tag{12}$$

The comparison of similar studies for dairy WW treatment showed that the EFSBR system has a higher efficiency, lower treatment cost, and lower chemical consumption compared with other hetero EF processes (Table 3). The combined Fenton-SBR process, when compared with biological treatment alone and AOP alone, had a synergistic effect. It followed the generation of more biodegradable by-products during Fenton pretreatment, which were further effectively degraded by microorganisms. Overall, this combined approach exhibited synergism, thereby providing enhanced TN and TP removal. The results confirmed that the combined EFSBR process used for industrial WW treatment reduces the pollution load to an acceptable level, thereby allowing the discharge of effluent into the environment.

Table 3
Comparison of dairy wastewater treatment processes.

Unit process	HRT (h)	COD loading rate [kg/(m^3·d)]	COD removal	TN removal	TP removal	Ref.
UASB reactor	12	2.4–13.5	90–92%	<80%	<50%	[34]
SBR	<80	2	91–97%	75%	<30%	[28]
Activated sludge	>100	1	97%	<30%	<20%	[47]
CSTR single-phase	>100	1	95%	<50%	<30%	[37]
Aerobic biological system	>100	1	96%	<50%	<30%	[6]
Pre-coagulated with $FeSO_4$	20	0.95	95%	<30%	<20%	[33]
Activated sludge with pre-electrocoagulation	<50		97%	65%	85%	[2]
EFSBR	10		98%	98%	98%	This study

4. Conclusion

In this study, the treatment of DIW was performed by an EFSBR, which involved the simultaneous reaction of chemical treatment via AOPs (EF) and biodegradation (SBR). This study reveals that the EFSBR system can achieve higher effectiveness in the mineralization of biologically persistent compounds compared with other available technologies. In addition, this hybrid process eliminates the weakness of aerobic biological systems, such as the inability to treat industrial WW with a high organic load, and reduces the operational costs. This aspect makes the development of a hybrid process advantageous to benchmark technologies based on physiochemical processes combined with the biological treatment stage. It provides operational flexibility based on the ability to effectively degrade all types of impurities present in dairy effluent. An HRT of 10 h, SRT of 15 d, MLSS concentration of 3500 mg/L, and SVI of 89 were found to be suitable for the treatment of DIW in the EFSBR system. Under the optimum conditions of the EFSBR, the simultaneous COD, TN, and TP removal efficiencies were greater than 95%. Therefore, the EFSBR not only allows the reduction of the HRT, SRT, and t_R, but also increases the treatment efficiency compared with the sole use of biological or EF treatment. Therefore, the EFSBR is a promising treatment system for the removal of organics, nitrogen, and phosphate compounds present in food industry WW. The experimental period used in this study ensures the ability of implementing the developed process in industrial practices.

CRediT authorship contribution statement

Evaluation and start-up of an extra-stage Sequential Batch Reactor with Electro-Fenton (EFSBR) for dairy wastewater treatment. Mohammad Reza Heidari: Investigation, Sampling, Formal analysis, Writing - Original Draft, Conceptualization, Methodology, Validation, Data Curation. Mohammad Malakootian: Conceptualization, Methodology, Validation, Investigation, Formal analysis, Writing - Original Draft, Writing - Review & Editing, Supervision, Project administration. Xun Sun: Formal analysis, Validation, Writing - Review & Editing. Yang Tao: Formal analysis, Validation, Writing - Review & Editing. Grzegorz Boczkaj: Conceptualization, Methodology, Validation, Formal analysis, Data Curation, Writing - Original Draft, Writing - Review & Editing. Shirish H. Sonawane:

Writing - Review & Editing. Hakimeh Mehdizadeh: Conceptualization, Methodology, Validation, Investigation, Formal analysis, Writing - Original Draft, Writing - Review & Editing, Supervision, Project administration.

Declaration of competing 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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