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GRANULAR SEQUENCING BATCH REACTOR AND PHOTO-FENTON PROCESSES FOR TREATMENT OF CATTLE MANURE WASTEWATER

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ABSTRACT

In this study, the photo-Fenton process was followed an aerobic granular sequencing batch reactor (SBR). The combined system was used for cattle manure wastewater treatment. In biological stage, the granulated SBR was resistant against feeding shocks, when imposed by substrate with food to microorganisms (F/M) ratio of 1.6. The bioreactor was operated at three different HRTs. Kinetic studies showed the most efficient performance was HRT of 32 h, rather than 24 and 48 h. Fenton and photo-Fenton (ph-F) reactions were individually performed as a polishing stage. The optimized ratios of H_2O_2/COD (1.5:1) and Fe^{2+}/H_2O_2 (1:10 M/M) were defined. In ph-F reactions, the impacts of UV-C and solar lights were evaluated, where the chemical oxygen demand (COD) removal efficiencies noticeably enhanced to 82.8 and 85.3%, respectively. The best COD, total Kjeldahl nitrogen (TKN) and total phosphorus (TP) removals for the combined SBR and ph-F systems were 97, 98.5 and 99%, respectively. For the first time, the feasibility of combined aerobic granular SBR and ph-F process for cattle manure wastewater treatment and recycling water were successfully investigated.

INTRODUCTION

Agro-industrial wastewater treatment (WWT) processes usually have a common problem for treating high concentrated wastes. Biological WWT techniques are widely recommended because they are the most cost-effective and environment-friendly methods than known physical and chemical WWT options (Prabhu, et al., 2017). Aerobic granular system is a novel biological treatment in which all aerobic, anaerobic and anoxic vessels compacted in a dense aggregate known as aerobic biogranule. The aerobic granulation is usually performed in sequencing batch reactor (SBR) because of its unique characteristic; since SBR's cycle consists of five sequential stages: fill, react, settle, decant and idle (Figueroa, et al., 2011, Sarvajith, et al., 2018). Aerobic granular SBRs are efficiently capable of eliminating nutriants from low-strength wastewater (He, et al., 2018). However, they will not solely achieve the target of recycling water in the least retention time. In order to furnish an economically and technically feasible WWT system, appropriate methods should be integrated into aerobic SBRs.

Recently, combining a biological system with an advanced oxidation process (AOP) is attracted great interests for WWT (Abedinzadeh, et al., 2018, Hu, et al., 2018). However, very few articles had been published until now about the combination of aerobic granular SBR and different treatment units for specifically treatment of waste water (Tu, et al., 2010, Winkler, et al., 2012). The AOPs are usually recommended for wastewater pre- and post-treatment. They are able to mineralize recalcitrant substances and treat highly concentrated and toxic wastewaters through oxidation reactions (Ganzenko, et al., 2018). The principle reactions in AOPs are generation and utilization of free radicals of hydroxyl ($OH^\bullet$) as an energy-maker. The main sources of these energetic radicals are hydrogen peroxide (H_2O_2), chlorine, ozone, etc. Among AOPs, Fenton and photo-Fenton (ph-F) reactions are considered as simple, efficient and fast methods capable to oxidize COD, nitrogen, phosphorus and turbidity from contaminated wastewater (Abedinzadeh, et al., 2018, Kitsiou, et al., 2014). Fenton based processes have been widely used for treating different wastewater such as agro–industrial (Ahmed, et al., 2011) and pharmaceutical (Ganzenko, et al., 2018) waste water; because Fenton reaction is short and efficient. Additionally, it is simply operated at room temperature and atmospheric pressure.

The base of Fenton reactions stand on the potent reaction between H_2O_2 and Fe^{2+} which is resulted in the generation of $OH^\bullet$ (E.1) and regeneration of Fe^{2+} in Fenton-like reaction (E.2) (Lee and Shoda 2008). Concurrently, photo assisted Fenton can minimize the microbial community of the effluent as UV emissions are able to partly deactivate the remained bacteria, fungi, etc. (Gárcia-Fernández, et al., 2012, Pouran, et al., 2015). Moreover, the high energy radiation of UV lights regenerate Fe^{2+} ions and extend the oxidation reaction (Nousheen, et al., 2014, Pouran, et al., 2015). In fact, H_2O_2 is also broken down to hydroxyl radical and HO^- that release energy in the presence of iron ions. Then, the ions utilize the generated energy and make flocs with organic and inorganic compounds present in aqueous phase. The process proceeds until all of the energetic free radicals are utilized and some low-energy ones remained in the system (like $^{\bullet}O_2H$). It is widely emphasized that Fenton process efficiency could be influenced by some effective variables such as H_2O_2 and Fe^{2+} concentrations, pH, reaction time, etc. In order to obtain efficient process with minimized chemical demands, the variables must be optimized in the preliminary stages of WWT design (Metcalf et al., 2003).

$$H_2O_2 + Fe^{2+} \rightarrow Fe^{3+} + HO^- + HO^{\bullet} \quad \text{(E.1)}$$
$$H_2O_2 + Fe^{3+} \rightarrow Fe^{2+} + HO_2^{\bullet} + H^+ \quad \text{(E.2)}$$

In this paper, the feasibility aerobic granular SBR followed by a ph-F process was assessed for decontamination of cattle manure effluent. In the biological treatment stage, the best HRT was evaluated by calculating the kinetic variables of substrate utilization rate. In the chemical treatment stage, the best chemical ratios (H_2O_2/COD and Fe^{2+}/H_2O_2) were optimized. Also, the effect of illumination on COD reduction was investigated. Finally, the obtained results were compared with the discharge standard limitations for COD, nutrients and turbidity levels, legislated by the Department of Environment of Iran.

2. MATERIALS AND METHODS

2.1. Wastewater characterization: Two types of cow manure wastewater were utilized in this project. The raw leachate was adopted from a livestock plant around Babolsar, Mazandaran, Iran. It was a high strength wastewater in respect of COD and pollutants such as phosphorus and nitrogen. The wastewater was diluted and stored in a refrigerator at 4-5°C, it was used as a feed stock for SBR. The second type of wastewater was collected from SBR outlet. It was used as a feed for Fenton and ph-F processes. The characteristics of added substrates are shown in Table 1.

Table1. The characteristics of substrates

Parameter	Feed of SBR	Feed of Fenton and ph-F	Unit
Total COD	11000±5800	1100±100	mgO_2/L
Soluble COD	5700±3600	1000±85	mgO_2/L
TKN	1185±940	44±12	mgN/L
TP	76±51	0.6±0.3	mgP/L
TSS	2500±2475	210±78	mg/L
Turbidity	-	7.04±0.5	NTU
pH	7.38±0.42	7.7±0.2	-

2.2. SBR set-up: The operating working volume of 1.5-L SBR was 1.2 L and H/D was 2.84. The SBR included five operating stages; fill (5 min), react (1410 min), settle (5 min), decant and idle (each was 10 min). The bioreactor was equipped with an aquarium pump for gentle aeration to keep dissolved oxygen (DO) level around 1 mg/L. Filling and decanting of the reactor were performed using a peristaltic pump and installed mid-valves, respectively. The volume exchange ratio (VER) was 50-75%. Granulation process was carried out aerobically in SBR. During the bio-granule formation process, the apparent structures of bio-aggregates were casually observed by optical microscope (Nikon, YS100) at several magnifications.

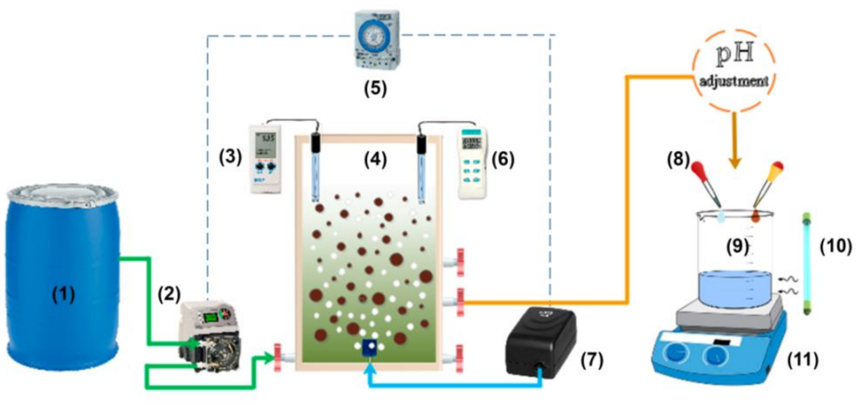


Figure 1. Schematic diagram of aerobic granular SBR and Fenton/ph-F as post-treatment stage. (1) Feed tank (2) peristaltic pump (3) pH-meter (4) SBR (5) timer (6) DO-meter (7) air pump (8) chemicals (9) Fenton vessel (10) light source (11) magnetic stirrer

2.3. Fenton and ph-F setup: The outlet stream of SBRwas collected for optimizing the variables of Fenton and ph-F processes. Its characteristics are summarized in Table 1. Since Fenton reaction is influenced by the concentration of employed chemicals, the H_2O_2/COD_{in} and H_2O_2/Fe^{2+} ratios were optimized. The prepared chemicalswere added into the reaction vessel after adjusting pH at 4. The pH was already optimized in our previous experiments (data not published). For 2 min, it was vigorously stirred by a magnetic stirrer (Velp, Italy). Then, the mixing was gently continued for

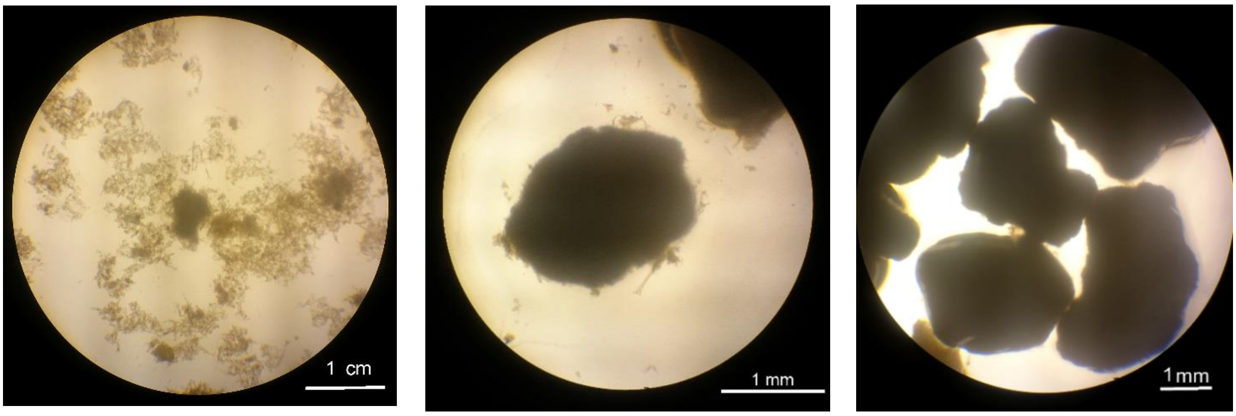
60 min in ambient temperature. In the first sets of experiment, the best H_2O_2 dosage that could lower COD was explored by the variation of H_2O_2/$COD_{initial}$ ratio 0.5:1, 1:1, 1.5:1, 2:1 and 2.5:1 at constant Fe^{2+}/H_2O_2 =1:20 (mol./mol.) and pH=4. In the second sets of experiment, Fe^{2+}/H_2O_2 ratio was changed in the range of 1:20, 1:10, 1:4, 1:2 and 1:1 to optimize the Fe^{2+} concentration while the optimized H_2O_2/COD_{in} and other parameters were kept constant. In ph-F experiments, the treatment was carried out in the presence of UV-C light (8 W, Philips, Poland) which was vertically installed beside the column with the distance of 3 cm. Samples were taken and neutralized for the next analysis. The samples were filtered through a 0.45 µm membrane-syringe filter to determine COD and Fe concentrations. But they were not filtered for turbidity measurements.

2.4. Analytical methods

The concentrations of COD, TP, TKN and total suspended solids (TSS) were measured according to APHA Standard Methods (APHA, 2005). Turbidity and pH were manually measured by HANNA instruments. The concentration of iron was detected by atomic absorption (Thermo Electron, S Series, USA).

3. RESULTS AND DISCUSSION

3.1. Granulation in SBR: The formation of aerobic granules in the SBR was monitored using optical microscope and the exhibited biogranules are shown in Figure 2. Fluffy flocs with irregular structure appeared just a week after inoculating the anaerobic seed sludge. The capacity of COD and nutrient removal gradually enhanced as granulation progressed. After three weeks (cycle 60), small dark aerobic granules (less than 0.2 cm) were observed (Figure 2c). Usually, the trend of COD and ammonia removal had ascending behaviors in whole process. After 75 days (cycle 175), the small fluffy granules were virtually replaced by dense and compact aerobic granules. They had clearer outer shapes and larger diameters (more than 0.2cm). They had high performance for COD and nutrients removal as it shall be discussed in sections 3.1.1 and 3.1.2. They were not disintegrated when sudden change imposed to SBR by increasing the VER to 75%.



a) Seed culture b) Cycle 60 c) Cycle 175

Figure 2. The evolution of aerobic granules and maturation in the SBR within 175 cycles

3.1.1. Effect of F/M ratio: The ratio of food to microorganism was monitored during SBR process. Figure 3 indicates the variations of F/M, COD and NH_4^+-N removal efficiencies from run 30 to 190. In the primary runs, COD and NH_4^+-N removal efficiencies were around 50%, although aerobic granules were generated. One of the main reasons of the problem is F/M fluctuation. Because F/M increased over 1 (Run 48), the active biomass could not consume the injected substrate and some poor settled sludge was washed out. As a result, COD and NH_4^+-N removal efficiencies decreased in the next runs. High F/M ratios (over 1) werealso loaded to the SBR in cycles 120 and 155. As shown in Figure 3, the granular sludge of SBR was not able to resist the loading shock in cycle 120. Therefore, COD and NH_4^+-N removals sharply decreased to 30 and 20%, respectively. However, the aerobic granular SBR appropriately controlled the treatment process after it was shocked at cycle 155. This desirable outcome relates to the flexibility of aerobic granules facing such shocks since they had matured (Figure 2c). Thus, it contributed to the granular sludge resistance to the high F/M loaded into the SBR. The ammonium removal resumed the upward trend until reached the completely ammonium removal point. Although COD removal efficiency had minor fluctuations after the imposed shock, it stabilized at 79%, the highest COD removal efficiency during the biological treatment runs.

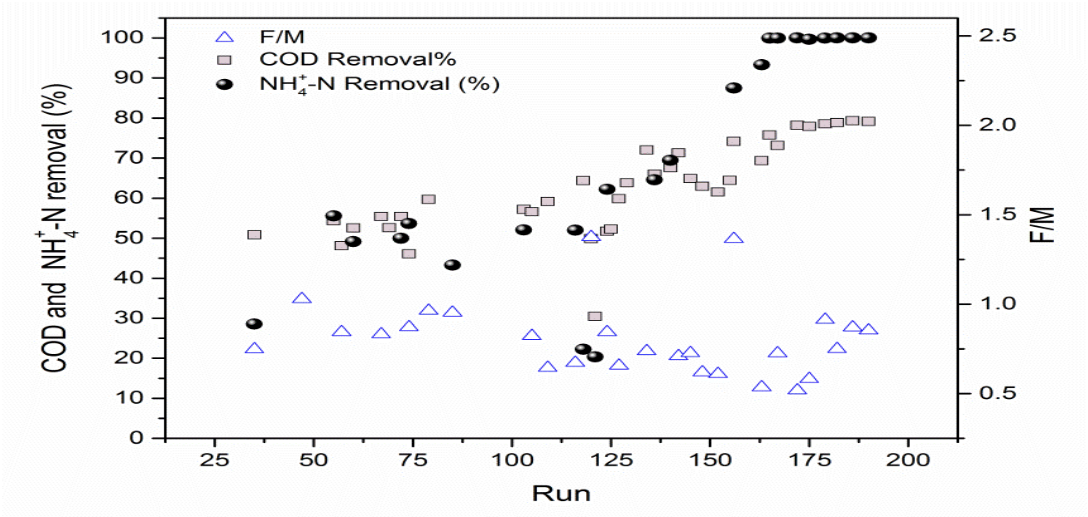


Figure 3: Long-term investigation of varied inlet F/M and COD and NH_{4+}-N removals in the SBR process.

3.1.2. Substrate utilization kinetics at various HRTs: The performance of SBR was investigated at 3 different HRTs. The SBR was operated at 24, 32 and 48 h hydraulic retention time. According to the literature, the following kinetic model of substrate utilization rate (U_s) has been utilized to validate the obtained results (Kushwaha, et al., 2013, Liu, 2007). The model correlates with solid retention time (SRT) as shown below:

$$\frac{1}{SRT} = Y.U_s - K_d \qquad \text{(E.3)}$$

where, Y and K_d are the observed growth yield and decay rate coefficient, respectively. A Monod-type equation was used in order to calculate U_s (see E.4). SRT was calculated by (E.5).

$$U_s = \frac{k.S_e}{K_s+S_e} = \frac{S_i-S_e}{X.t} \qquad \text{(E.4)}$$

$$SRT = \frac{1}{[0.6 \times \frac{S_i}{MLSS} \times \frac{S_i-S_e}{S_i}] - 0.1} \qquad \text{(E.5)}$$

where, K_s is the Monod constant, k is the maximum amount of U_s and X is the biomass concentration ($MLSS$). Also, S_i and S_e are regarded to the COD concentrations in influent and effluent. The depicted results in Figure 4, indicate that the best fitted correlation belongs toHRT of 32 h with R^2 of 0.999. Although the other HRTs (24 and 48 h) had proper R^2, they had higher K_d than HRT of 32 h. It means that sludge age should be shortened or much more amount of sludge has to be removed in the idle phase of SBR. The kinetic and experimental data of each HRT are tabulated in Table 2. The observed growth yields of HRT of 24 and 48 h were higher than 32 h. But it did not lead to significant COD removal efficiency during the operations. This could be due to the insignificant difference between Y and K_d values that imbalanced the growth, consumption and decay rates in those processes. According to the results, higher average COD removal efficiency was obtained at HRT of 32rather than the two others.

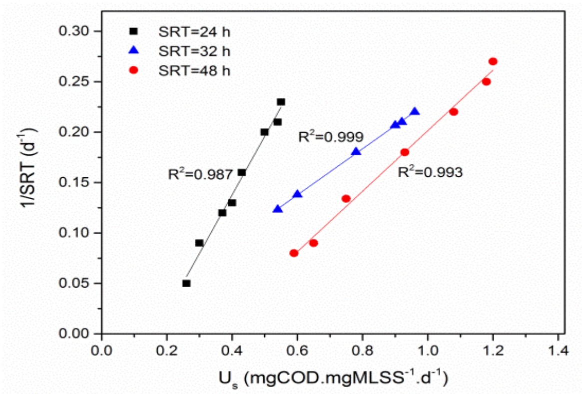


Figure 4. Kinetic behavior of the aerobic granular SBR at different HRTs.

Table 2. Calculated kinetic data for various HRTs.

HRT	Y	K_d	R^2	Average COD removal (%)
24	0.50	0.0940	0.984	59.5
32	0.229	0.0027	0.999	72
48	0.30	0.0987	0.991	65.5

3.2. Fenton and ph-F processes: The effluent of aerobic granular SBR was introduced into Fenton and ph-F reaction vessels. Since the generation of hydroxyl free radicals accelerates in acidic phase, the process performed at pH 4. The applied pH was previously optimized in our unpublished project.

3.2.1. **Optimization of H_2O_2/COD:** The efficiency of ph-F process directly relates to the amount of hydroxyl radicals generated from the principle reaction between H_2O_2 and the catalyst. Figure 5 depicts the relation between the different H_2O_2/COD ratios versus COD concentration. Turbidity of the neutralized ph-F outlet was also measured and presented in Figure 5.

In all ph-F optimization experiments, the concentration of COD and turbidity of the inlet were 1112 mgO_2/L and 7.04 NTU, respectively. It is represented that the highest COD removal (81.57 %) obtained at H_2O_2/COD of 1.5, which resulted in COD concentration of 204.9 mgO_2/L in outlet. Concomitantly, the turbidity of effluent reached its minimum amount (less than 1 NTU). In the smaller H_2O_2/COD ratios (0.5 and 1), the COD concentration of the neutralized effluent was more than 250 mg/L. The lower COD removal efficiency in H_2O_2/COD of 0.5 and 1 experiments was related to the deficiency of hydroxyl free radicals. Since the generated $OH^{\bullet}$ can be scavenged by presented ferrous ions and H_2O_2, the rate of OH oxidizing reactions would decrease. The main scavenging reactions of $OH^{\bullet}$ in Fenton reactions are shown in (E. 6) and (E. 7).

$$Fe^{2+} + OH^{\bullet} \rightarrow Fe^{3+} + OH^{-} \qquad \text{(E.6)}$$

$$H_2O_2 + OH^{\bullet} \rightarrow HO_2^{\bullet} + H_2O \qquad \text{(E.7)}$$

Additionally, the measured turbidity index was higher in the H_2O_2/COD of 0.5 and 1 than that of in 1.5. This could be due to more particulate matter and unprecipitated iron complexes remained in the effluents because of low dosage of oxidizing agent and incomplete Fenton reaction chains.

The COD removal efficiency was enhanced to 81.5% at H_2O_2/COD ratio of 1.5:1. But it did not improve in the presence of higher H_2O_2 dosages (H_2O_2/COD ratios of 2, 2.5 and 3) and a limited ferrousion concentration (Fe^{2+}/H_2O_2= 1:20). Thus, the optimum ratio for ph-F reaction was 1.5:1 in this condition.

On the other side, the turbidity index enlarged to 8 and 14 NTU by increasingthe H_2O_2/COD ratios above 1.5, because of high loadingof iron concentration. The higher H_2O_2/COD required the higher equivalent Fe^{2+} salt concentration to provide the constant Fe^{2+}/H_2O_2 ratio. To lessen the COD concentration lower than standard limits (200 mgO_2/L), $Fe^{2+}/H_2O_2$was increased in the next set of experiments.

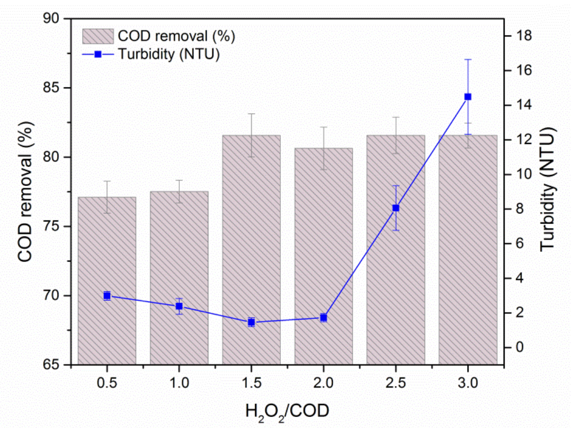


Figure 5. Turbidity and COD concentration of ph-F effluent at different H_2O_2/COD ratios (1:2, 1:1, 1.5:1, 2:1, 2.5:1 and 3:1); Fe^{2+}/H_2O_2=1:20, pH=4 and retention time= 1 h

3.2.2. Optimization of Fe^{2+}/ H_2O_2: The higher organic removal efficiency can be achieved in the presence of proper amount of metal catalyst. The excess amount of Fe^{2+} would resulted in increasing the amount of total dissolved solids (TDS) in effluent, iron sludge production and incapability for enhancing the COD removal efficiency (Arimi, et al., 2016). Thus, the amount of proper Fe^{2+} (or Fe^{2+}/H_2O_2 in many cases) must be optimized primarily. Keeping this in mind, the Fe^{2+}/H_2O_2 ratio of 1:50, 1:20, 1:10, 1:5 and 1:2 (with the equivalent Fe^{2+} of 54.8, 136.9, 273.8, 547.7 and 1369.2 mg/L) were examined. The results, which are demonstrated in Figure 6, indicate that COD was reduced under the specified limit for irrigation water when Fe^{2+}/ H_2O_2 was set on 1:10 ratio. In that case, final COD concentration was 191.4 mg/L (COD removal of 82.8%) despite the turbidity increased slightly. However, the greater Fe^{2+}/H_2O_2 of 1:5 and 1:2 not only increased the turbidity, but also decreased the COD removal efficiency because of overloading of $FeSO_4.7H_2O$ and scavenging phenomena between the oxidant and catalyst.

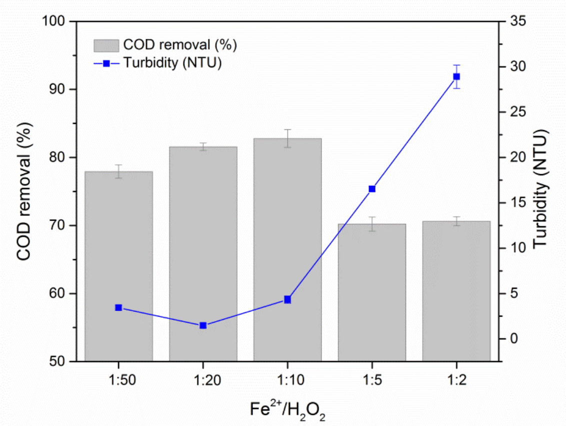


Figure 6. Turbidity and COD concentration of ph-F effluent at different Fe^{2+}/H_2O_2 ratios (1:50, 1:20, 1:10, 1:5 and 1:2); H_2O_2/COD=1.5, pH=4 and retention time= 1 h.

3.2.3. **Effect of light:** Radiating high energetic lights will have positive effects on performance of Fenton reaction. It can increase the regeneration rate of Fe^{2+} as well as production of hydroxyl radicals (Funai, et al., 2017). The results of COD removal efficiency obtained through UV-Fenton, dark Fenton and Solar-Fenton processes were compared.

As shown in Figure 7, the absence of light in dark Fenton experiments resulted in the lowest

COD removal efficiency (69.8%). The low efficiency of Fenton reaction is due to the accumulation of Fe^{3+}complexes, quick consumption and scavenging effect of OH• free radicals (see E. 6).

Considering the results of ph-F processes, the Solar-Fenton had higher COD removal efficiency than UV-Fenton. UV-C and solar lights improved the rate of Fenton reactions, noticeably. According to the literature, radiation of UV-vis light with a wavelength up to 600 nm would increase the production rate of OH•; the Fe^{3+} complexes are broken down (photolysis) and Fe^{2+} is regenerated for the catalytic activity. The (E. 8) exhibits the photo-reduction of $[Fe(OH)]^{2+}$ complex (Oller, et al., 2011). In addition, much free radical can be generated through photolysis of the oxidant. Decomposition of H_2O_2 occurs in the presence of lights with low wavelengths (below 300 nm), as shown in (E. 9) (Galehdar, et al., 2009).

$$[Fe(OH)]^{2+} \xrightarrow{hv} Fe^{2+} + OH^{\circ} \quad \lambda < 600 \text{ nm} \qquad \text{(E.8)}$$

$$H_2O_2 \xrightarrow{hv} 2OH^{\circ} \quad \lambda < 300 \text{ nm} \qquad \text{(E.9)}$$

The radiation of UV-C and solar lights increased COD removal efficiencies up to 82.8 and 85.3%, respectively; the equivalent COD concentrations were 191 and 163 mgO_2/L.

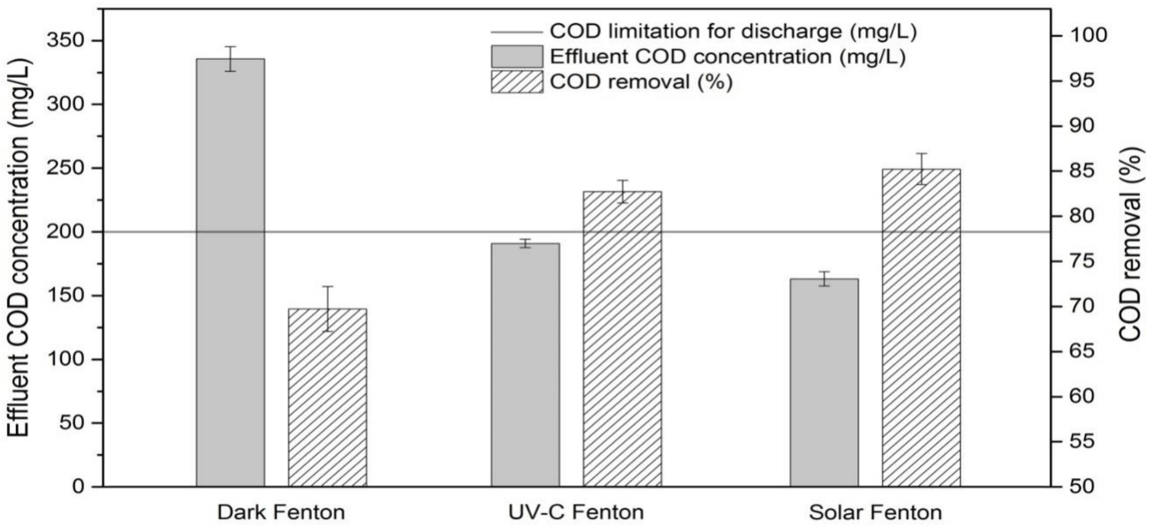


Figure 7: Effluent COD concentration and the removal capacity of dark Fenton, UV ph-F and Solar ph-F process.

The final results of SBR and ph-F were tabulated in Table 3. Considering the same processes, application of a hybrid system including aerobic granular SBR and ph-F process led to efficient treatment of cattle manure wastewater in this project (Elmolla and Chaudhuri 2011, Guzmán, et al., 2015, Wu, et al., 2013). The aerobic granular sludge was capable to consume most of the influent ammonium and phosphorus contents. Also, the great COD reduction of 79% was obtained during 32 h as hydraulic retention time (24 h cycle time, VER=75%). Solar and UV-C ph-F reaction significantly reduced the effluent's turbidity and COD concentrations.

Performances of aerobic granular SBR and ph-F treatment processes are showed in Table 3. In one hand, aerobic granular SBR degraded 79% COD, 97.1% TKN and over 99% of ammonium and phosphorus concentrations at the steady-state condition. Efficiency of the biological treatment process for eliminating suspended solid content was noticeable (92% TSS removal). On the other hand, solar Fenton process resulted in high COD removal efficiency (85.3%) at the optimized conditions. The performance of solar Fenton was not very noteworthy for decreasing TKN and TSS content. However, it was an effective method because the final effluent successfully met the national discharge standards (ISIRI No. 2439) legislated for irrigation water[1].

[1]http://standard.isiri.gov.ir/

Table 3: The characteristics of effluents from aerobic granular SBR and solar-Fenton unit

Parameter	SBR outlet	Solar-Fenton neutralized effluent	Allowable limits (IEPA standards)	SBR removal efficiency (%)	ph-F removal efficiency	Standard measuring methods*
COD (mgO_2/L)	1112	163.6	200	79	85.3	5220 C
TKN (mg/L)	34	18	25	97.1	47	4500 N_{org}, B
NH_4^+-N (mg/L)	<0.05	–	1	99.9	-	4500 NH_3, C (54)
TP (mg/L)	<1	–	6	99	-	4500-P (73)
TSS (mg/L)	200	70	100	92	65	2540 D
Turbidity (NTU)	7.04	4.33	50	-	-	2130 B
Fe (mg/L)	0	0.65	3	-	-	Atomic absorption

* All measurements were conducted according to (APHA, A. P. H. A. 2005), except Fe analysis.

Conclusion

In this study, the feasibility of water recycling using a combined biological and chemical system was assessed. Cattle manure wastewater was successfully treated through an aerobic granular SBR followed by a photo-Fenton post treatment. Full ammonium and TP removal were obtained in aerobic granular SBR. The granular sludge had proper resistance against substrate shock (F/M over 1) at last runs. The best results of substrate utilization rates were observed at hydraulic retention time of 32 h. Although noticeable COD removal of 79% was obtained in the granular bioreactor, it was enhanced to 97% after solar ph-F reaction. In this project, the solar and UV-C ph-F processes had much higher COD removal efficiencies rather than dark Fenton. Concluding all remarks, the efficient combination of aerobic granular SBR with solar ph-F shall be a technical approach for degradation of pollutants and recycling of irrigation water.

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