Original Article

Sequencing treatment of industrial wastewater with ultraviolet/H_2O_2 advanced oxidation and moving bed bioreactor

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ABSTRACT

Aims: The main purpose of this study was to determine the efficiency of a sequencing treatment including ultraviolet (UV)/H_2O_2 oxidation followed by a moving bed bioreactor (MBBR).

Materials and Methods: Effect of solution pH, reaction time, and H_2O_2 concentration were investigated for an industrial wastewater sample. The effluent of the advanced oxidation processes unit was introduced to the MBBR operated for three hydraulic retention times of 4, 8, and 12 h.

Results: The optimum condition for industrial wastewater treatment via advanced oxidation was solution pH: 7, H_2O_2 dose: 1000 mg/L and 90 min reaction time. These conditions led to 74.68% chemical oxygen demand (COD) removal and 66.15% biochemical oxygen demand (BOD_5) removal from presedimentation step effluent that initially had COD and BOD_5 contents of 4,400 and 1,950 mg/L, respectively.

Conclusion: Combination of UV/H_2O_2 advanced oxidation with MBBR could result in effluents that meet water quality standards for discharge to receiving waters.

Key words: Industrial wastewater treatment, moving bed bioreactor, refractory compounds, ultraviolet/H_2O_2 oxidation

INTRODUCTION

There are many reports about feasibility of advanced chemical and physical treatment methods such as advanced oxidation processes, chemical oxidation, chemical precipitation, adsorption, and membrane filtration for treatment of industrial wastewaters; however, biological processes may be the most environmental friendly approach for wastewater treatment and comprise the main stage in treatment trains for many industrial and domestic wastewater treatment plants.[1,2] In the case of industrial wastewaters that contain nonbiodegradable, toxic, and refractory compounds, a biological process is usually used as the main treatment step to achieve effluent standards, while other chemical, photochemical, or physical processes are designed as pretreatment steps.[3] Therefore, biological wastewater treatment processes are preferred for wastewaters with high biodegradable organic content and wastewaters

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containing nutrients such as nitrogen and phosphorus.[4] Other conventional or advanced chemical or photochemical treatment processes are most commonly used as a pre or post treatment step in addition to the main biological treatment, or are directly used for nonorganic wastewaters.[4] Biological treatments may be limited by the presence of toxic pollutants, the need for a large area and large volume tanks, and low reaction rate (i.e., time intensive). Alternative methods like advanced oxidation processes (AOP) can effectively overcome these limitations.[5,6] Despite the capacity of conventional biological processes to remove the majority of organic matter, the presence of low amounts of xenobiotics and refractory compounds requires the application of a supplementary option like AOP.[7,8] In the current study, ultraviolet (UV)/ H_2O_2 advanced oxidation is used as a pretreatment alternative to enhance the biodegradability of wastewater.[9] The benefits of AOP include effective removal of refractory organics in a short period of time, no precipitate after reaction, degradation, and mineralization of a variety of contaminants including hydrocarbons, halogenated solvents, phenolics, pesticides, glycols, and microorganisms (such as *Escherichia coli*), rapid and intensive reaction, and easy startup and shutdown of equipment.[10] Findings in the literature indicate that integration or sequence operation of biological treatment processes with various AOP can substantially enhances the treatment efficiency or biodegradability of wastewater. These processes are characterized by no or very low amounts of sludge production and rely on the generation of highly reactive hydroxyl radicals (OH) for oxidation of refractory organics present in wastewater.[11] Some AOP such as Fenton reaction or photo-oxidation with H_2O_2 are commonly used for enhancement of biochemical oxygen demand (BOD_5)/ chemical oxygen demand (COD) ratio of low biodegradable wastewaters or to complete the degradation of contaminants.[12] The conventional Fenton reaction is, usually, functioning at low pH 3 for efficient $OH^\bullet$ production and, therefore, full-scale applications in combination with biological processes can be limited.[13] De la Cruz *et al.* studied the degradation of emergent contaminants by a UV/H_2O_2 process in a pilot-scale domestic wastewater treatment plant. Their study focused on the removal of 22 selected micropollutants from municipal wastewater treatment plant effluent and removed >80% of the micropollutants for the majority of flow rates studied.[14] Cao and Mehrvar studied the efficiency of a UV/H_2O_2 process as an additional step for slaughterhouse wastewater treatment. They declared that individual anaerobic biological reactor (ABR) and UV/H_2O_2 processes enhanced the biodegradability of the treated effluent by increasing the carbonaceous biochemical oxygen demand$_5$/ chemical oxygen demand (COD) ratio from 0.4 to 0.6. An optimum H_2O_2 dosage of 3.5 mg H_2O_2 per mg TOC in h was also found for the UV/H_2O_2 process.[15] Application of integrated bioreactors like moving or fixed bed reactors has been widely investigated.[16,17] Their advantages include high concentrations of biomass, possible application of low cost beds, capacity to treat high flow rates, greater removal efficiency compared to suspended growth reactors with the same flow rate, effective treatment of low concentration wastewaters, capacity to treat organic compounds that have low degradation rates, resistance to hydraulic and organic shocks, lower energy and space requirements, lower microbial yield — less waste excess sludge production, and better quality of secondary effluent have been reported by many investigations.[18,19] There are many reports related to the application of a mixed bed bioreactor (MBBR) for treatment of domestic and industrial wastewater, such as by Dong *et al*. who used MBBR for treatment of oilfield wastewater,[19] or Li *et al*. who investigated removal of phenol by MBBR.[2]

Therefore, we used UV/H_2O_2 oxidation as a pretreatment step for an industrial wastewater that had a characteristically low biodegradability. The wastewater sample contained high concentrations of refractory, toxic, and nonbiodegradable constituents that limit the direct biological treatment. The study focused on real wastewater obtained from Shiraz industrial estate, Iran [Figure 1]. The Shiraz industrial estate is located in the vicinity of Shiraz city with an area of about 1407 ha. Among the 1253 different industries having valid certificates, 906 industries are active in the Shiraz industrial estate including various food, chemical, metallic and nonmetallic, health care products, electronic, textile, and cellulosic industries. A wastewater treatment plant with a final capacity of 2500 m^3/d is used for the treatment of wastewater collected from all industries. The present research assesses the viability of a UV/H_2O_2 process followed by MBBR as a pretreatment step for influent to the wastewater treatment plant. According to the literature review, no similar study was carried out in industrial estates of Iran.

MATERIALS AND METHODS

Characteristics of raw wastewater

The wastewater samples were collected from the collecting line of a Shiraz industrial estate and transferred to the

Figure 1: The layout map of Shiraz industrial Estate

laboratory at 4°C. Important characteristics of wastewater are presented in Table 1.

Optimization of the operational parameters of ultraviolet/H_2O_2 oxidation

A presedimentation step with 60 min retention time was used at the beginning of the treatment train to overcome high turbidity wastewater and to enhance the UV penetration into the liquid. The presedimentation reactor was subdivided into two sequence steps, with the addition of 5 mg/L cationic polymer that was gently mixed with the aid of an internal stirrer at 30 rpm for 10 min, followed by sedimentation for 50 min. Two UV lamps with constant intensity of 5.7 mW/cm^2 were placed into cylindrical quart containers and inserted into the wastewater tank. An 80 cm × 40 cm × 20 cm container was used as a UV/H_2O_2 reactor. Optimization included adjusting conditions of the UV/H_2O_2 reactor to pH (5, 6, 7, 8, and 9), H_2O_2 concentrations of 200, 400, 600, 800, and 1000 mg/L, and reaction times of 0-120 min. With a constant H_2O_2 concentration of 200 mg/L and reaction time of 30 min, pH (5-9) was varied in the first optimization step using sulfuric acid or sodium hydroxide. After the optimum pH was determined, the H_2O_2 concentrations were varied from 200 to 1000 mg/L at a constant reaction time of 30 min. Finally, the optimum reaction time adjusted from 0 to 120 min, while holding the pH and H_2O_2 concentrations at predetermined optimums. Samples were collected every 10 min and concentrations of BOD_5 and COD were immediately analyzed.

Moving bed bioreactor (MBBR)

The MBBR was comprised of a cubic plexiglass container with a total effective volume of 20 L. Aeration volume was filled to 40% with a commercial activated sludge media (2-H™). The high-density polyethylene media, used in this study, had a specific surface area of 767 m^2/m^3 and porosity of 93%. The contents of the MBBR were mixed and aerated with the aid of an air compressor. The lab-scale reactor was operated at room temperature (21°C ± 4°C), and the pH was adjusted from 6.7 to 7.4 using sodium bicarbonate. The activated sludge was transferred from an existing industrial wastewater treatment plant to the lab-scale MBBR at 4°C. After 30 min retention, the supernatant was withdrawn, and the settled sludge was fed to the MBBR. The MBBR was started up with synthetic wastewater containing glucose (to attain an initial COD concentration of 1000 mg/L) as the main source of carbon and energy for activated sludge. The composition of synthetic wastewater is presented in Table 2. Nitrogen and phosphorus sources were NH_4Cl and KH_2PO_4, respectively. The C/N/P ratio was adjusted 100/5/1 for optimum growth conditions of microorganisms.[20,21] The MBBR was operated in batch mode for approximately 70 days to provide enough biofilm growth on media carriers, and to obtain consistent results in terms of COD removal. The MBBR was continuously aerated to maintain dissolved oxygen (DO) concentrations of 4-5 mg/L. Thereafter, the MBBR was fed continuously with industrial wastewater that passed through a polymer addition-sedimentation pretreatment, and UV/H_2O_2 oxidation in three different hydraulic retention times (HRT) of 4, 8, and 12 h. An intermediate storage tank was used between UV/H_2O_2 oxidation and MBBR to provide adequate flow of wastewater to MBBR. All of the experimental data were collected under steady-state conditions and are expressed in terms of arithmetic averages of a minimum of three replicates. A schematic of the treatment train is shown in Figure 2.

Analytical methods

The BOD_5, COD, DO, NH_3-N, PO_4, total suspended solids (TSS), turbidity, bacterial density, and oil and grease content were determined according to standard methods of examination water and wastewater.[22] Temperature was determined with a thermometer and pH measured with a digital pH meter. Biofilm mass was determined using 200 media elements that were sampled randomly from the MBBR. Media elements were separated from the wastewater and dried in an oven at 104°C until they reached a constant weight. The dried samples were weighed to determine the total mass (*M* total) composed of media element mass (*M* media) and the attached biomass. The biomass was then washed off, the clean media elements were weighed, and the amount of biofilm attached to the 200 media elements was calculated using Eq (1). The amount of biomass in the reactor was then

Table 1: The characteristics of raw wastewater

Parameter	Value
COD (mg/L)	6085 ± 310
BOD_5 (mg/L)	2280 ± 340
BOD_5/COD	0.37
TSS (mg/L)	980 ± 135
NH_3-N	610 ± 49
P mg/L (mg/L)	27 ± 98
Oil and grease (mg/L)	40 ± 110
MPN/100 mL (fecal coliform)	350 ± 9100
pH	7.5-6.7

Table 2: Composition of synthetic wastewater for startup of MBBR[a]

Constituent	Amount
$C_6H_{12}O_6$[b] mg/L	1780
NH_4Cl mg/L	56
KH_2PO_4 mg/L	11.2
$MgSO_4$ mg/L	139.2
$NaHCO_3$ mg/L	210
$CaCl_2 \cdot 2H_2O$ mg/L	45
$CuSO_4 \cdot H_2O$ mg/L	0.16
$Na_2MoO_4 \cdot 2H_2O$ mg/L	0.30
$MnSO_4 \cdot H_2O$ mg/L	0.26
$ZnCl_2$ mg/L	0.46
$CoCl_2 \cdot 6H_2O$ mg/L	0.84
$FeCl_2 \cdot 4H_2O$ mg/L	17.5
pH	7 ± 0.3

[a]Amounts for total COD of 1000 mg/L, [b]Purity of $C_6H_{12}O_6$ = 60% (1).

determined using the known number of carrier elements in the reactor with filing grade of 40%.[23]

$$BS_{100} = M\ \text{total} - M\ \text{media} \quad \text{Eq (1)}$$

RESULTS

Presedimentation step efficiency

A pretreatment step including cationic polymer addition and 50 min sedimentation was used to remove excess amounts of TSS and turbidity and, therefore, enhance UV penetration into the wastewater. The TSS of raw wastewater decreased from 985 to 130 mg/L (86% removal) as shown in Table 3. Final turbidity of pretreated wastewater was 16 NTU and was acceptable for the UV/H_2O_2 treatment. COD and BOD_5 values of raw wastewater decreased from 6085 and 2280 mg/L to 4400 and 1950 mg/L, respectively (27.6% COD removal, 14.47% BOD_5 removal).

Determination of optimum conditions of ultraviolet/ H_2O_2 oxidation

pH

The results of pH optimization are presented in Figure 3a and b. The minimum BOD and COD concentrations of 1300 and 2650 mg/L, respectively, were observed at pH 5. The BOD_5/COD ratio, which is an indicator of wastewater biodegradability, was equal to 0.49 at pH 5 and increased to 0.6 at pH 7. This verifies the enhancement of biodegradability, despite the presence of more BOD and COD compared to pH 5. The BOD and COD concentrations at pH 7 were 1650 and 2710 mg/L, respectively. Considering the following biological treatment step, pH 7 was selected as the optimum value since biological reactions proceed more efficiently in neutral pH.

H_2O_2 concentration

After the optimum pH was determined, the desired concentration of H_2O_2 in the range of 200-1000 mg/L was investigated. Results for optimization of H_2O_2 concentration in optimum pH 7 are shown in Figure 4a and b. Minimum BOD and COD concentrations of 890 and 1225 mg/L were achieved at H_2O_2 concentration of 1000 mg/L in constant reaction time of 30 min and pH 7, respectively. The BOD_5/COD ratio of the effluent after the presedimentation step was increased from 0.44 to 0.72 at H_2O_2 concentration of 1000 mg/L. Thus, the H_2O_2 concentration of 1000 mg/L was selected as the optimum value for following experiments.

Reaction time

The results of time optimization for effluent of the presedimentation step in optimum pH and H_2O_2 concentrations are shown on Figure 5a and b. In general, COD and BOD_5 removal efficiencies increased directly with reaction time until 120 min, but reaction times of >90 min did not significantly improve the removal efficiency. The residual BOD_5 and COD concentrations at a reaction time of 120 min were 645 and 810 mg/L, respectively, and

Table 3: The efficiency of presedimentation step

Parameter	In	Out	Removal (%)
TSS	985	130	86.8
COD	6085	4400	27.69
BOD	2280	1950	14.47

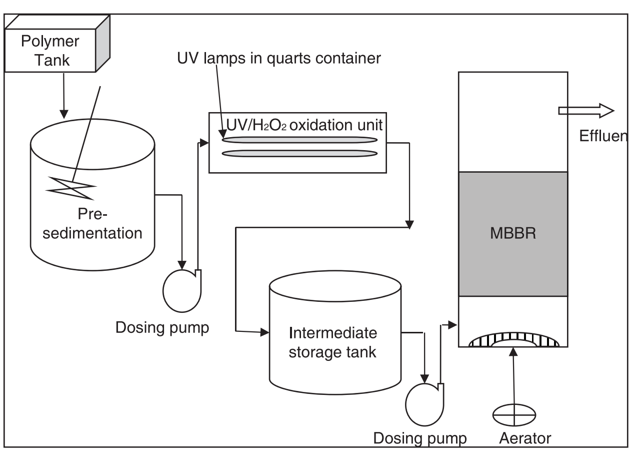

Figure 2: A schematic diagram of lab scale treatment reactor including presedimentation, ultraviolet/H_2O_2 advanced oxidation and MBBR

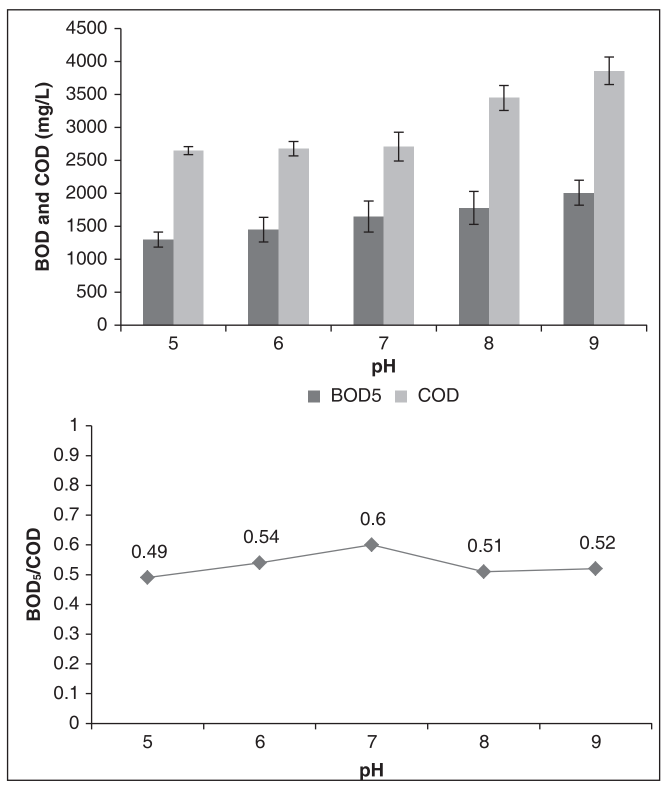

Figure 3: (a) The variations of BOD_5 and COD in different pH for ultraviolet (UV)/H_2O_2 advanced oxidation process, (b) the variations of BOD_5/COD ratio in different pHs for UV/H_2O_2 advanced oxidation process

the BOD_5/COD ratio reached 0.74. A reaction time of 90 min was selected as the optimum value, because acceptable removal efficiency was achieved at this time. Residual COD and BOD_5 concentrations of 1114 and 660 mg/L, respectively, were achieved with a reaction time of 90 min, pH 7, and H_2O_2 concentration of 1000 mg/L and resulted in a BOD_5/COD ratio of 0.8. The COD concentration can easily be removed by MBBR and the BOD_5/COD ratio of 0.8 for a 90 min reaction supports the biodegradation of organic matter. Thus, the reaction time of 90 min was determined as the optimum value for UV/H_2O_2 process.

The biological treatment by MBBR

A final biological treatment step including MBBR was used to achieve standards required to discharge effluent to receiving waters. Effluent from the UV/H_2O_2 oxidation unit having BOD_5, COD, and TSS concentrations of 660, 1114, and 56 mg/L, respectively, became the influent for the MBBR. The MBBR was first operated with synthetic wastewater to start up the reactor and obtain an adequate biofilm for effective degradation of organic matter. Then, the MBBR was operated in three HRT of 4, 8, and 12 h with wastewater effluent from the UV/H_2O_2 oxidation unit. A summary of results of these experiments is presented in Table 4. Residual COD for HRTs of 12, 8, and 4 h were 68, 84, and 93 mg/L, respectively. The residual BOD_5 for HRTs of 12, 8, and 4 h were 39, 52, and 70 mg/L, respectively. Residual TSS for the feed TSS of 56 mg/L and HRTs of 12, 8, and 4 h were 25, 30, and 34 mg/L, respectively. The variations of biomass in MBBR are presented in Figure 6. The volatile suspended solids (VSS) concentrations of 3550, 3942, and 4325 mg/L were observed

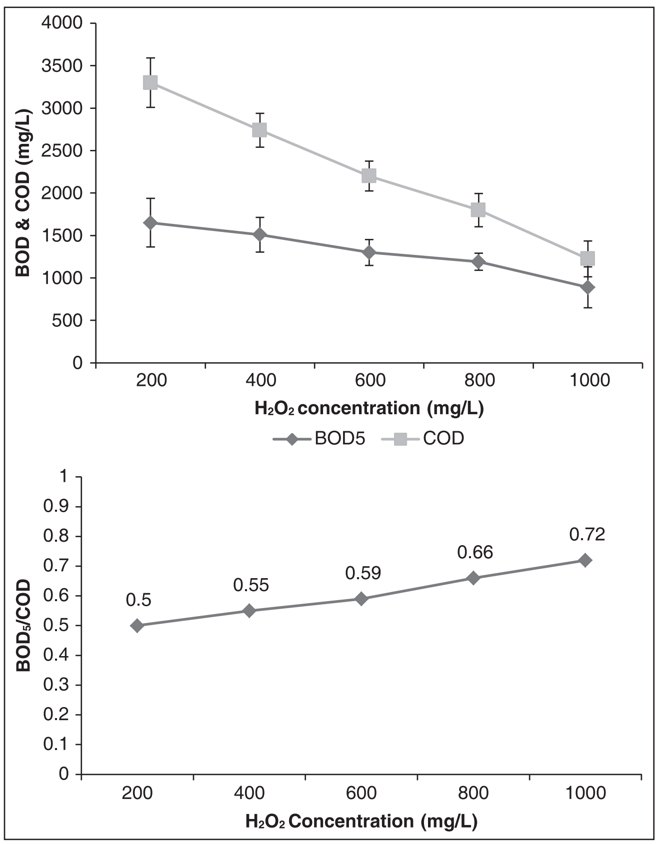

Figure 4: (a) The variations of BOD_5 and COD in different H_2O_2 concentrations for ultraviolet (UV)/H_2O_2 advanced oxidation process (b) The variations of BOD_5/COD ratio in different H_2O_2 concentrations for UV/H_2O_2 advanced oxidation process

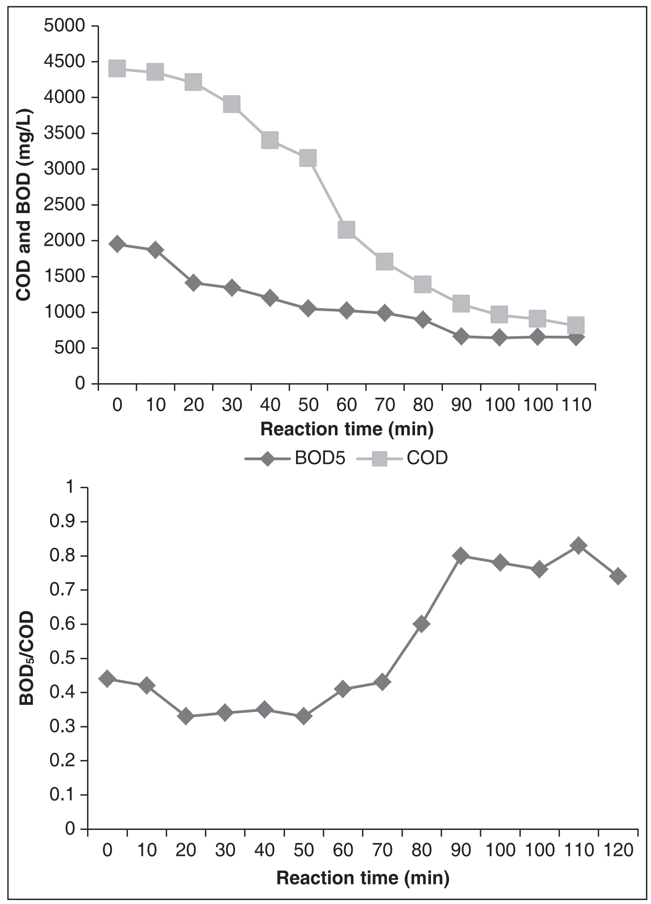

Figure 5: (a) The variations of BOD_5 and COD along with reaction time, (b) the variations of BOD_5/COD ratio along with reaction time, for ultraviolet/H_2O_2 advanced oxidation process

Table 4: The variations of characteristic parameters in MBBR

HRT (h)	Parameter									
	COD in (mg/L)	BOD_5 in (mg/L)	TSS in (mg/L)	OLR (kg COD/m^3/d)	COD out (mg/L)	BOD_5 out (mg/L)	TSS out (mg/L)	COD removal (%)	BOD_5 removal (%)	Day[a]
12	1114	660	56	2.228	68	39	25	93.8	94.09	34
8	1114	660	56	3.342	84	52	30	92.4	92.12	48
4	1114	660	56	6.684	93	70	34	91.6	89.39	52

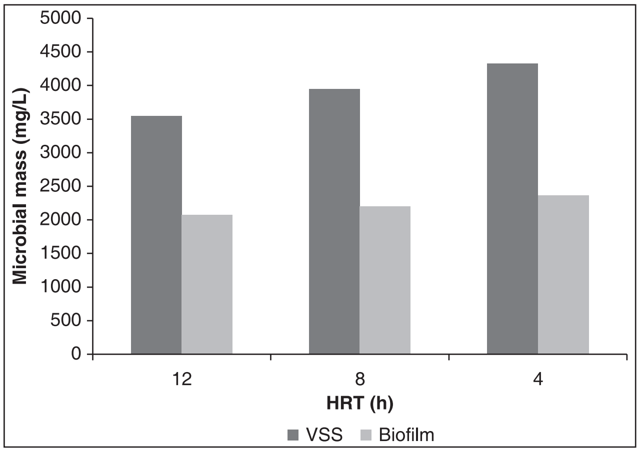

Figure 6: The variations of biomass along with hydraulic retention times in operation of MBBR as biologic treatment step

for HRTs of 12, 8, and 4 h, respectively. Furthermore, the biofilm attached to media carriers were 2074, 2196, and 2364 mg/L for HRTs of 12, 8, and 4 h, respectively.

DISCUSSION

Results indicate that along with pH increase, the COD removal decreased as a result of the UV/H_2O_2 treatment process. This agrees with findings of a study by Yonar *et al*., which reports that COD removal decreases along with a pH increase.[24] In a similar study by Elmorsi *et al*., the optimum pH 7 was reported for dye removal via UV/H_2O_2 oxidation.[25] In the current study, the BOD_5 concentrations directly increased with pH and reached 1870 mg/L at pH 8. Since a major function of AOP is partial oxidation of refractory compounds, the increase of readily biodegradable BOD_5 can be expected. Higher COD removal efficiency, relative to BOD_5 removal, may be due to the production of intermediates and incomplete degradation of organics from partial oxidation refractory organics and insufficient contact time. These intermediates increase the BOD_5 value; despite the progress of biodegradation. In general, a characteristic of AOP is the rapid breakdown of compounds and mineralization over a long period of time. The raw wastewater had a low BOD_5/COD ratio of 0.37, which made the direct application of biological process infeasible. Thus, the UV/H_2O_2 oxidation was used to increase the biodegradability in terms of the BOD_5/COD ratio and the criteria for determining optimum pH was the BOD_5/COD ratio reached >0.5. In pH 5, 6, and 7, the BOD_5/COD ratio was 0.49, 0.54, and 0.6, respectively. Therefore, despite the lower COD removal, pH 7 was selected as the optimum pH. Because biological reactions function more effectively in pH range from 6.8 to 7.5, the application of MBBR was suitable as a final biological treatment step after UV/H_2O_2 oxidation.[26] Optimization of H_2O_2 concentration indicated that along with H_2O_2 increase, the COD and BOD_5 removal and also the BOD_5/COD ratio were increased. This is in accordance with Daneshvar *et al*. who reports an increase of H_2O_2 concentration from 50 to 450 mg/L also increased the dye removal efficiency from 30% to 72%.[27] Valderrama *et al*. stated that biological treatment comprises part of a treatment scheme, and investigation of higher H_2O_2 concentrations was not considered because excess amounts of H_2O_2 concentration can inhibit the biological reactions and microorganism's metabolism.[28] According to the literature, the reaction time for the UV/H_2O_2 process for removal of different organics varies from 15 min to 5 h.[29-31] The contact time of 90 min obtained in the current study was within this range. According to results presented in Table 4, along with an HRT decrease, the effluent COD and BOD_5 concentrations increased, but still are lower than standards for discharge to receiving waters. Despite the COD and BOD_5 concentration increases during HRT depletion, the quantity of organic matter removed in HRT of 4 with organic loading rate (OLR) of 6.684 kg COD per m^3d is much higher than an HRT of 8 h with OLR of 3.342 kg COD per m^3d and 12 h with OLR of 2.228 kg COD per m^3d. This suitable removal efficiency, due to oxidation and partial oxidation, indicates that AOP can be applied before a biological treatment process, thus providing readily biodegradable organic matter to the next treatment process. The presence of a biofilm in addition to the suspended biomass provides sufficient microbial mass in the bioreactor to then have a high capacity for biodegradation of organic matter [Figure 6]. In the HRTs of 12, 8, and 4 h, the total biomass of 5624, 6138, and 6689 mg/L were observed, respectively. Therefore, retention time after initial startup is important for obtaining consistent results. According to Table 4, 52 days are required for MBBR to obtain consistent results with HRT of 4 h, which is more than required for HRT of 8 h (48 day) and HRT of 12 h (34 days). We believe that HRT of 4 h is optimal, because the treatment process benefits from smaller volumes in structural units and tanks, which results in higher flow rates compared to HRTs of 8 and 12 h. Therefore, this treatment train can effectively be used to achieve effluent standards for industrial wastewaters that initially have low biodegradability.

CONCLUSION

A pretreatment step including cationic polymer addition and 50 min sedimentation yielded the TSS, COD, and BOD_5 removal of 86%, 27%, and 14%, respectively. The minimum BOD and COD concentrations of 890 and 1225 mg/L were achieved at H_2O_2 concentration of 1000 mg/L in constant reaction time of 30 min and pH 7, respectively. The residual COD and BOD_5 concentrations of 1114 and 660 mg/L were achieved in the reaction time of 90 min, pH 7 and H_2O_2 concentration of 1000 mg/L. Optimum conditions of UV/H_2O_2 advanced oxidation process were pH 7, H_2O_2 concentration of 1000 mg/L, and the reaction time of 90 min. In the biologic treatment step (MBBR), the residual

COD for HRTs of 12, 8, and 4 h were 68, 84, and 93 mg/L, respectively. The residual BOD_5 for HRTs of 12, 8, and 4 h were 39, 52, and 70 mg/L, respectively. Findings of the present study indicate that sequencing treatment including presedimentation with polymer addition, UV/H_2O_2 oxidation, and MBBR can efficiently achieve water quality standards for discharge of industrial effluent to receiving waters.

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