

Treatment of highly polluted grey waters using Fenton, UV/H₂O₂ and UV/TiO₂ processes

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Abstract. Household or office wastewater consists of two parts of faecal and non-faecal wastewater. Non-faecal section, which accounts for more than 60% of household wastewater, is known as grey water. Treating this part of sewage and using it in various areas can greatly reduce the consumption of potable water. In this research, three methods of advanced oxidation processes including Fenton, UV/H₂O₂, and UV/TiO₂ were investigated for treatment of two grey water sourced from Shif Island and Persian Gulf University restaurant in Bushehr province of Iran. These grey waters were highly polluted with COD content of 600 mg/L and 1400 mg/L, respectively. The efficiency of each process was determined by measuring the COD removal rate. Results showed that at optimum reaction conditions, the COD removal efficiency of the UV/H₂O₂ process was the best, compared to the other two processes. The COD removal efficiency of UV/H₂O₂ process at pH = 3 and H₂O₂ = 1500 and 750 mg/L for grey water of the Shif Island and Persian Gulf University restaurant were 88% and 90%, respectively. Therefore, the treated grey waters can be reused economically.

Keywords: grey water; Fenton process; UV/H₂O₂ process; UV/TiO₂ process

1. Introduction

Water is one of the most important resources for the survival of planetary organisms and an essential tool for the healthy development of humans. Today, due to various reasons such as industrial growth, population growth, climate change, and the simplification of water use water consumption and pollution have increased, and on the other hand, access to clean water has decreased (Khan *et al.* 2016, Wang *et al.* 2019, Behrouzeh *et al.* 2020, Melo *et al.* 2020). As a result, in addition to managing water resources, planning for the reuse of contaminated water resources is inevitable (Cha *et al.* 2020, Kumar *et al.* 2020). One of the resources available for reuse is household sewage. Household sewage includes sewage of the toilet, kitchen, and bathroom. Moreover, it includes part of sewage from stores, restaurants, and other institutions (Shamabadi *et al.* 2015, Leong *et al.* 2018b). This resource is one of the largest water resources and its recycling can reduce 30-50% of potable water consumption (Al-Jayyousi 2003, Oh *et al.* 2018). Household sewage consists of two categories: the first is black-water that defined as toilet sewage and the second is grey water that includes other household sewage sources, which is 67% of total household sewage (Chanakya and Khuntia 2014, Boguniewicz-Zablocka *et al.* 2019, Ligaray *et al.* 2019). The amount of pollutants in grey water depends on several factors such as living standards,

population structure, customs and habits, water and wastewater distribution systems, water abundance, type and amount of chemicals that usually used by people (Tsoumachidou *et al.* 2017). The existence of a wide range of contaminants makes researchers to use different physical, chemical, and biological methods to achieve the best method for treating grey water (Ghaididak and Yadav 2013). Physical methods such as screening and filtration (Šostar-Turk *et al.* 2005, Katukiza *et al.* 2014, Leong *et al.* 2018a), chemical methods such as coagulation, ion-exchange, and advanced oxidation processes (AOPs) (Šostar-Turk *et al.* 2005, Pidou *et al.* 2008, Ghaididak and Yadav 2013, Chong *et al.* 2015) and biological processes such as rotary biological contactors, continuous batch reactors, membrane bioreactors, and moving bed biofilm reactor (Nolde 2000, Merz *et al.* 2007, Chrispim and Nolasco 2017, Prajapati *et al.* 2019) have been investigated. Each of these methods has advantages and disadvantages and choice of the appropriate method for treatment of grey water depends on the grey water source, the quantity, quality, location specifications and its reuse (Edwin *et al.* 2014). However, among the methods mentioned, AOPs have been getting increasingly important due to the high strength of organic compounds decomposition (Fang *et al.* 2017). In these methods, •OH is produced which is the strongest free radical after fluorine. This compound is highly reactive, unstable, and has an average life of less than nanoseconds (Munter 2001), which quickly reacts with compounds in grey water and decomposes pollutants. So far, many studies have been carried out on grey water treatment with AOPs. Li *et al.* (2004) utilized constructed wetlands and subsequently photocatalytic oxidation. They

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were able to eliminate all organic compounds in a relatively long time period. Yonar *et al.* removed 95% COD content using UV/H₂O₂ method in optimum conditions for solution pH and H₂O₂ concentration (Yonar *et al.* 2006). Chin *et al.* reduced 87% of COD content using UV/H₂O₂ under optimal conditions (Chin *et al.* 2009). They also investigated kinetic of the reaction and concluded that the reduction of COD follows the second-order kinetic equation. Sanchez *et al.* investigated grey water treatment using photocatalytic process and recorded 65% TOC removal (Sanchez *et al.* 2010). They also could eliminate anionic surfactants, which are major contaminants of grey water. Hernandez *et al.* investigated four methods consist of aerobic, aerobic and anaerobic, aerobic and active carbon, and aerobic and ozone combinations. They concluded that aerobic biological treatment, followed by the use of activated carbon and ozone, could eliminate the contaminants of grey water (Leal *et al.* 2012). Tony *et al.* (2016) used Fenton method for treatment of grey water. They showed the ability of this process for removing 95% of COD content of grey water. Concerning the high potential of AOPs, in this research, it has been tried to compare three methods of AOPs consist of Fenton, UV/H₂O₂, and UV/TiO₂ for real grey waters treatment. Grey water sources are collected from two rural and urban sites. The first source is collected from the household sewage of the rural Shif Island in the Bushehr province of Iran, which lacks a wastewater treatment plant. The second source of gray-water is collected from the Persian Gulf University (PGU) restaurant in Bushehr city.

2. Materials and methods

Ferrous sulfate heptahydrate (purity \geq 99%), Hydrogen peroxide (purity 30%), Titanium dioxide Degussa P-25 (surface area = 50 m²/g and particle size = 21 nm), Sulfuric acid (purity 97%), and Sodium hydroxide (purity \geq 99%) were purchased from Merck Chemical Company (Germany).

A 8W 254 nm UV lamp was utilized in the UV process. Chemical oxygen demand (COD) measurement was carried out using DR900 Hach photometer and quick test cuvettes ranging from 0-1500 mg/L. To prepare each sample for COD analysis, 2 mL of the sample was poured into quick test cuvette and the cuvette placed in the COD reactor at 150°C for 2 h. Then the cuvette was placed out of the reactor to reach ambient temperature. Biochemical oxygen demand (BOD) was analyzed using BD-600 Lovibond tintometer. The pH and electrical conductivity (EC) of the grey waters were measured with a multi-9620 WTW multimeter. Also, nitrate, phosphate, and sulfate, chloride, oil and surfactant concentrations were determined using 4500 NO₃-E, 4500 P-C, 4500 SO₄-C, 4500 Cl-B, 5520-B, and 5540-C standard protocols, respectively. The Fecal coliform as well as total coliform were determined by most probable number (MPN) method. The measured parameters of the two grey waters are shown in Table 1.

To perform various experiments related to the designed processes, a 1.5-liter pyrex cylindrical glass reactor was

Table 1 Detailed parameters of two types of real grey waters

Grey water	Shif Island	PGU Restaurant
COD (mg/L)	600	1400
BOD (mg/L)	225	415
Fat and oil (mg/L)	129.6	383
pH	7.8	8.5
EC (mS/m)	16.72	18.58
Chloride	177	280
Fecal coliform (MPN)	90	0
Total coliform (MPN)	300	0
Sulfate (mg/L)	64	665
Nitrate (mg/L)	100.4	10.5
Phosphate (mg/L)	38.3	22.5
Surfactant	0.252	0.796

used. In each experiment, 1 liter grey water was introduced into the reactor and then the reactor was transferred on a digital magnetic stirrer at 300 rpm.

After that, in the Fenton process: 150-1250 mg/L Ferrous Sulfate- as the source of Fe²⁺ ion- and 250-3000 mg/L H₂O₂ was poured into the reactor. After the reaction had finished, excess Fe²⁺ was precipitated during 2 h by adding 1 M NaOH solution. Also, for the UV/H₂O₂ process: UV lamp was placed inside the reactor and then 125-2500 mg/L H₂O₂ was added to the reactor. In addition, for the UV/TiO₂ process, after inserting the UV lamp, 0.25 to 1.5 g/L TiO₂ was added to the reactor. For all processes, after the process had started the sampling took place at regular intervals. After that, each sample was filtered by glass fiber filter to separate precipitated solids and TiO₂-except for UV/H₂O₂ samples-and to ensure that no interactions were caused by H₂O₂, samples were placed at 50°C in an oven for 1 h. For each of three processes, sulfuric acid and sodium hydroxide were used to adjust the solution pH.

3. Results and discussion

3.1 Application of Fenton process for treatment of grey waters

To investigate the ability of the Fenton process for COD removal of grey water, the effect of various parameters such as process time, Fe²⁺ content, H₂O₂ content and the solution pH were examined on the process efficiency. The design of experiments is based on that the effect of each parameter will be carried out at fixed value of the other variables.

3.1.1 Effect of time on COD removal by the Fenton process

To find out the effect of time on COD removal by the Fenton process, COD removal test was carried out for two samples of grey water. The results of the COD removal due to Fenton process are shown in Fig. 1.

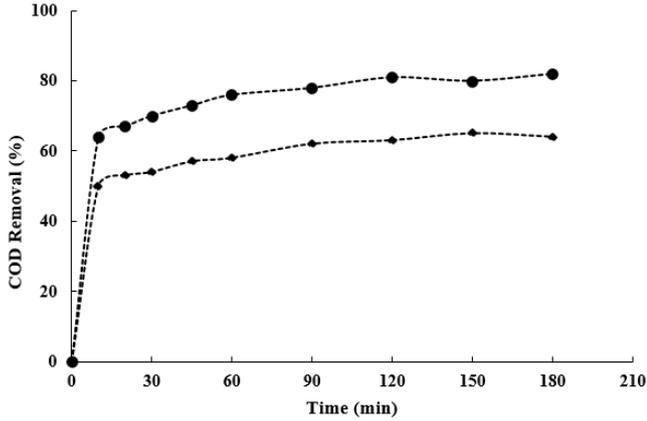
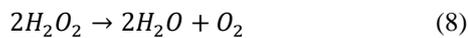
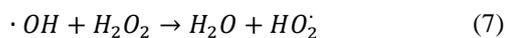
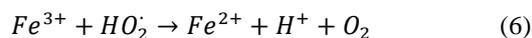
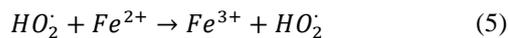
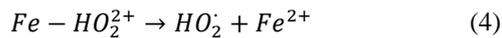
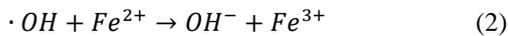
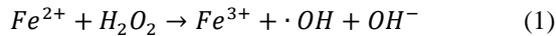
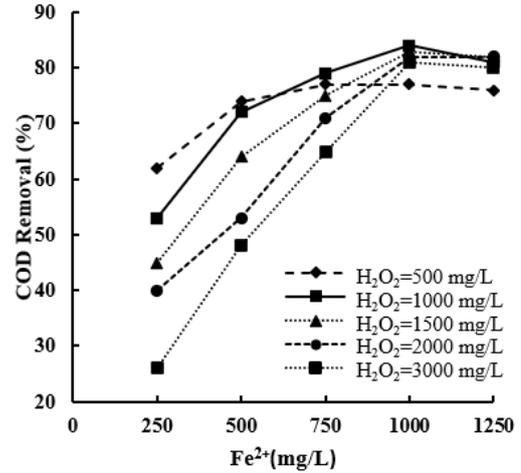


Fig. 1 Effect of time in Fenton process for COD removal of grey waters of Shif Island (●) and PGU restaurant (◆) (pH = 3, Fe²⁺ = 250 mg/L and H₂O₂ = 500 mg/L)

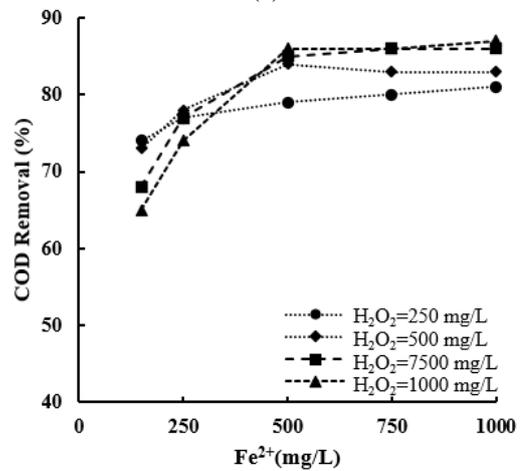
As it can be seen from the Fig. 1, COD removal due to organic compounds decomposition for both grey waters from the Shif Island and PGU restaurant is rapid in the early times (0-10 min), and then the rate of decomposition of the compounds decreases after 90 min. Also, the process of decomposition continues at a very slow rate until 180 min. To justify this phenomenon, the reactions associated with the initiation of the Fenton process can be used as below (Harimurti *et al.* 2010):



Fenton process initiate with reaction 1 that $\cdot OH$ is generated and this step is carried out rapidly. After that, a complex reaction sequence will generate products that were shown in reactions 2 to 8. In the presence of grey water, generated $\cdot OH$ quickly enter the chain reactions for decomposition of grey water compounds. Therefore, the decomposition process rate is fast at early minutes. After that, based on the reactions 3 and 4, the reduction of Fe³⁺ to Fe²⁺ will compete with reaction 1 and hence it will limit the efficiency of COD removal (Neyens and Baeyens 2003). Another reason for the slowdown COD removal efficiency is carbonate and bicarbonate production during the reaction that can act as a scavenger for $\cdot OH$ (Buxton *et al.* 1988).



(a)



(b)

Fig. 2 Effect of various H₂O₂ and Fe²⁺ concentration on COD removal efficiency of: (a) PGU restaurant and (b) Shif Island grey water at pH= 3 & Time=90 min

3.1.2 Effect of Fe²⁺ and H₂O₂ concentration in Fenton process

In this section, effect of various concentration of Fe²⁺ and H₂O₂ on COD removal efficiency have been investigated. Fe²⁺ and H₂O₂ concentration were selected 150-1250 mg/L and 250-3000 mg/L, respectively. The results of COD removal for Shif Islands and PGU restaurant grey waters are shown in Fig. 2. Analysis of data presents that for both grey waters, there is an optimum value for Fe²⁺ concentration at constant H₂O₂. Under this optimum value, raising Fe²⁺ concentration causes increase in COD removal efficiency. In fact, the higher the Fe²⁺ concentration means the higher amount of catalyst to react with H₂O₂ and hence the higher $\cdot OH$ production according to the reaction 1. However, when the Fe²⁺ concentration goes higher than optimum value, causes reduction in COD removal efficiency, which is converting Fe²⁺ to Fe³⁺ according to the reaction 2. In fact, Fe²⁺ at high concentrations reacts with the $\cdot OH$ and neutralizes its effect. Another point that can be drawn from the results is that increasing H₂O₂ concentration at a constant Fe²⁺ concentration can reduce COD removal efficiency. This point is also justified by reactions 7 and 8.

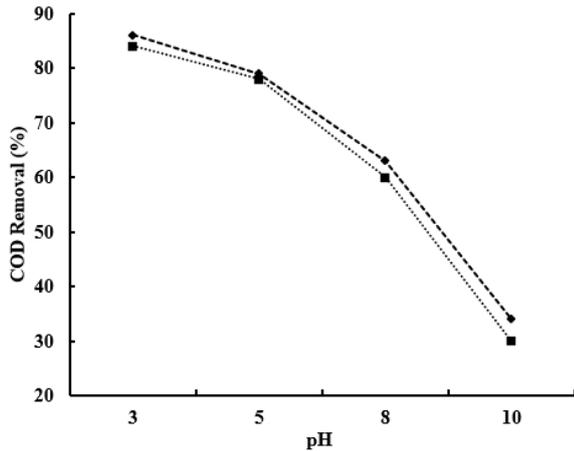


Fig. 3 Effect of different pH on COD removal efficiency in Fenton method (PGU restaurant (■): $Fe^{2+}=1000$ mg/L, $H_2O_2=1000$ mg/L; Shif Island (◆): $Fe^{2+}=500$ mg/L and $H_2O_2=500$ mg/L; Time=90 min)

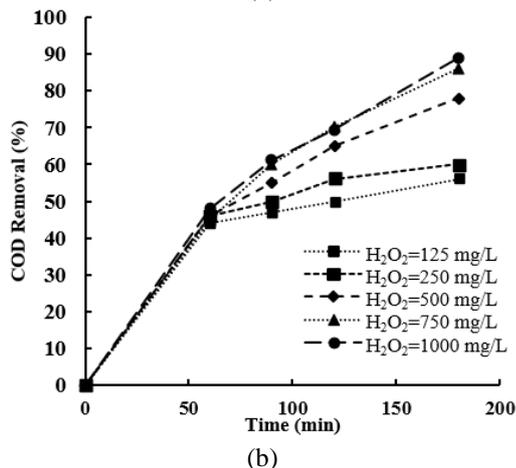
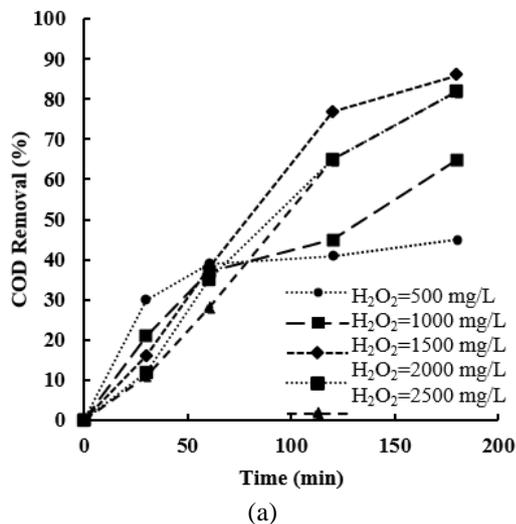


Fig. 4 Effect of time and various H_2O_2 concentrations on COD removal efficiency of: (a) PGU restaurant and (b) Shif Island grey water by UV/ H_2O_2 process; at initial pH = 8

Increasing H_2O_2 concentration will increase the pair collisions of $H_2O_2-H_2O_2$ and $H_2O_2 \cdot OH$ which would

neutralize H_2O_2 effect and eventually reduce the reaction efficiency (Wu *et al.* 2011, Babuponnusami and Muthukumar 2014).

In order to evaluate the results in the best way and to obtain optimal parameters for the highest efficiency in COD removal, $H_2O_2:Fe^{2+}$ ratio can be used. According to the trends of Fig. 2, when the ratio is 1:1, the highest efficiency is observed in COD removal (84% COD removal for PGU restaurant at $Fe^{2+} = 1000$ mg/L and $H_2O_2 = 1000$ mg/L and 86% COD removal at $Fe^{2+} = 500$ mg/L and $H_2O_2 = 500$ mg/L for Shif Island grey water), and if this 1:1 ratio kept constant, increasing the Fe^{2+} and H_2O_2 concentration, will improve COD removal efficiency (Hassanshahi and Karimi-Jashni 2018).

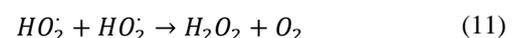
3.1.3 Effect of pH in Fenton process

To investigate the effect of solution pH on COD removal efficiency, the pH range of 3 to 10 were selected for both grey water sources in Fenton process. Fig. 3 shows the results of both PGU restaurant and Shif Island samples. The results indicate that pH around 3 has the highest COD removal efficiency. It should be noted that at this solution pH, the concentration of Fe^{2+} is at its maximum and therefore the rate of Fenton process will be as high as possible (Rivas *et al.* 2001, Babuponnusami and Muthukumar 2011). At pH higher than 5, Fe^{3+} that produced according to the reaction 1, will deposit and hence the concentration of Fe^{2+} in the solution will decrease. Also, at high pH, there is a potential for H_2O_2 decomposition according to the reaction 8 and consequently less $\cdot OH$ is produced.

3.2 Application of UV/ H_2O_2 process for treatment of grey waters

3.2.1 Effect of time and H_2O_2 concentration in UV/ H_2O_2 process

To obtain the effect of time and H_2O_2 concentration on COD removal efficiency in the UV/ H_2O_2 process, different concentration of H_2O_2 from 125 to 2500 mg/L were tested. After the reaction was begun, samples were taken from the solutions. Results of COD removal for Shif Islands and PGU restaurant are shown in Fig. 4. As shown in Fig. 4, COD reduction increases with time significantly at initial according to equation 9 (Muruganandham and Swaminathan 2004), due to the high concentration of H_2O_2 , $\cdot OH$ radical production is much higher, which leads to the accelerating the COD removal process. After that, due to decreasing H_2O_2 concentration and also the creation of side reactions, the rate of COD removal is reduced. About effect of H_2O_2 concentration it must be noted that, increasing the concentration of H_2O_2 has improved the efficiency of the COD removal. But it has an optimum value, and excess H_2O_2 may act in a reverse way.



Indeed, as noted in section 3.1.2 and according to the reaction 7, increasing the concentration of H₂O₂ increases the possibility of the collision between H₂O₂ molecules and •OH radical. Thus, H₂O₂ can acts as •OH scavenger and reduces the process efficiency (Yonar *et al.* 2006).

3.2.2 Effect of pH in UV/H₂O₂ process

The pH values between 3 and 12 were selected for investigating the effect of solution acidity or alkalinity on the COD removal in UV/H₂O₂ process. As shown in Fig. 5, at the pH value of 3 the COD removal is the highest values respect to other values of the solution pH. In fact, in the neutral and alkaline pH, the presence of initial carbonates in grey water has the potential to consume •OH radicals and reduce process efficiency. Also, the possibility of H₂O₂ decomposition increases according to the reaction 8 (Muruganandham and Swaminathan 2004).

3.3. Application of UV/TiO₂ process for treatment of real grey waters

In this method, the UV radiation as well as TiO₂ nanoparticles were used for COD removal from grey water samples. The effects of process time, nanoparticle concentration, and initial solution pH were investigated for getting the best results of COD removal efficiency.

3.3.1 Effect of time and TiO₂ concentration in UV/TiO₂ process

To evaluate the effect of time and TiO₂ concentration on COD removal efficiency in the UV/ TiO₂ process, the TiO₂ concentration was varied between 0.25 to 1.5 g/L. After the reaction was begun, samples were taken from the solution. The results of COD removal were shown in Fig. 6 using UV/TiO₂ process. The trends of the results are the same for both UV/ TiO₂ and UV/H₂O₂ processes. At early times, the rate of COD removal is high but after with the continuation of the reaction, the rate of COD removal reduced. The removal efficiency of COD was increased by increasing photocatalyst concentration. This phenomenon may be due to increasing the number of active sites by increasing photocatalyst concentration. But, the COD removal from grey water of PGU restaurant was decreased when the photocatalyst concentration increased from 1.0 g/L to 1.5 g/L. The results showed that photocatalyst concentration has an optimum value and above this concentration, the photon absorption coefficient will decrease rapidly. The presence of excess amounts of TiO₂ nanoparticles result in the scattering of photons, a decrease in the surface area of the TiO₂ nanoparticles which is exposed to radiation, reduction of light penetration through the solution, and a reduction in the efficiency of the photocatalyst process (Gaya and Abdullah 2008). For the Shif Island grey water, an increase in TiO₂ concentration from 0.5 g/L to 0.75 g/L causes a rapid increase in the COD removal efficiency. But afterward, at higher catalyst concentration, increasing TiO₂ concentration has not a significant impact on the COD removal efficiency. Of course, for Shif Island grey water, due to the lower initial COD concentration, the phenomenon of reduced light transmission in grey water occurred at higher concentrations of TiO₂ nanoparticles.

3.3.2 Effect of pH in UV/TiO₂ process for treatment of real grey waters

In photocatalytic systems, the solution pH is one of the most important parameters. The pH will have an effect

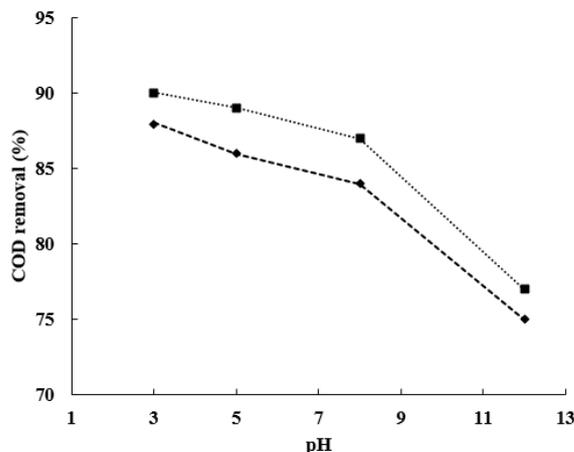


Fig. 5 Effect of different pH on COD removal efficiency in UV/H₂O₂ process (PGU restaurant (♦): H₂O₂ = 1500 mg/L and Shif Island (■): H₂O₂ = 750 mg/L; Time = 180 min).

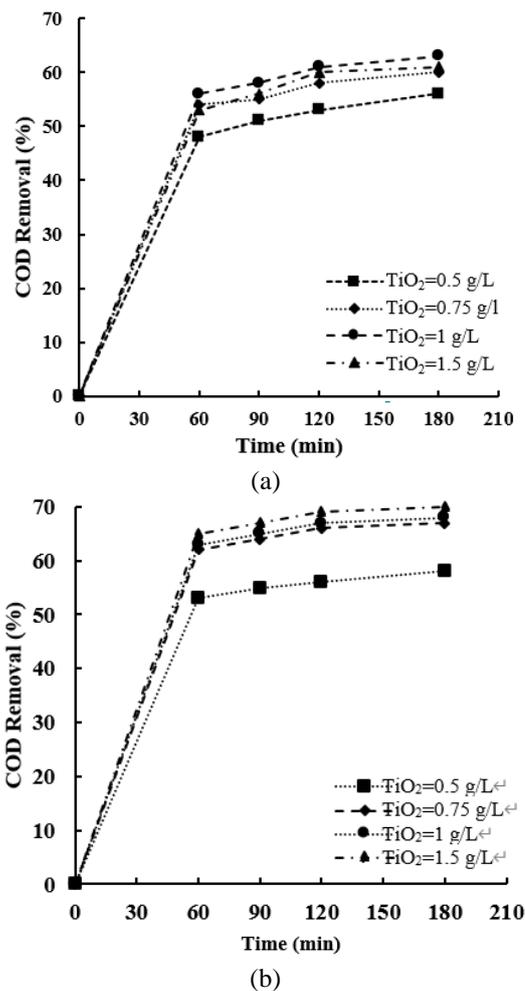


Fig. 6 Effect of time and various TiO₂ concentrations on COD removal efficiency of: (a) PGU restaurant and (b) Shif Island grey water by UV/ TiO₂ process; at initial pH

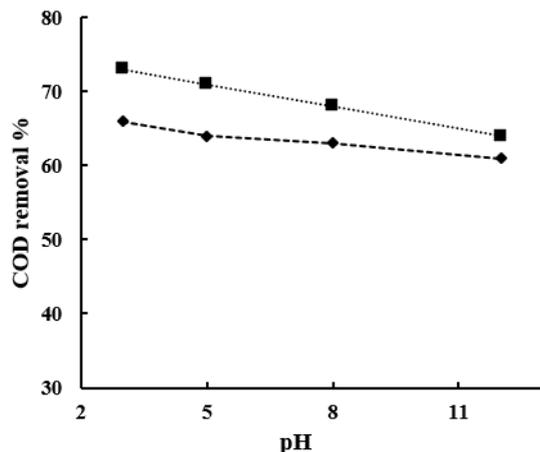


Fig. 7 Effect of different pH on COD removal efficiency in UV/TiO₂ process (PGU restaurant (♦): TiO₂ = 1 g/L and Shif Island (■): TiO₂ = 1 g/L; Time=180 min).

on the charge of photocatalyst particles, photocatalyst agglomeration, and the position of valence/conduction bands. To evaluate the effect of acidity and alkalinity on the photocatalytic process, pH values between 3 and 12 were selected. According to Fig. 7, the best COD removal efficiency was been obtained at pH = 3. The COD removal efficiency has decreased by increasing the pH of the solution. This phenomenon relates to the properties of the photocatalyst. In fact, the point of zero charge (PZC) of the photocatalyst is important in evaluating the initial solution pH effect. At lower and higher pH of the solution respect to the PZC, the net surface charge will be positive and negative, respectively. When the pH of the solution is less than the PZC of photocatalyst particles, more negatively charged compounds (anion) adsorbed on the surface of the photocatalyst due to increasing positive charge of these nanoparticles. Conversely, when the surface charge of the photocatalyst is negative, the tendency to adsorb cations increases in the solution. It should be noted that the grey waters used contains many compounds that contain cations and anions, each of which exhibits different behavior at different pH of the solution. Due to the high concentration of surfactants in the grey water, both wastewaters contain a wide range of anions. The PZC of TiO₂ solution is around 6.5, thus, it can adsorb more anions in acidic pH of the solution. Also, the pH of solution can cause increasing radical scavenging. Therefore, the process has higher efficiencies in acidic solution pH (Kang *et al.* 2011, Boyjoo *et al.* 2012).

3.4 Comparison of the best COD removal efficiencies of processes

The optimal COD removal results for both grey water sources with the Fenton, UV/H₂O₂, and UV/TiO₂ processes are summarized in Table 2. Based on the results on both PGU and Shif Island grey water samples, 90 min and 180 min process time is the best operating time for Fenton and UV/H₂O₂ as well as UV/TiO₂, respectively. Moreover, the results showed that high acidity of samples (pH = 3) is the most favorable solution pH for COD removal in all

Table 2 Comparison of optimal COD removal efficiencies (%) for Fenton, UV/H₂O₂, and UV/TiO₂ processes for PGU restaurant and Shif Island grey waters

Grey water	Method		
	Fenton	UV/H ₂ O ₂	UV/TiO ₂
PGU restaurant	84	88	66
Shif Island	86	90	73

processes. Based on the results, the UV/H₂O₂ process for both grey waters has the highest COD removal efficiency under the optimum conditions. These results are acceptable in comparison with previous literature results such as: 87% COD reduction of gray water using UV/H₂O₂ process (Chin *et al.* 2009), 65% TOC removal of gray water using photocatalytic process (Sanchez *et al.* 2010) and removing 95% of COD content of grey water using Fenton method (Tony *et al.* 2016).

4. Conclusions

In this research, besides of the investigation of the ability of Fenton, UV/H₂O₂, and UV/TiO₂ processes for COD removal of grey water, various parameters including time, Fe²⁺ content, H₂O₂ and TiO₂ concentrations, and pH of aqueous solution were evaluated. Experimental results showed that for the Fenton process, the ratio of Fe/H₂O₂ set to 1 was the best, and it was observed that at this ratio, increasing Fenton concentration can enhance the COD removal efficiency. Moreover, it was found that optimal acidity for this process was pH = 3. For the UV/H₂O₂ process, it was also found that increasing the H₂O₂ concentration in a limiting value could raise the COD removal efficiency. The limiting value of H₂O₂ concentration corresponded to the acidity of the aqueous solution that its optimized value is pH = 3. Also, in the photocatalytic method, it was found that by increasing the photocatalyst concentration, COD removal efficiency will increase. But over-optimal concentration can reduce the efficiency of the process. In fact, it can be concluded that the UV/H₂O₂ process for both Shif Island and PGU restaurant has the highest COD removal efficiency that corresponds to 88% and 90%, respectively. Of course, for discharging the treated wastewaters to the environment or treat/eliminate the intermediaries, additional processes such as adsorption, membrane or novel processes probably needed because the degradation process must have non-toxic end products. In the other hand, various parameters plus ultimate COD of treated wastewaters is considered in different environmental standards for discharging treated wastewaters, and this subject can be investigated in future researches.

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