Journal of Advances in Environmental Health Research

Removal of p-chlorophenol from aqueous solution using ultraviolet/zerovalent-iron (UV/ZVI)/persulfate process

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Original Article

Abstract

In this study, degradation of p-chlorophenol (p-CP) was evaluated using persulfate (PS) activated zerovalent iron (ZVI) based ultraviolet (UV) in a bench scale photoreactor. The effect of operational parameters such as solution pH (3, 7, and 11), reaction time (0-60 minutes), ZVI dosage (0.15, 1.25, 0.5, 1, and 1.5 mM), PS concentration (0.5, 1.5, 2, 2.5, 3, and 4 mM), and initial p-CP concentration (0.22, 0.44, 0.88, 1.32, and 1.76 mM) were examined on the degradation of p-CP in batch experiments. The experimental results indicated that the p-CP removal rate significantly depends on operational parameters. The highest p-CP removal rate was achieved after 45 minutes (> 0.99%) in pH = 3, ZVI = 1 mM, and PS = 3 mM, and with initial p-CP concentration = 0.44 mM. The results revealed that excess amount of PS and ZVI could reversely affect p-CP removal efficiency. In addition, an increase in p-CP initial concentration from 0.22 to 1.76 mM significantly decreased its removal rate. This study indicated that PS activated ZVI based UV process is practically feasible for the effective degradation of p-CP in aqueous solution.

KEYWORDS: Zerovalent Iron, Sulfate Radical, Persulfate Activation, p-Chlorophenol

Date of submission: 17 Sep 2015, ***Date of acceptance:*** 11 Dec 2015

Citation: Sharifi Z, Samadi MT, Seid-Mohammadi A, Asgari G. **Removal of p-chlorophenol from aqueous solution using zerovalent-iron-based ultraviolet (UV/ZVI)/persulfate process.** J Adv Environ Health Res 2016; 4(1): 42-8.

Introduction

Aromatic compounds are common pollutants existing in the effluent of different industries in concentrations ranging from trace quantities to thousands of milligrams per liter.[1-3] P-chlorophenol (p-CP) as an important chlorophenol is released into the environment through a number of routes, including discharge of wastewater generated from refineries, petrochemical industries, pesticide and herbicide production industries, antimicrobial agent manufacturers, production of compounds like 2-bezyl-4-chlorophenol, and industrial wood preservatives in the range of 150 µg/l to 100–200 mg/l.[4]

This pollutant is toxic and largely non-biodegradable, and poses serious risks to the environment, particularly when released into natural waters which enter the human body through the skin, breathing, and digestion. Due to its toxic and corrosive natures, it causes irritation in the eyes, skin, throat, and nose, and coughing, wheezing, and respiratory problems. Long-term exposure to this chemical results in headaches, exhaustion, anxiety, liver and kidney disorders, paresis, nausea, and finally, coma and death.[5,6] Therefore, it is important to efficiently remove or degrade this aqueous pollutant before the wastewater is discharged into the environment. A variety of physical,

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physicochemical, chemical, and biological methods have been developed for the degradation of chlorophenols in wastewater. However, the production of chemical or biological sludge, energy consumption, and transference of the pollutant from the liquid phase to the solid phase may limit the practical application of these techniques. Moreover, many chlorophenols are non-biodegradable and recalcitrant water pollutants. To date, many studies have investigated oxidative degradation of chlorophenols in wastewater by means of advanced oxidation processes (AOPs) such as photocatalytic oxidation, photo-Fenton, electro-Fenton, ultraviolet (UV)/H_2O_2, MW/H_2O_2, and ozonation processes based on the production of highly reactive radicals which are responsible for the degradation and mineralization of organic compounds.[5-11]

Based on the results of publications,[5] UV-based AOPs have been proved to be highly effective in photochemical degradation of p-CP. However, the use of oxidant agents [i.e., H_2O_2, persulfate ($S_2O_8^{2-}$), periodate (IO_4^-)] has been recommended in combination with UV for high rate of degradation of herbicides and toxic organic compounds instead of direct photolysis.[5,12,13] Among all of the community oxidants, persulfate receives the most attention due to its higher oxidation-reduction potential (E^o = 2.01 V) than H_2O_2 (E^o = 1.76 V).[14,15] Compared to other oxidants, PS has numerous advantages, i.e., high solubility in water, non-selective reactivity, widespread reactivity with environmental communities, highly stable in aqua solution, and economical.[15] When the PS anion is activated with UV, US, transition metal, and heat, it produces a stronger oxidant and reactive sulfate radical ($SO_4^{\bullet -}$) with an oxidation-reduction potential of 2.6 V according to equations 1 and 2.[16]

$$S_2O_8^{2-} + \frac{\text{heat}}{\text{UV}} \rightarrow 2SO_4^{\cdot -} \quad (1)$$

$$S_2O_8^{2-} + Me^{n+} \rightarrow SO_4^{\cdot -} + SO_4^{2-} + Me^{(n+1)+} \quad (2)$$

Moreover, hydroxyl radicals can be produced in PS aqueous solution which may participate in the oxidation of pollutants according to equations 3 and 4.[17]

$$SO_4^{\cdot -} + H_2O \rightarrow SO_4^{2-} + {}^{\cdot}OH + H^+ \quad (3)$$

$$SO_4^{\cdot -} + OH^- \rightarrow SO_4^{2-} + {}^{\cdot}OH \quad (4)$$

Both sulfate and hydroxyl radicals are strong oxidants; however, sulfate radicals have a longer half-life and more effective oxidant than hydroxyl radicals. This is due to the fact that $SO_4^{\bullet -}$ operates primarily via oxidation, while $^{\bullet}OH$ may also act by hydrogen addition.[18]

Recently, Fe^{2+}, Fe^{3+}, and Fe^{o} have been generally considered as famous transition metal activators which activate PS to generate sulfate radicals at ambient temperature according to equations 2, 5, and 6.[19]

$$S_2O_8^{2-} + Fe^{2+} \rightarrow Fe^{3+} + SO_4^{2-} + SO_4^{\cdot -} \quad (5)$$

$$Fe^0 + S_2O_8^{2-} \rightarrow Fe^{2+} + 2SO_4^{2-} \quad (6)$$

When Fe^{2+} is used as an activator due to its environmentally benign nature and cost effectiveness, the overall reaction between Fe^{2+} and PS is described by equation 5. However, it has been demonstrated that undesired and unproductive radial consumption in the presence of excessive Fe^{2+} or rapid conversion of Fe^{2+} to Fe^{3+}occurs via equation 7.[12,20]

$$SO_4^{\cdot -} + Fe^{2+} \rightarrow Fe^{3+} + SO_4^{2-} \quad (7)$$

Therefore, PS activation using Fe^{2+} may be limited through PS radical scavenging when excessive Fe^{2+} is employed according to equation 7.

To resolve this problem, zerovalent iron (ZVI) was proposed as an activator or chelating agent.[12] Previous studies indicated that ZVI-activated PS decomposition proved to be effective on the degradation of polyvinyl alcohol,[21] p-CP,[12] and Azo dyes. They predicted that in the presence of PS, Fe^{o} is a suitable source of Fe^{2+} activating PS according to equations 8 and 9.[20,22]

$$Fe^0 + 2H^+ \rightarrow Fe^{2+} + H_2 \quad (8)$$

$$Fe^0 + S_2O_8^{2-} \rightarrow Fe^{2+} + 2SO_4^{2-} \quad (9)$$

To the best of our knowledge, no studies have been conducted on the degradation of p-CP via ZVI and UV activated PS. Therefore, this study was performed to evaluate the efficacy of p-CP degradation using UV/PS/ZVI via batch experiments. The effect of operational parameters, such as the solution pH, persulfate and ZVI dosages, and initial p-CP concentration, on the degradation of p-CP was evaluated.

Materials and Methods

P-CP, C_6H_5ClO, (> 99%), $Na_2S_2O_8$ (> 99%), 4-aminiantipyyrine (> 99%), NH_4OH (> 99%), KH_2PO_4 (> 99%), K_2HPO_4 (> 99%), and potassium ferricyanide (> 99%) were purchased from Merck Chemical Company (Germany). The ZVI (> 99% pure, particle size of 21 nm and surface area of 0.14 m^2/g) used was purchased from Sigma-Aldrich (USA).

The photocatalytic degradation experiments were performed in a cylindrical reactor containing 2.5 l of fresh p-CP made of a very smooth stainless steel (Pakan Ab Co., Iran). UV radiation was achieved using UV lamp (low-pressure Hg vapor lamp of 55 W, radiation flux used for only degradation of 253.7 nm, lifetime of 5000 hours, and UV radiation intensity equal to 50,000 μWs/cm from Philips Company, USA). The UV radiation source was placed in the hallow quartz located at the center of the reactor. The schematic diagram of the experimental apparatus is shown in figure 1.

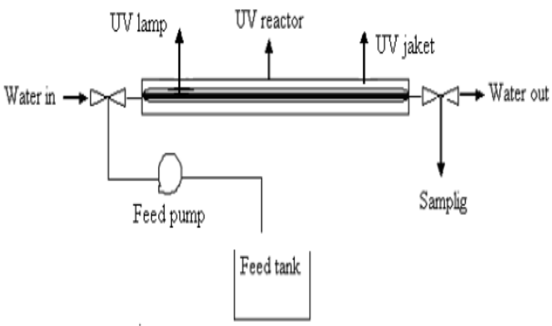

Figure 1. Plan of the experimental apparatus

Batch experiments were performed with stock solution of p-CP (0.1 M) and PS (1 M) which was prepared by diluting the corresponding amount of dried p-CP and sodium persulfate in ultrapure deionized water as solvent perior to each batch experiment. Several sets of the experiments were conducted to determine the effect of operational parameters such as pH of solution, PS concentrations, dosages of ZVI, and initial p-CP concentration. Unless otherwise stated, the batch experimental procedures were performed as follows: (1) The pH of solution was adjusted with 0.1 M HCl and/or NaOH; (2) After the addition of ZVI and PS, the solutuion was immidiately agitated with a mechnical stirrer at 350 rpm for 30 minutes before being injected into the reactor in the storage tank; (3) At regular time intervals, 25 ml of samples were taken from the reaction vessel and immidiately analyzed to avoid further reaction. The p-CP removal degradation was examined with UV, UV/ZVI, UV/PS, and ZVI processes.

To determine the effect of operational parameters, first, solution pH values ranging from 3 to 11 were examined while ZVI, PS, and p-CP were constant at 0.5 mM, 2 mM, and 0.44 mM, respectively. Second, the p-CP solution was set at the optimum pH value and different PS concentrations of 0 mM to 4 mM were studied with constant ZVI and p-CP values of 0.5 mM and 0.44 mM, respectively. Next, different ZVI dosages ranging from 0 mM to 1.5 mM were evaluted in the optimal PS concentration and pH value, and initial p-CP concentration of 0.44 mM. Finally, the effect of different initial p-CP concentrations ranging from 0.22 to 1.76 mM were examined in optimal PS and ZVI concentrations, and pH of solution. In the subsequent stage, experiments with UV irradiation alone were carried out in the UV reactor by adding 0.44 mM of p-CP.

In order to identify the effect of ZVI alone on p-CP removal rate, batch experiments were performed in a rotary shaker at 125 rpm and room temperature, optimum amount of ZVI, and p-CP concentration of 0.44 mM.

Variation in p-CP concentration was determined using ultraviolet-visible spectrophotometry (UV–VIS) at the wavelength of 500 nm according to standard methods for the examination of water and wastewater.[23] The p-CP removal efficiency was defined using the equation 10:[13]

$$R\% = \frac{(C_0 - C_1)}{C_0} 100 \qquad (10)$$

Where C_0 is the initial concentration of p-CP (mg/l), Ct is the instant concentration of p-CP (mg/l), and R% is the percentage of p-CP removal rate.

Results and Discussion

Influence of solution pH

Previous researches have demonstrated that for sulfate radical-based AOPs, the total radical amount is pH-dependent.[1] Moreover, it has been demonstarated that pH has a key role in iron catalyzed and iron mediated reactions.[24] Therefore, a set of experiments was performed to evalute the effects of different pH values (3, 7, and 11) on p-CP degradation with UV-based ZVI activated PS.

Figure 2 displays time-dependent changes in p-CP as a function of the initial pH (ZVI: 0.5 mM, persulfate: 2 mM, p-CP: 0.44 mM). The results indicated that the degradation of p-CP was siginficantly influenced by the pH of solution. As can be seen in figure 2, after 60 minutes of reaction, p-CP removal efficiency at pH of 3, 7, and 11 were 88.5, 71.0 and 52.6 percent, respectively; and, the highest idential p-CP removal rate was obtained at pH of 3.

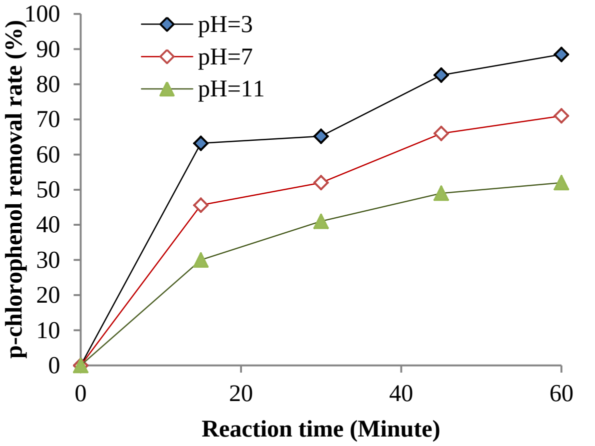

Figure 2. Influence of initial pH on the degradation of p-chlorophenol
Initial experiments conditions: zerovalent iron = 0.5 mM, persulfate = 2 mM, p-chlorophenol = 0.44 mM

The results demonstrated that the precipitation of Fe^{3+} ions occurred when the pH of solution was higher than 4. Oxyhydroxides of Fe^{3+}, such as $FeOH^{2+}$, $Fe(OH)^{2+}$, and $Fe_2(OH)_2^{4+}$, may be produced which have low efficency in the generation of sulfate radical.[25] Additionally, the conversion of $SO_4^{\cdot-}$ from PS anions can be accelerated by increase in the acidity of the solution according to equations 11 and 12.

$$S_2O_8^{2-} + H^{+-} \rightarrow HS_2O_8^{-} \quad (11)$$

$$HS_2O_8^{-} \rightarrow SO_4^{\cdot-} + SO_4^{2-} + H^{-} \quad (12)$$

The above results were similar to the findings of the studies by Rastogi et al.,[26] Hussain et al.,[12] and Zhou et al.,[16] who investigated the impact of pH on the oxidation of organic and inorganic pollutants through ZVI activated PS process.

Effect of ZVI dosages

The effect of different ZVI dosages on p-CP removal was investigated at 0.15, 0.25, 0.5, 1, and 1.5 mM activated by 2 mM PS empolyed in the batch experiments at optimum pH while keeping concentration of p-CP constant at 0.44 mM. According to the results illustrated in figure 3, the p-CP removal rate increased with an increase in ZVI dosage from 0.15 to 1 Mm. The p-CP removal rate was 88.5 84.2, 88.4 and 93.0 percent at 0.15, 0.25, 0.5 and 1 mM of ZVI, respectively.

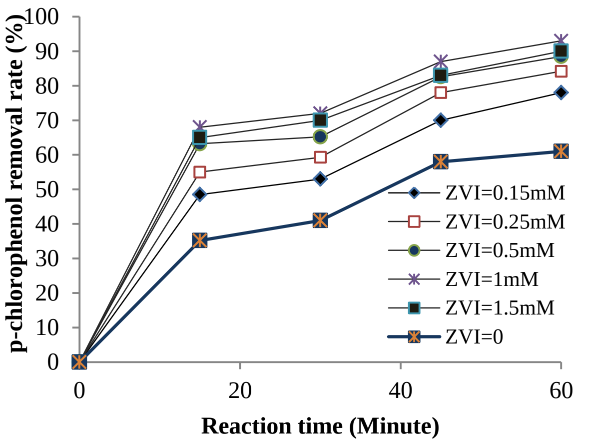

Figure 3. Influence of initial zerovalent iron (ZVI) dosage on the degradation of p-chlorophenol
Initial experiment conditions: pH = 3, persulfate = 2 mM, p- chlorophenol = 0.44 mM

The UV/PS experimental results showed that the degradation rate of p-CP was 61% after 60 minutes of irradiation without ZVI. The fact that higher p-CP degradation efficacy was achieved at high ZVI dose is mainly attributed to the higher generation of sulfate radicals with increasing ZVI dosage.[12,16,25] Hussain et al. showed that the addituon of ZVI was highly effective in the degradation of p-chloroaniline and maximum degradation rate was achieved at ZVI of 4 g.[12] Similarly, Zhou et al. reported that a high rate of oxidation of diuron was achieved when optimum dosage of ZVI (5 mM) was used.[16] However, our results revealed that further increase in the ZVI dosage (above 1 mM) exhibited no enhancment in p-CP removal rate. Therefore, ZVI dosage of 1 mM was considerd as the optimal dosage in this research. A possible reason for the inhibitory effect was that the generation and consumption of sulfate radicals by iron anions reached a balance immediately when the iron anions dosage was higher than 1 mM, leading to the exhibition of no enhancement.[27]

Effect of initial PS concentration

Figure 4 shows the effect of different concentrations of PS on degradation rate of p-CP as function of different contact times. Six different concentrations of PS (0.5, 1, 2, 2.5, 3, and 4 mM) activated with 1 mM of ZVI were examined (p-CP constant at 0.44 mM and optimum pH).

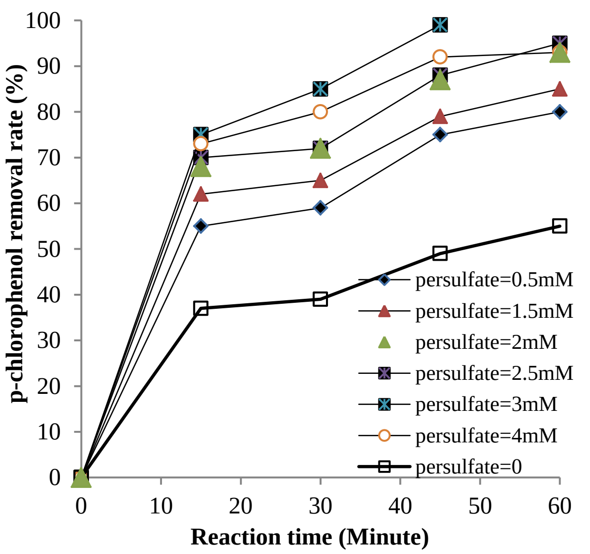

Figure 4. Influence of initial persulfate concentration on the degradation of p-chlorophenol
Initial experiment conditions: pH = 3, zerovalent iron = 1 mM, p-chlorophenol = 0.44 mM

From figure 4 it is apparent that p-CP removal rates first increased with the increase in PS concentration from 0.5 mM to 3 mM. P-CP removal rates were 80, 85, 93, 95 and 99 percent when initial concentrations of PS were 0.5, 1, 2, 2.5, 3, and 4 mM after 60 minutes of contact time, respectively. As can be seen in figure 4, the percentage of p-CP degradation at 60 minutes was 55% in UV/ZVI process when carried out at optimal ZVI concentration without adding PS.

In the PS-based photochemical oxidation processes, the initial concentration of PS plays an important role in the degradation of organic and inorganic pollutants.[13,22] The enhancment of p-CP degradation with the addition of PS is due to the increase in sulfate radicals (E^o = 2.6 V) as an oxidizing agent. A similar study by Seid-Mohammadi et al. revealed that the removal efficiency of pollutants significantly increased in the persence of PS and was influenced by its concentration.[13] However, with the increase of PS concentration up to 3 mM, p-CP removal rate gradually decreased. An excessively high initial PS concentration may lead to the production of higher quantity of sulfate radicals that could inhibit the p-CP removal efficiency according to equation 13.

$$SO^{-}_{4(aq)} + S_2O^{2-}_{8(aq)} \rightarrow SO^{2-}_{4(aq)} + S_2O^{-}_{8(aq)} \quad (13)$$

Effect of initial p-CP concentration

To evaluate the effect of different concentrations of p-CP on its removal efficiency, the 5 p-CP concentrations of 0.22, 0.44, 0.88, 1.32, and 1.76 mM were studied at optimum conditions determined in previous steps (PS = 3 mM, ZVI = 1 mM, and pH = 3) at contact time ranging from 0 to 60 minutes. Figure 5 shows the effect of initial p-CP concentration on its removal efficiency.

As can be seen in the figure, as initial concentration of p-CP increased, the p-CP removal efficiency significantly decreased. Complete degradation of p-CP was observed in 30 and 45 minutes when initial concentrations of p-CP were 0.22 and 0.44 mM, respectively. Correspondingly, the p-CP removal rate at 60 minutes contact time was 80, 71, and 63 percent when the initial p-CP was 0.88, 1.32, and 1.76 Mm, respectively. This negative effect can be explained through considering that when initial p-CP concentration was lower, sulfate radical production rate was higher than consumption rate by p-CP degradation, leading to its higher removal efficiency. However, an insufficient rate of sulfate radicals was used up when initial p-CP concentration increased, resulting in lower p-CP removal.[22,27]

Similarly, Seid-Mohammadi[13] and Weng and Tsai[22] have shown that the degradation of organic pollutants was decreased with an increase of initial p-CP concentration.

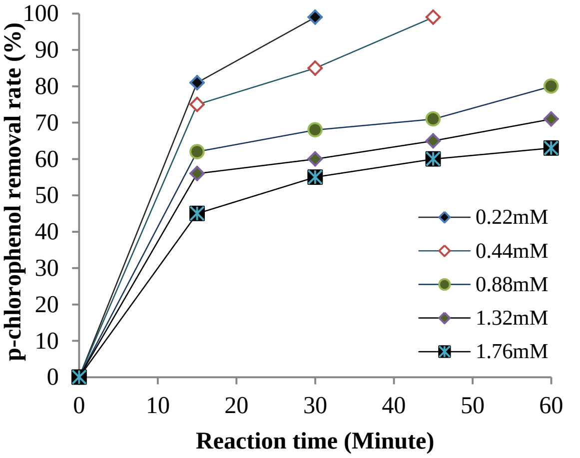

Figure 5. Influence of initial p-chlorophenol concentration on its degradation
Initial experiments conditions: pH = 3, zerovalent iron = 1 mM, PS = 3 mM

Degradation Rate

The reduction of p-CP by different types of oxidation processes followed first order rate decay kinetics according to equation 14 as shown in figure 6.[1]

$$\text{Ln}\frac{C}{C_0} - \text{-Kt} \quad (14)$$

In equation 14, C° and C are the p-CP concentrations at 0 and t time and K° is the expected pseudo-first-order rate constant. The rate constants were 0.031, 0.003, 0.057, 0.3, and 0.04 minute^{-1} for UV, ZVI, UV/PS, UV/PS/ZVI, and UV/ZVI processes, respectively. Optimum conditions of processes were used to illustrate first order changes.

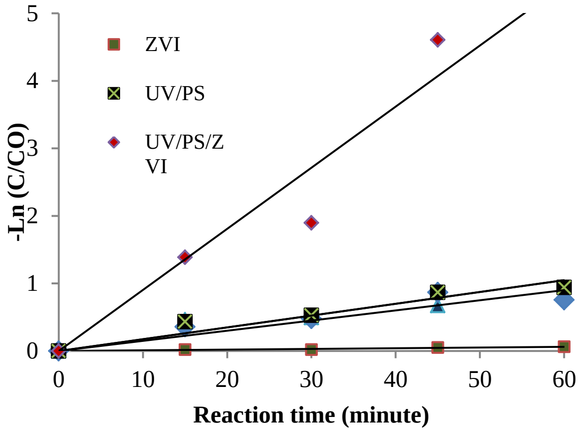

Figure 6. First order plot for degradation of p-chlorophenol using different oxidation processes in optimum conditions

Conclusion

The degradation of p-CP was investigated through PS activated with ZVI based UV process under different operation parameters. This oxidation process to generate sulfate radicals has been demonstrated to be a more effective oxidant reagent for degrading p-CP. The maximum degradation rate of p-CP was 88.5% and was attained at pH of 3 in 60 minutes. Moreover, p-CP was completely (> 0.99%) degraded within 45 minutes with PS:ZVI molar ratio of 3:1 at the initial p-CP concentration of 0.44 mM. The p-CP removal rate was inversely related to its initial concentration; p-CP removal rate decreased with the increase of its initial concentration. The degradation of p-CP by different types of oxidation processes followed first order rate decay kinetics. Furthermore, the control system (ZVI only) showed that only a small amount of p-CP (6%) was removed by ZVI alone.

Conflict of Interests

Authors have no conflict of interests.

Acknowledgements

The authors wish to express their graduate to the Vice Chancellor of Research of Hamadan University of Medical Sciences for financial supporting (Grant number 9403231397).

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