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Detoxification of As(III) in aqueous media by using photo-Fenton method

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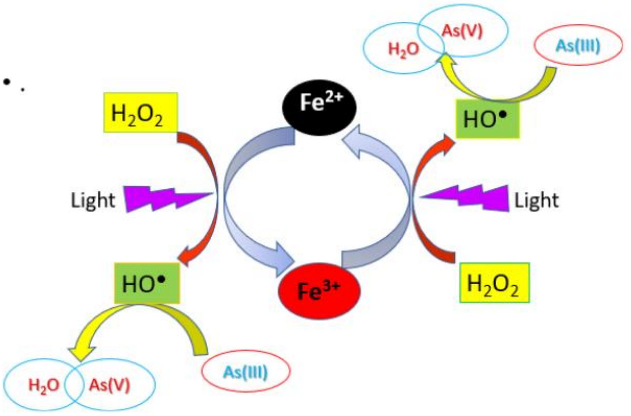
Graphical abstract



Abstract

In this paper, photo-Fenton method has been systematically examined to oxidize the toxic and mobile As(III) in the solution by $^{\bullet}$OH radicals resulted from the reactions between Fe^{2+} and H_2O_2 under UV light, to form the less toxic and insoluble As(V). The effects of various experimental parameters including initial Fe^{2+} and H_2O_2 concentrations, working pH, and contact time were studied through a batch experiment. The concentration of As(V) resulted from the photo-oxidation was determined by visible spectrophotometry method based on the formation of arsenate molybdate solution. The results of the research attributed noticeably that the As(III) could be oxidized through photo-Fenton process to form As(V) that was assigned by a considerable decline in the concentratio n. The maximal oxidation of As(III) with 10 mg L^{-1} of the concentration in 50 mL of the aqueous solution, that was about 85%, can be reached in the presence of Fe^{2+} 10^{-2} mole L^{-1}, H_2O_2 5. 10^{-2} mole L^{-1} at the working pH 3 and within 3 h of the reaction time. It has been also detected that for reaching the permissible level (lower than 0.01 mg L^{-1}), three stages of the photo-Fenton process were required.

Keywords: Photo-oxidation, photo-Fenton, As(III), As(V)

1. Introduction

The presence of arsenic in the environment is mainly from minerals and geogenic sources, but human activities such as mining, burning of fossil fuels, medicine, use of arsenic-containing chemicals in agriculture (pesticides, herbicide, insecticides,), and wood preservation also cause arsenic distribution in the environment (Almohammadi *et al.,* 2017; Garcia *et al.,* 2021; Iervolino *et al.,* 2019; Kunthia *et al.,* 2014; Saleh *et al.,* 2021; Shumlas *et al.,* 2016; Yang *et al.,* 2019).

The presence of arsenic in the environment can contaminate the ground water, and in turn to contaminate drinking water, rice, fruits or vegetables (Sharman and Shon, 2019). Consuming drinking water and food contaminated by arsenic can create health problems such as nerves damaged, circularly problem in skin, increasing cancer risks of kidney and liver, pigment change and skin damaged (Almohammadi *et al.*, 2017; Ding *et al.*, 2015; Hasanpour and Hatami, 2020; Iervolino *et al.*, 2019; Kunthia *et al.*, 2014; Lescano *et al.*, 2011; Saleh *et al.*, 2021; Sharman and Shon, 2019; Sorlini *et al.*, 2010; Yang *et al.*, 2019).

In the solution, arsenic can be found as arsenite or As(III) and arsenate or As(V). Compared to As(V), As(III) is more toxic and mobile, causing it is more difficult to remove by coagulation or precipitation (Ding *et al.,* 2015; Garcia *et al.,* 2021; Iervolino *et al.,* 2019; Kunthia *et al.,* 2014; Lescano *et al.,* 2011; Li *et al.,* 2019; Pozdnyakov *et al.,* 2017; Qin *et al.,* 2016; Saleh *et al.,* 2021; Saleh *et al.,* 2021; Shumlas *et al.,* 2016). Therefore, oxidation of As(III) to As(V) is believed as the most suitable method, that does not only diminish the concentration, but also convert the toxic As(III) into the less toxic As(V), suggesting a detoxification process of As(III).

A variety of treatment methods which oxidize As(III) to As(V) have received greatly attention, that are heterogeneous photocatalytic oxidation in the presence of TiO_2 (Iervolino *et al.,* 2019), modified TiO_2 (Garcia *et al.,* 2019; Qin *et al.,* 2016; Saleh *et al.,* 2021), Bi_2WO_6/bentonite (Yang *et al.,* 2019), kaolinite clay (Ding *et al.,* 2015), birnessite (Shumlas *et al.,* 2016), iron and fulvic acid (Pozdnyakov *et al.,* 2017), as well as homogenous photo-oxidation with hydrogen peroxide (Lescano *et al.,* 2011; Sorlini *et al.,* 2010), electro-catalytical oxidation (Li *et al.,* 2019), and ozonation

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(Kunthia *et al.,* 2014). The photo-oxidation of As(III) was induced by OH radicals that were released during the light exposure process. The OH radical is known as a powerful oxidation agent, as indicated by its high oxidation potential (E^o): 2.8, resulting in the effective oxidation (Garcia *et al.,* 2019; Qin *et al.,* 2016; Saleh *et al.,* 2021).

Compared to the heterogeneous oxidation, the homogenous photo-Fenton-like (UV + H_2O_2) processes posse low mass-transfer resistance and favor to faster degradation of pollutants (Lescano *et al.,* 2011; Sorlini *et al.,* 2010). In the photo-Fenton, OH radicals are not only produced from reaction between Fe(II) with H_2O_2, but can also be resulted from the H_2O_2 irradiation with UV light, as presented as equations (1) and (2) (Azhdarpoor *et al.,* 2015; Buitrago *et al.,* 2020; Ebrahiem, *et al.,* 2015; O'Dowd and Pillai, 2020; Reza *et al.,* 2016; Villages-Guzman, *et al.,* 2017; Wahyuni *et al.,* 2016; 2019).

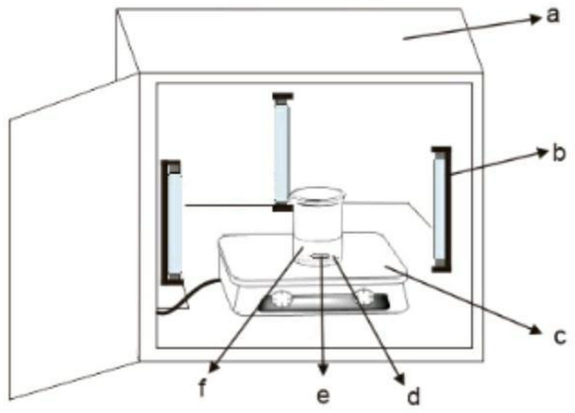

Figure 1. A set of apparatus used for As(III) photo-oxidation processes, composed of: (a) melamine box, (b) UV lamps, (c) magnetic stirrer plate, (d) beaker glass, (e) magnetic stirrer bar and (f) sample solution.

$$Fe^{2+}+H_2O_2 \rightarrow Fe^{3+}+2^{\bullet}OH+^{-}OH \quad (1)$$

$$H_2O_2+UV_{light} \rightarrow 2^{\bullet}OH \quad (2)$$

Due to the rich $^{\bullet}OH$ radicals provided, photo-Fenton method has accepted intensively attention and has been frequently used to treat a large number of organic pollutants such as dyes (Reza *et al.,* 2016), cosmetic residual in industrial wastewater (Ebrahiem *et al.,* 2015), anionic surfactant in the laundry wastewater (Wahyuni *et al.,* 2016), and amoxicillin (Buitrago *et al.,* 2020) with satisfying results. Additionally, some other studies have also examined photo-Fenton method to destroy bacteria (Villages-Guzman, *et al.,* 2017; O'Dowd and Pillai, 2020), leach several heavy metals from sediments (Azhdarpoor *et al.,* 2015) and oxidize Pb(II) in aqueous media to form the insoluble Pb(IV) (Wahyuni *et al.,* 2019). The major advantage of photo-Fenton process is that the reagent components are safe to handle and environmentally benign. In addition, highly complicated apparatus and pressurized systems are not required for the oxidation process, making it a feasible technology for applying directly from laboratory scale to large scale (Ebrahiem *et al.,* 2015).

However, to the best of our knowledge, very few reports regarding the of photo-Fenton method to oxidize As(III) becomes As(V) are traceable (Sorlin *et al.,* 2010; Lescano *et al.,* 2011). Under the circumstance, the main aim of the present study is to explore the possibility of photo-Fenton method in detoxifying As(III) through oxidation into As(V). By oxidation, the concentration of As(III) in the media can be depleted into an acceptable level, at the same time the toxic As(III) can also be detoxified due to As(V) formation. Factors to control the photo-Fenton process including the concentrations of Fe^{2+} and H_2O_2, the working pH and the reaction time are optimized through batch laboratory experiment to get the best condition for As(III) photo-oxidation.

2. Experimental Section

2.1. Materials

The materials including $FeSO_4.7H_2O$ (Panreac), H_2O_2 (Merck, Perhydrol, 30% w/w), As_2O_3, $NaAsO_3$, HCl, Ammonium molybdate, ascorbic acid, and NaOH from Merck were used without purification because the chemicals are in the high purity reagent grade. All solutions were prepared by dissolving requisite quantity in the deionized water. The $FeSO_4.7H_2O$ and H_2O_2 solution were functioned as Fenton agent. The As_2O_3 was used as a starting material of As(III), while the $NaAsO_3$ was used to make standard solution of As(V). The ammonium molybdate and ascorbic acid were coloring agents in the As(V) determination by using UV/Visible spectrophotometer. HCl and NaOH were used for pH adjustment. A Spec Cord 200 Plus UV/Visible spectrophotometer was used for analysis of As(V) in the solution.

2.2. Methods

All photo-Fenton experiments were carried out in a batch mode laboratory scale unit, using a set of apparatus as displayed in Figure 1. Into a 250 mL beaker glass filled by 100 mL of the solution containing As(III) 10 mg/L was added with HCl solution to adjust at pH 3. Then, Fe^{2+} ions and H_2O_2 solution were added into beaker glass to produce hydroxyl radicals. Subsequently, the mixture was subjected to magnetic stirring and UV radiation (254 nm wavelength) in the apparatus (Figure 1) for a desired time. Under the irradiation time, hydroxyl radicals were also resulted from the photolysis of H_2O_2 and the As(III) in the solution should be oxidized.

The solutions after being photo-Fenton processed were analyzed by using visible spectrophotometer based on reaction of As(V) with ammonium molybdate (Iervolino *et al.,* 2019). For that purpose, 10 mL of the solution was added with ammonium molybdate solution and ascorbic acid to form green solution. The absorbance of the green solution was observed at 560 nm of the wavelength. The As(V) concentration was calculated by interpolating its absorbance to the corresponding standard curve, that was constructed based on the observed absorbances of a series of standard solutions. The procedure was duplicated for processes with different Fe^{2+} and H_2O_2 concentration, working pH, and reaction time, When one parameter was varied, the other parameters were kept to be constant.

3. Results and discussion

3.1. Effect of Fe^{2+} concentration

The role of Fe^{2+} is very important in the photo-Fenton process, since the ion acts to initiate and catalyze the decomposition of H_2O_2, resulting in the generation of •OH radical, as presented by reaction in Eq. (1). Simultaneously, formation of •OH radical due the decomposition of H_2O_2 by UV light also occurred (Eq. 2) . Accordingly, the effect of Fe^{2+} concentration on the oxidation of As(III) was observed and the results were displayed in Figure 2.

$$Fe^{2+}+H_2O_2 \rightarrow Fe^{2+}+{}^{\bullet}OH+{}^{-}OH \qquad (1)$$

$$H_2O_2 + UV \rightarrow 2{}^{\bullet}OH \qquad (2)$$

It is notable that the increase of Fe^{2+} concentration up to 10^{-2} mole L^{-1} can raise the efficiency of As(III) oxidation to reach maximum level, but the opposite effect is observed when the Fe^{2+} concentration was further increased. The extension of Fe^{2+} concentration could generate more •OH radicals, and so improvement of the oxidation. In contrast, the excessive Fe^{2+} could act as •OH radical scavenger (Azhdarpoor *et al.,* 2015; Buitrago *et al.,* 2020; Ebrahiem *et al.,* 2017; Reza *et al.,* 2016; Wahyuni *et al.,* 2016; 2019), following reaction (Eq 3), that caused the •OH radicals diminished. Then, Fe^{3+} and ^{-}OH resulted from Eq.(1), were allowed to mutually react to form un-soluble $Fe(OH)_3$ [Eq.8] that can screened the light penetration (Ebrahiem *et al.,* 2017; Reza *et al.,*2016; Wahyuni *et al.,* 2019). These cases inhibited the •OH radicals formation, that was less conductive for the oxidation of As(III).

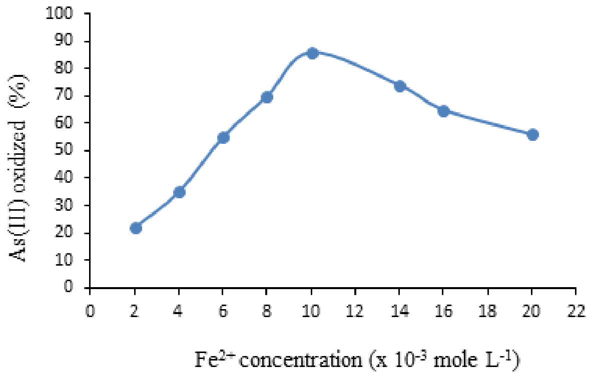

Figure 2. Effect of Fe^{2+} concentration on the oxidation of As(III) (As(III): 10 mg L^{-1}, H2O2: 5 10^{-2} mole L^{-1}, pH: 3, time: 30 min).

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

$$Fe^{3+}+3{}^{-}OH \rightarrow Fe(OH)_3 \qquad (4)$$

It is obviously observed that the optimal Fe^{2+} concentration is 10^{-2} mole L^{-1} for H_2O_2 5. 10^{-2} mole L^{-1} giving 1/5 of the mole ratio. Many studies also reported that the amount of Fe^{2+} were always lower than that of H_2O_2, with the various mole ratio ranging from 1/25 to 1/4 for corresponding substrates, such as organic pollutant in industrial wastewater (Ebrahiem *et al.,* 2017), heavy metals leaching (Azhdarpoor *et al.,* 2015), anionic surfactant (Wahyuni *et al,,* 2016), amoxicillin (Buitrago *et al.,* 2020), bacterial (O' Dowd and Pillai, 2020), and Pb(II) (Wahyuni *et al.,* 2019). In technical applications, the lesser Fe^{2+} concentration is beneficial since it can prevent formation of large amounts of iron sludge (Azhdarpoor *et al.,* 2015; Buitrago *et al.,* 2020; Ebrahiem *et al.,* 2017).

3.2. Effect of H_2O_2 concentration on the oxidation of As(III)

H_2O_2 is one of a primary factor in the determining the effectiveness of photo-Fenton process, and the effect of the H_2O_2 concentration on the oxidation of As(III) is presented in Figure 3. Improvement of the oxidation is observable as the concentration of H_2O_2 increased until H_2O_2 5. 10^{-2} mole L^{-1} that can be attributed to the increase in the amount of hydroxyl radicals. The higher H_2O_2 concentration than the optimum level, shows the opposite effect. Some studies presented similar finding trend (Azhdarpoor *et al.,* 2015; Buitrago *et al.,* 2020; Ebrahiem *et al.,* 2017; O' Dowd and Pillai, 2020; Reza *et al.,* 2016; Wahyuni *et al.,* 2016, 2019). The excess of H_2O_2 contributed to the •OH radical scavenging to form radical of $\bullet O_2H$ (Eq.5), and consecutive scavenging •OH by $\bullet O_2H$ resulting in water and oxygen gas (Eq. 6). These reactions caused the OH radicals depletion, that deceased the As(III) oxidation.

$$\bullet OH+H_2O_2 \rightarrow H_2O+\bullet O2H \qquad (5)$$

$$\bullet O_2H+\bullet OH \rightarrow H_2O+O_2 \qquad (6)$$

Therefore, H_2O_2 should be added at an optimal concentration to achieve the best oxidation of As(III), that were also carried out by many Authors (Azhdarpoor *et al.,* 2015; Buitrago *et al.,* 2020; Ebrahiem *et al.,* 2017; O' Dowd and Pillai, 2020; Reza *et al.,*2016; Wahyuni *et al.,* 2016, 2019).

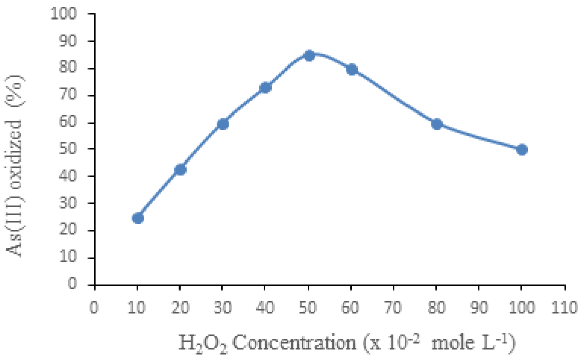

Figure 3. Effect of H_2O_2 concentration on oxidation of As(III) (As(III): 10 mg L^{-1}, Fe^{2+} : 10^{-2} mole L^{-1}, pH: 3, time: 3h).

3.3. *Effect of the reaction time*

One of the determining factor toward the efficiency of the photo-Fenton process is irradiation time. Figure 4 illustrates the effect of the reaction time on the oxidation of As(III). It is attributed that the oxidation remarkably increases with the extension time up to 3 h, but for longer reaction time than 3 h the oxidation slightly declined. Some studies obtained similar trend (Azhdarpoor *et al.,* 2015; Buitrago *et al.,* 2020; O'Dowda and Pillai, 2020; Wahyuni *et al.,* 2016, 2019). Extension of the reaction time has improved the collision between Fe^{2+} ions with H_2O_2, between H_2O_2 and light, and between •OH radicals with As(III) ion. However, longer process allowing Fe^{3+} to react with ^-OH ions present, to form un-soluble $Fe(OH)_3$, that was shown by brown and turbid solution (Azhdarpoor *et al.,* 2015; Buitrago *et al.,* 2020; O'Dowd and Pillai, 2020; Wahyuni *et al.,* 2016, 2019). The high turbidity could retard the light penetration to contact with the H_2O_2, having consequence of the lower oxidation. The photo-treatment time must be as short as possible to avoid a high electricity consumption, which represents about 60% of the total operational cost when using electric light sources (Ebrahiem *et al.,* 2017; Wahyuni *et al.,* 2016; 2019).

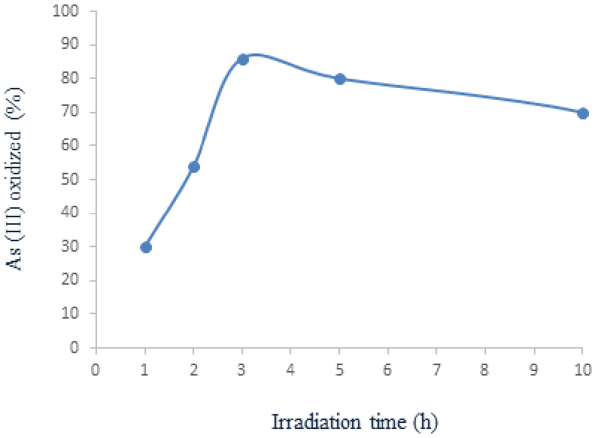

Figure 4. Effect of the irradiation time on the oxidation of As(III) (As(III): 10 mg L^{-1}, Fe^{2+} : 10^{-2} mole L^{-1}, H_2O_2 : 5 10^{-2} mole L^{-1}, pH: 3).

3.4. *Effect of working pH*

One of the key factors affecting the efficiency of photo-Fenton process is pH. It plays an important role in controlling the activity of the oxidizing agent and the substrate, formation of iron species, and hydrogen peroxide stability. pH also affects •OH radicals production and, therefore, influences the efficiency of oxidation. Hence, the role of pH in the photo-assisted Fenton reaction must be determined. Figure 5 indicates that solution pH significantly influences the oxidation of As(III). At lower pH than 3 we have a very low efficiency of As(III) oxidation, then the oxidation appears to raise, but increasing pH higher than 3 leads to a decrease in the As(III) oxidation. For pH values below 3, H_2O_2 in the solution could react with Fe^{2+} ion to form a stable complex compound, which neutralized the iron catalyst and significantly reduced the oxidation efficiency (Azhdarpoor *et al.,* 2015; Buitrago *et al,* 2020; Ebrahiem *et al.,* 2017; Reza *et al.,* 2016; Villegas-Guzman *et al.,* 2017; Wahyuni *et al.,* 2016, 2019). Another reason for the inefficient oxidation at pH lower than 3 was due to the dissociation and auto-decomposition of H_2O_2 (Ebrahiem *et al.,* 2017; Azhdarpoor *et al.,* 2015; Wahyuni *et al.,* 2016, 2019). For pH values above 3 the oxidation steeply decreases since the Fe^{3+} precipitates as hydroxide derivate (Eq 4), diminishing the Fe^{2+} availability and the light penetration (Azhdarpoor *et al.,* 2015; Buitrago *et al.,* 2020; Ebrahiem *et al.,* 2017; Reza *et al.,*2016; Wahyuni *et al.,* 2016, 2019). Obviously, the optimal solution pH in this work was obtained at 3.0, that was same result reported in previous publications (Azhdarpoor *et al.,* 2015; Buitrago *et al.,* 2020; Ebrahiem *et al.,* 2017; O'Dowd and Pillai, 2020; Reza *et al.,*2016; Wahyuni *et al.,* 201, 2019).

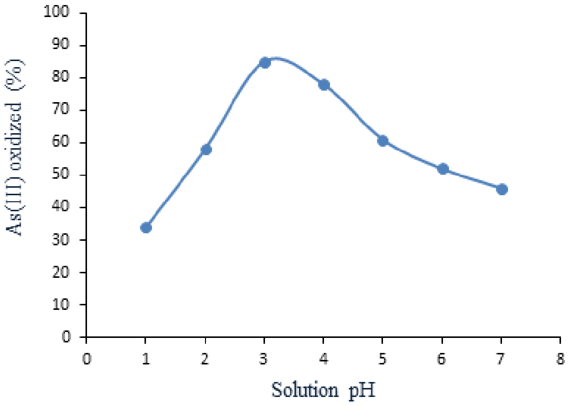

Figure 5. Effect of working pH on the oxidation of As(III).

3.5. *Oxidation of As(III) through various conditions*

To evaluate the efficiency of photo-Fenton in the As(III) oxidation, a series of As(III) solutions were treated with several conditions, that were in the presence of UV light, H_2O_2, H_2O_2 + UV light, H_2O_2 + Fe^{2+} (Fenton), and Fe^{2+} + H_2O_2 + UV light (photo-Fenton). The results displayed in Figure 6, demonstrates that oxidation of As(III) in the aqueous media at pH 3, can proceed with low effectiveness only under UV light irradiation. This oxidation of As(III) was induced by few number of •OH radicals resulted only from water photolysis, as seen in equations (7) and (8), where at pH 3, As(III) is formed as acid of H_3AsO_3 (Saleh *et al.,* 2021). The oxidation effectiveness is observed very low because the water photolysis was less able to release •OH radicals (Ebrahiem *et al.,* 2017; Wahyuni, *et al,* 2019).

$$H_2O+UV\rightarrow\bullet OH+H^++e^- \qquad (7)$$

$$H_3AsO_3+2\bullet OH\rightarrow HAsO_3+2H_2O \qquad (8)$$

$$H_2O_2+H_3AsO_3\rightarrow 2H_2O+HAsO_3 \qquad (9)$$

The oxidation of As(III) at pH 3 induced only by H_2O_2 is observed slightly higher than the former condition. It is well known that H_2O_2 is a good oxidizing agent with standard reduction potential (E) =1.78 V (Ebrahiem *et al.,* 2017; Wahyuni *et al.,* 2019), that could stimulate adequate amount of oxidation. The oxidation of As(III), as H_3AsO_3, by H_2O_2 was presented as a reaction of Eq. 9.

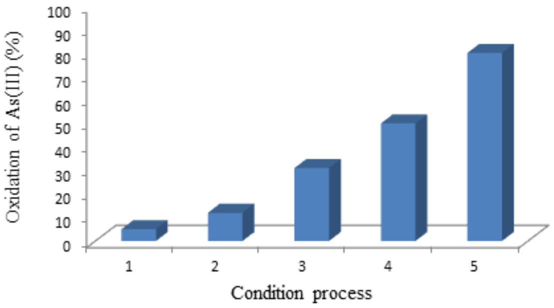

Figure 6. Comparison several conditions on the As(III) oxidations, with 1) UV, 2) H_2O_2, 3) H_2O_2 +UV, 4) Fe^{2+} + H_2O_2, and 5) Fe^{2+} + H_2O_2 + UV light (As(III): 10 mg L^{-1}, Fe^{2+}: 10^{-2} mole L^{-1}, H_2O_2: 5 10^{-2} mole L^{-1}, pH: 3, time: 3 h).

Next data appears that the combination of H_2O_2 and UV light in the aqueous media can result in the higher oxidation of As(III). This was because such condition could produce more number of •OH radicals (Eq.2), that further raised the oxidation results.

From Figure 6, it is also possible to say that the Fe^{2+} catalyzing decomposition of H_2O_2 through Fenton process is more efficient than the UV catalyzing decomposition of H_2O_2, to form •OH radicals. Among the experimental conditions, it is clearly seen that photo-Fenton process resulted in the highest oxidation, that was promoted by much more •OH radicals, resulted from the reaction between Fe^{2+} with H_2O_2 (Eq.1) and the photolysis of H_2O_2 by UV light (Eq.2). This signifies that UV lamp has a sensible increase on As(III) oxidation comparatively to the Fenton process. A study found also the same results (Ebrahiem *et al.,* 2017), except for H_2O_2 that was not able to degrade the dyes (Figure 7).

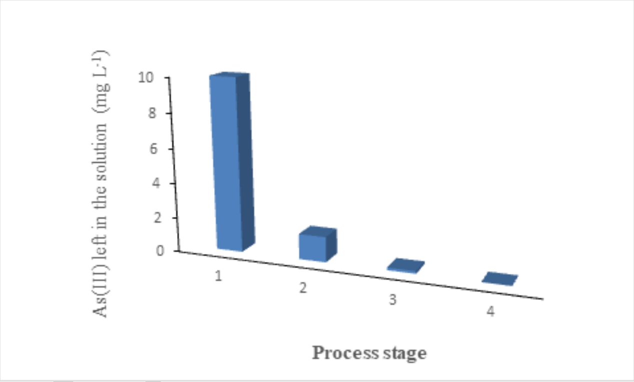

Figure 7. Influence of the process stages on the As(III) concentration left in the solution, where : 1) initial concentration, and 2) 1st, 3) 2nd and 4) 3th stage.

3.6. Influence of the process stages

From the optimal condition of As(III) oxidation, the concentration As(III) that was un-oxidized or left in the solution is found to be 1.46 mg L^{-1}. This concentration is still higher than the acceptable level, that is 0.01 mg L^{-1} (Shumlas, *et al.,* 2016). In order to fulfill the permissible level, the solution from first photo-Fenton step, was processed under optimal condition that was by using Fe^{2+} and H_2O_2 as much as 10^{-2} mole L^{-1} and 5 10^{-2} mole L^{-1} at pH 3 for 3 h. The second stage process resulted in the concentration of As(III) left in the solution as low as 0.15 mg L^{-1} that has not reached the acceptable level. Accordingly, the third stage was further also proceeded under the optimum condition, and the As(III) was almost totally oxidized, that has well met the permissible level.

4. Conclusions

The data obtained in this research show a strong proof that oxidation of As(III) into As(V) can be successfully performed by photo-Fenton process. The effectiveness of the As(III) oxidation was considerably controlled by the concentrations of Fe^{2+} and H_2O_2, the working pH and the irradiation time.

The best condition of As(III) oxidation that was around 85% from 10 mg L^{-1}, could be obtained by treating with 10^{-2} mole L^{-1} of Fe^{2+}, H_2O_2 as much as 5. 10^{-2} mole L^{-1}, working pH at 3 and irradiation time within 3 h. Additionally, to reach the permissible level, it needed 3 stages of the oxidation process.

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