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ORIGINAL ARTICLE

Comparing the efficiency of UV/ZrO_2 and UV/H_2O_2/ZrO_2 photocatalytic processes in furfural removal from aqueous solution

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Abstract

Furfural is a toxic chemical compound that is widely applied as a solvent in a great many of industries, and it can cause many problems to the human beings and environment. Various methods of removing furfural from the wastewaters have been studied. AOPs methods are utilized for the elimination of a vast majority of the pollutants due to their high efficiency as well as for their lack of creating secondary contamination. Therefore, the present study aims at comparing the efficiency of UV/ZrO_2 and UV/H_2O_2/ZrO_2 photocatalytic processes in removing furfural from aqueous solutions. The solution's initial pH, furfural's concentration, zirconium catalyst dosage and time were investigated as the parameters influencing the removal efficiency by the two foresaid processes, and the effect of H_2O_2 addition in various concentrations into UV/H_2O_2/ZrO_2 process was also evaluated. Spectrophotometer device was employed to assay the concentration of the residual furfural. The results indicated that the pH of the environment, the amount of the nanoparticle and H_2O_2 input concentration largely influence the furfural omission. The optimal condition for the removal of furfural in UV/ZrO_2 process in an initial concentration of 20 mg/L, a pH equal to 3, a catalyst dose of 0.25 g/L during a period of 60-min time was 81.6%, and it was 99% for UV/H_2O_2/ZrO_2 process in a pH equal to 7 with the addition of H_2O_2 for a concentration of 0.75 mL/L under the same conditions. Generally, it can be concluded that UV/H_2O_2/ZrO_2 and UV/ZrO_2 photocatalytic processes can effectively be applied to remove furfural from the aqueous solutions, especially in lower concentrations.

Keywords Furfural · Photocatalytic processes · Zirconium dioxide · Hydrogen peroxidation

Introduction

Furfural has numerous use cases and is extensively and widely utilized. Furfural and its derivatives are used in the production processes of solid resins as well as plastic and paper (Fazlzadeh et al. 2018). Also, this chemical compound is most frequently used in petroleum and oil purification industry (Hoydonckx et al. 2000; Presto et al. 2007; Borghei and Hosseini 2008). In such a manner that the furfural concentrations of the wastewaters stemming from rubber and plastic manufacturing, furfural production and oil refineries are reportedly 500 mg/L, 600 mg/L and 1000 mg/L, respectively (Wirtz and Dague 1993; Presto et al. 2007; Sahu et al. 2007). Since furfural is a toxic chemical compound and persistent in the environment (Faramarzpour et al. 2009; Leili et al. 2013), the wastewaters containing furfural should be appropriately treated before being discharged to the environment so as to preserve the human health and conserve the environment.

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There are numerous methods studied for the removal of furfural from the wastewaters including the surface absorption and oxidation methods (Sahu et al. 2007, 2008; Borghei and Hosseini 2008; Singh et al. 2009). Advanced oxidation processes (AOPs) such as UV/H_2O_2, TiO_2/UV, UV/O_3, O_3/H_2O_2, $US/H_2O_2/Fe^{2+}$ and others of the like are inter alia these methods. These processes featuring very high efficiency and lacking secondary pollutions can be used for the degradation of a great many of the pollutants (Rahmani et al. 2017; Seid-Mohammadi et al. 2017).

Photocatalysis is an AOP process, that is, a light absorption-based process by a solid substrate (Samarghandi et al. 2011; Parastar et al. 2013). In this process, the nanoparticles act as catalysts to absorb the UV high-energy photons subsequent to which active chemical materials like hydroxyl radicals are formed (Parga et al. 2003; Fazlzadeh et al. 2016a, 2017a, b; Azizl et al. 2017; Khosravi et al. 2018). Photocatalytic oxidation processes by the use of metal oxides have been taken into consideration in the process of removing organic pollutants and microbial factors during the recent years, and the photocatalytic characteristics of metal oxides such as ZrO, ZnO and TiO_2 have been explored (Kamat et al. 2008; Fazlzadeh et al. 2016b, 2017a). Recently, a compound, called ZrO_2, has been considered as a photocatalyst in inhomogeneous photochemical reactions. This compound has been applied in the production of hydrogen from water as well as for the oxidation of 2-propanol to acetone, and propane and ethane oxidation, photolysis of 4-chlorophenol, 4-nitrophenol and 1, 4-pentandiole due to its high band gap energy and the high negative conduction band potential (Botta et al. 1999).

The study by Karunakaran et al. (2012) on phenol decomposition by the use of reinforced ZrO_2 with various semiconductors in the presence of UV rays indicated that the photocatalytic decomposition linearly increases with the phenol concentration and the light intensity and decreases with the increase in pH. Also, semiconductors' precipitation enhanced the efficiency of ZrO_2 (Karunakaran et al. 2012). In the study that was conducted by Malakootian et al. (2013) on the efficiency of UV/ZrO_2 and $UV/H_2O_2/ZrO_2$ in removing cyanide from aqueous solutions, it was concluded that the efficiency of UV/ZrO_2 process is increased with the increase in the amount of nanoparticle used and the extension of irradiation time and reduction in pH and that the increase in the cyanide concentration depreciates the efficiency of both of the processes. Increasing the H_2O_2 to an optimal amount (0.5 mL/100 mL) enhances the efficiency of $UV/H_2O_2/ZrO_2$ process, but the use of higher H_2O_2 amounts decreases the process output (Malakootian et al. 2013).

The objective of the present study is to investigate the efficiency of photocatalytic process of furfural removal by the use of UV/ZrO_2 and $UV/H_2O_2/ZrO_2$ from the aqueous solutions and then to compare the two processes. Moreover, the study evaluates the direct photolysis states (UV alone), UV/H_2O_2 and ZrO_2/H_2O_2 in furfural removal. The COD of the solution was also measured under optimal conditions so as to assess the COD variations caused by the changes in furfural concentration.

Study method

Materials specification

The study was carried out in a laboratory scale by the use of a photoreactor on a solution containing furfural in various concentrations. The required samples were prepared synthetically. The study utilized a 55-W quartz-coated low-pressure UVC mercury lamp installed inside a steel chamber with a high reflection degree. The reaction spots were on the water specimens as well as on the UVC lamp–steel cover interface, as shown in Fig. 1. The foresaid photoreactor was connected to a 1-L tank for samples loading and sampling operation, and a vacuum pump was applied to create a continuous flowing and mixing of the reactor contents.

The experiments were conducted by the use of zirconia nanoparticles and hydrogen peroxidation in various ranges of pH so as to determine the optimal pH value. The pH ranged from 3 to 11 for both of the processes. pH regulation was conducted by the use of normal 0.1 NaOH and H_2SO_4.

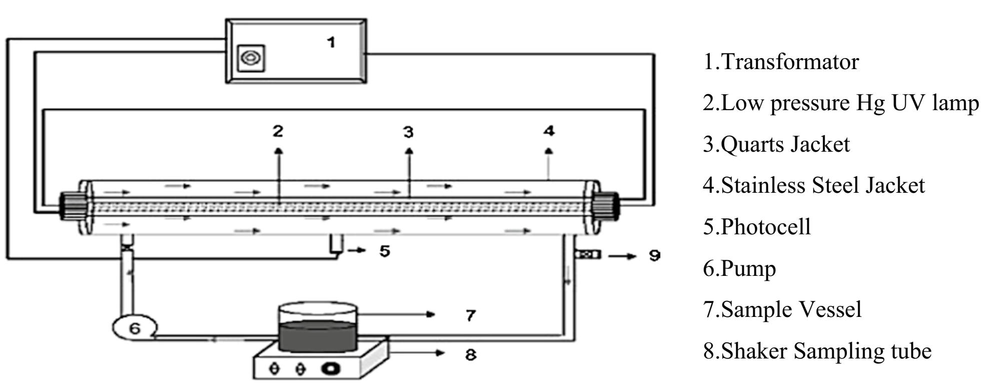

Fig. 1 Schematic view of the photoreactor containing a 55-W quartz-coated low-pressure UVC mercury lamp

To perform the experiment, first of all, the initial furfural solution, with a 10 g/L concentration, was made in deionized distilled water. According to the studies undertaken in this regard, the furfural concentration in the current research paper was selected equal to its real concentrations in wastewaters. Zirconia nanoparticle concentration in UV/ZrO_2 process was in a range from 0.1 to 1 g/L with an optimal pH of the first stage (Malakootian and Hashemi Cholicheh 2012; Malakootian et al. 2013). After acquiring the optimal concentration of each of the oxidant materials (zirconia and hydrogen peroxide), furfural removal was continued by blending H_2O_2 and ZrO_2 in UV/H_2O_2/ZrO_2 process so as to attain a high furfural elimination output. Hydrogen peroxidation concentration in UV/H_2O_2/ZrO_2 process in an optimal pH value in a range from 0.1 to 1.5 mL/L was increased and an optimal concentration was obtained. Next, the effect of furfural concentration was investigated in a 20–1000 mg/L range on its removal (Wirtz and Dague 1993; Sahu et al. 2007; Borghei and Hosseini 2008). COD of the initial and secondary solutions was measured under optimum conditions (only for a 500 mg/L concentration of furfural) so that the COD variations caused by the furfural concentration changes can be assessed. To determine the effect of reaction time on UV/ZrO_2 and UV/H_2O_2/ZrO_2 processes' output, the sampling was carried out within 5–60-min retention times of the experiment in the entire study.

Chemical analysis

To segregate the catalyst particles from the solution, the samples were centrifuged (3 min in 5000 rpm) and then filtered in 0.22-μm filter papers. UV–Vis spectrophotometer device (Perkin-Elmer Lambda 25) was employed to assess the concentration of the residual furfural at 228 nm (Cuevas et al. 2014). Then, the standard curves were delineated for various concentrations so that the equivalent concentration of each absorption process can be determined (Borghei and Hosseini 2008). COD assessment was also carried out based on chromometry in a spectrometer device.

To compare the processes, besides the two above-mentioned processes, UV alone conditions, UV/H_2O_2 and ZrO_2/H_2O_2, as well, were evaluated in furfural degradation process. The data were analyzed by the use of SPSS and Excel.

Results and discussion

The effect of pH on the efficiency of furfural removal in UV/ZrO_2 and UV/H_2O_2/ZrO_2 processes

pH is a crucial factor in AOPs processes, and it has been well identified to have effects on the contaminants' removal. In this stage, pH, in various amounts of 3, 5, 7, 9 and 11, was investigated in the two UV/ZrO_2 and UV/H_2O_2/ZrO_2 processes. As shown in Fig. 2a, pH variations are effective on UV/ZrO_2 efficiency. Furfural removal efficiency for pH equal to 3 and 11 was 36% and 29%, respectively, whereas, corresponding to Fig. 2b, the pH variations' effect on UV/H_2O_2/ZrO_2 process has been somewhat different. The highest furfural removal efficiency for a pH 7 and the lowest furfural removal efficiency for a pH 11 have been 47% and 32%, respectively.

Generally, it can be stated that the furfural removal efficiency decreases with the creation of more basic conditions. There was a significant correlation found between pH and UV/ZrO_2 process in 60 min after the reaction initiation ($P<0.01$). One reason behind such an increase in efficiency in lower pHs is that the ZrO_2 nanocatalyst surface becomes positively charged and absorbs furfural as a result of which more furfural is absorbed, $OH^{\bullet}$ is produced, the

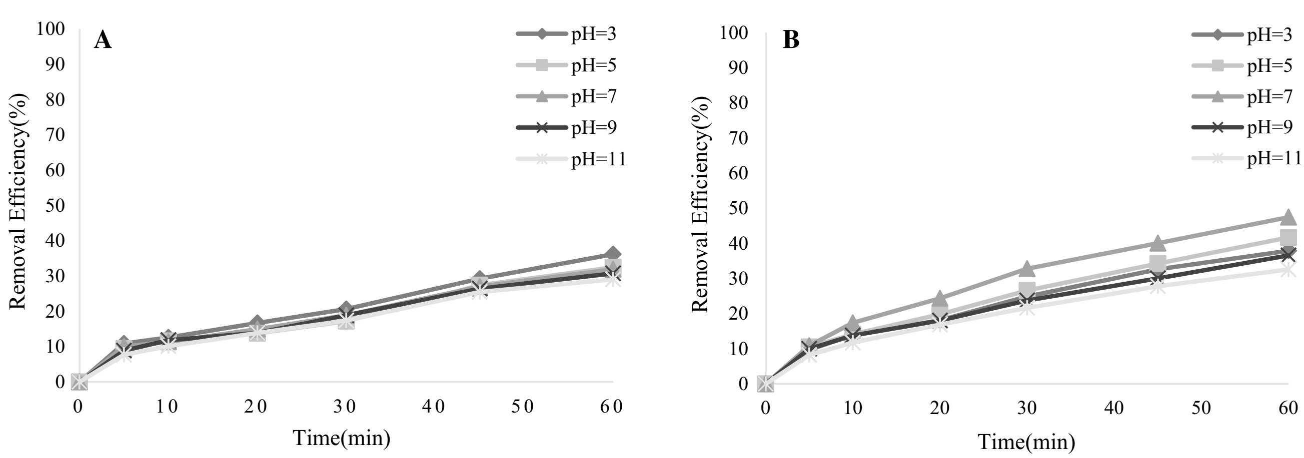

Fig. 2 Effect of pH on furfural removal efficiency, **a** UV/ZrO_2 process ($ZrO_2=0.5$ g, furfural concentration = 500 mg/L); **b** UV/H_2O_2/ZrO_2 process ($ZrO_2=0.5$ g/L, furfural concentration = 500 mg/L and $H_2O_2=0.5$ mL/L)

decomposition becomes more intensified in acidic environment, and then the removal efficiency increases eventually. Also, in UV/H_2O_2/ZrO_2 process, H_2O_2 acts as the dominant oxidizing agent in the environment and the hydroxyl radical produced resultantly will display a higher oxidation potential. But, in basic environments, the nanocatalyst becomes negatively charged on its surface as a result of which absorption is reduced and then less $OH^{\bullet}$ is produced. The results of the present study were similar to what was found by Malakootian et al. (2013). They studied the efficiency of UV/ZrO_2 and UV/H_2O_2/ZrO_2 processes in elimination of cyanide, and the highest removal efficiency found in pH values was equal to 4 and 8. Also, the results of the present study are consistent with what was found by Samarghandi et al. who studied photocatalytic decomposition of cyanide by the use of TiO_2 as well as pentachlorophenol elimination by the use of UV/H_2O_2/ZrO_2 process (Malakootian et al. 2013; Samarghandi et al. 2015). The optimal pH values in UV/ZrO_2 and UV/H_2O_2/ZrO_2 processes were 3 and 7, respectively.

The effect of zirconia nanoparticles on furfural removal efficiency in UV/ZrO_2 and UV/H_2O_2/ZrO_2 processes

Figure 3a and b, respectively, demonstrates the effect of zirconia nanoparticle amounts of 0.1 g/L, 0.25 g/L, 0.5 g/L, 0.75 g/L and 1 g/L on furfural removal efficiency in UV/ZrO_2 and UV/H_2O_2/ZrO_2 processes. In UV/ZrO_2 process, the highest and the lowest removal efficiency were, respectively, related to 0.25 g/L and 0.1 g/L during a 60-min retention time. The process efficiency in 1 g/L dosage was only higher than the efficiency of 0.1 g/L dosage. In UV/H_2O_2/ZrO_2 process, as shown in Fig. 3b, the furfural removal efficiency increases from the dosage 0.1 to 0.25 g/L, but the removal efficiency underwent a decrease with the increase in the nanoparticle dosage any further than this range. There was not found any significant relationship between the photocatalyst dosage and removal efficiency in any of the two processes ($P > 0.05$).

The reason why the number of absorbed photons is increased with the initial increase in the nanoparticle's concentration from 0.1 to 0.25 g/L has been the increase in the number of available activated sites as a result of which more furfural molecules are absorbed. Furthermore, the reason behind the lower furfural removal efficiency in a concentration of 1 g/L as compared with the concentrations 0.25 g/L and 0.5 g/L is that the increase in catalyst can even reduce the light infiltration to the solution and increase the light scattering of the nanoparticle surface that results in the reduction in the light-activated volume of the nanoparticle (Yang et al. 2008; Kashif and Ouyang 2009; Mahvi et al. 2009; Abdoallahzadeh et al. 2016), following which a small amount of ZrO_2 is activated. The study by Chan et al. as well as the study by Parastar et al. on the nitrate removal in a ZnO/UV process also confirms this same finding of the present study (Chun et al. 2000; Mahvi et al. 2009; Biglari et al. 2015). But, in the study conducted by Samarghandi et al., it was shown that the pentachlorophenol removal efficiency reduces in UV/ZrO_2/H_2O_2 process with the increase in catalyst amount from 0.1 to 1 g/L (Samarghandi et al. 2015). Therefore, the present study introduces 0.25 g/L as the optimal amount of the catalyst for performing the experiment.

The effect of hydrogen peroxidation amount addition on the furfural removal efficiency of UV/H_2O_2/ZrO_2 process

In this stage, the effect of hydrogen peroxidation concentrations in UV/ZrO_2/H_2O_2 process has been investigated in a range from 0.1 to 1.5 mL/L. According to Fig. 4, it can be

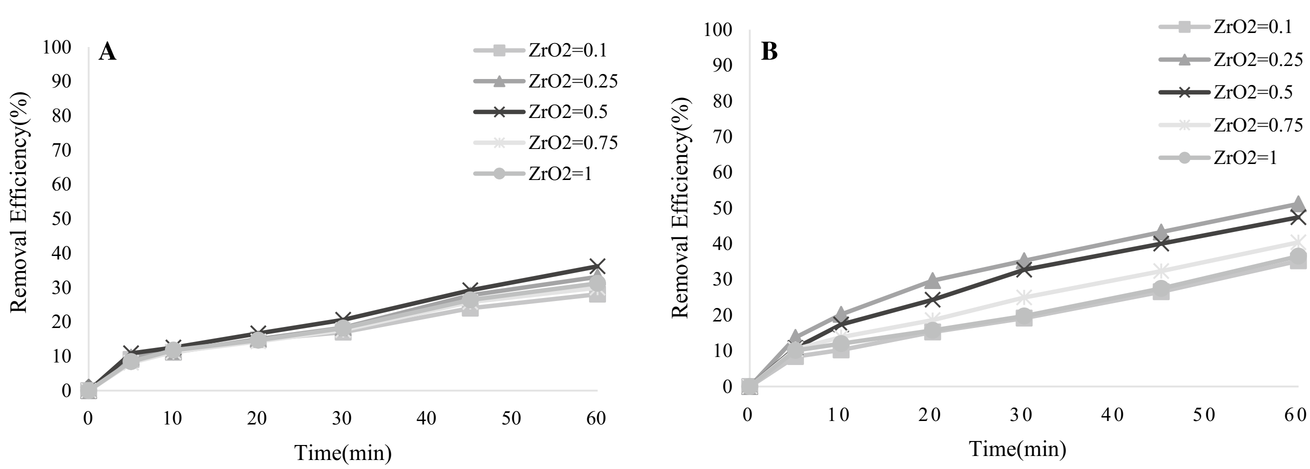

Fig. 3 Effect of ZrO_2 amount on furfural removal efficiency; **a** UV/ZrO_2 process (pH 3, furfural concentration = 500 mg/L); **b** UV/H2O2/ZrO_2 (pH 7; furfural concentration = 500 mg/L, H_2O_2 = 0.5 mL/L)

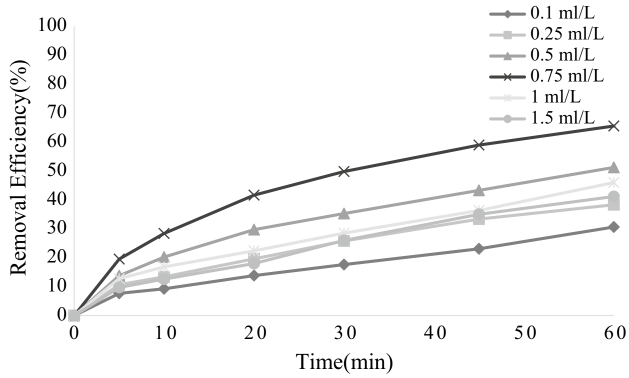

Fig. 4 Effect of H_2O_2 amount on $UV/H_2O_2/ZrO_2$ process efficiency (pH 7, $ZrO_2=0.25$ g/L, furfural concentration = 500 mg/L)

observed that the highest removal efficiency has occurred in 0.75 mL/L concentration of the hydrogen peroxidation (that differs from 19 to 65% depending on the reaction time), while the lowest efficiency was observed for 0.1 mL/L concentration (between 9 and 30%). In other words, the removal efficiency is first increased and then decreased with the increase in the hydrogen peroxidation concentration ($P>0.05$). That is because hydroperoxyl radicals, featuring weaker oxidation power, are produced in higher concentrations of hydrogen peroxidation due to the reaction with the hydroxyl radical (Eq. 1), and the additional amounts of the hydroxyl radical are again converted to hydrogen peroxide (Eq. 2). The results of the present study correspond with the findings of the studies conducted by Samarghandi et al. (2015), Malakootian et al. (2013) and Rahmani et al. (2015).

$$H_2O_2 + OH^{\bullet} \rightarrow HO_2^{\bullet} + H_2O \quad (1)$$

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

The effect of furfural concentration on furfural removal efficiency in UV/ZrO_2 and $UV/ZrO_2/H_2O_2$ processes

Figure 5a and b, respectively, shows the efficiency of UV/ZrO_2 and $UV/ZrO_2/H_2O_2$ processes in a furfural concentration ranging from 20 to 1000 mg/L. The highest frequency of both of these processes within a 60-min period after the reaction initiation for a concentration of 20 mg/L was 81% and 99%, respectively, and the lowest efficiency of the processes for a concentration of 1000 mg/L, respectively, was 23% and 43%. The relationship was significant for both of the processes ($P<0.05$ and $P<0.01$, respectively). The increase in furfural concentration causes a reduction in removal efficiency because furfural, per se, absorbs a fraction of the irradiated rays and acts as a light filter (Parastar et al. 2012). Also, the concentration of the buffers resulting from the contaminant decomposition and photon absorption by the contaminant and the overall buffer concentration is increased in higher contaminant concentrations that will lead to a reduction in the energy required for the production of hydroxyl radicals (Rahul and Damodar 2008; Parastar et al. 2013). Furthermore, it can be stated that the amount of the hydroxyl radicals is generated per every insufficient furfural amount, and hence the removal efficiency is decreased (Biglari et al. 2015). These findings conform to the results obtained in the above-mentioned studies (Rahul and Damodar 2008; Parastar et al. 2012, 2013; Biglari et al. 2015). It is worth mentioning that the processes' efficiency increases with the increase in the time considered for the reaction, and it was found perfectly significant ($P<0.01$).

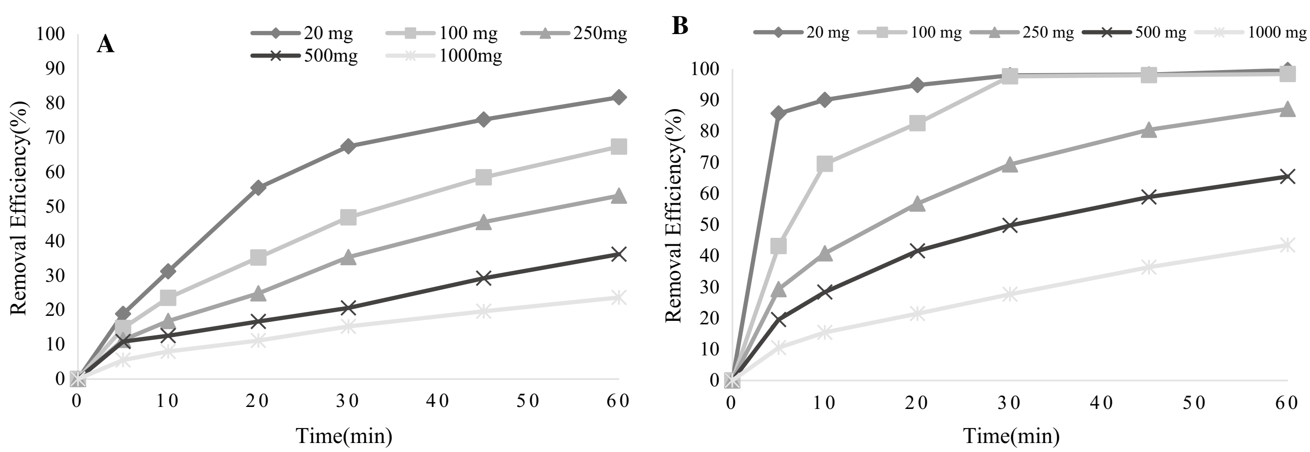

Fig. 5 Effect of furfural concentration on its removal efficiency, **a** UV/ZrO_2 process (pH 3; $ZrO_2=0.25$ g/L); **b** $UV/H_2O_2/ZrO_2$ process (pH 7, $ZrO_2=0.25$ g/L and $H_2O_2=0.75$ mL/L)

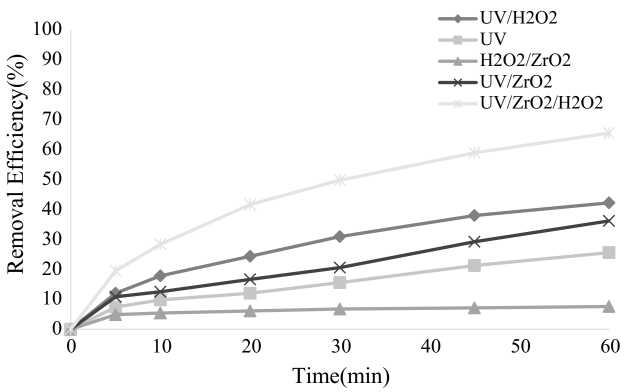

Fig. 6 Comparing the efficiency of the studied processes in furfural removal, **a** $UV/H_2O_2/ZrO_2$ process ($ZrO_2=0.25$ g/L, furfural concentration = 500 mg/L and $H_2O_2=0.75$ mL/L), **b** UV/ZrO_2 (pH 3) and the other processes (pH 7)

Comparing the efficiency of UV irradiation and UV/H_2O_2 and ZrO_2/H_2O_2 processes in furfural removal

In this stage, the efficiency of hybrid UV/H_2O_2, ZrO_2/H_2O_2, $UV/H_2O_2/ZrO_2$, UV/ZrO_2 and UV alone processes was studied and compared. As it is observed in Fig. 6, the removal efficiency increases with the more time elapsed since the initiation of the reaction in all of the processes. Also, the efficiency of the $UV/H_2O_2/ZrO_2$, UV/H_2O_2, UV/ZrO_2, direct photolysis and finally ZrO_2/H_2O_2 processes was 65%, 42%, 36%, 25% and 7% at the end of the process. These results demonstrated that hydrogen peroxide is more effective in respect to ZrO_2 in a comparison of the two UV/H_2O_2 and UV/ZrO_2 processes. These results are in accordance with the results obtained in the studies carried out by Jefferson et al. In their study, they studied the UV/H_2O_2 and UV/ZrO_2 processes in the decomposition of metaldehyde and organic carbon and concluded that UV/H_2O_2 process outperforms the other one (Jefferson et al. 2016). The results of the study by Baeza et al. were also similar to these same findings (Baeza et al. 2003). But, some of the other studies have proved the opposite (Nguyen and Juang 2015; Nguyen et al. 2016). In this study, $UV/H_2O_2/ZrO_2$ process was found the most perfect one. Similar results were also attained in some of the other researches (Malakootian et al. 2013; Nguyen and Juang 2015; Samarghandi et al. 2015). Although the combination of these three processes done in the current research paper brought about an increase in efficiency, Nguyen et al. indicated that the combination of these three processes is never a criterion insuring the achievement of higher removal efficiency because it has been shown by them that the elevation of the UV lamp wattage (100 W) in TiO_2/UV process causes an increase in its efficiency in contrast to $UV/H_2O_2/ZrO_2$ (20-W UV lamp) (Nguyen and Juang 2016). Additionally, it was found out that 55-W UV lamp has a little effect on furfural removal and the ZrO_2/H_2O_2 process's efficiency is trivial. The reason why the sole use of UV irradiation has had such a significant effect on furfural removal pertains to the UV rays' ability in furfural absorption. Biglary et al., as well, in their study on phenol, came to the conclusion that 125-W UV beams can degrade phenol up to 80% (Biglari et al. 2015). In the study by Parastar et al., it was concluded that direct photolysis removes nitrate from water up to 32% (Parastar et al. 2013). According to the low efficiency of UV irradiation, the simultaneous use of UV light and a photocatalyst can be effective.

Under the conditions of H_2O_2 and ZrO_2 combination, furfural removal efficiency attained was 4–7%. At this stage, due to the absence of UV irradiation and resultant lack of valence band electron activation and the lack of active holes formation on ZrO_2 photocatalyst surface as well as due to the lack of H_2O_2 photolysis and eventually for such a reason as the lack of hydroxyl radical's generation, as the main agent contributing to the contaminant decomposition, the removal efficiency reached to its least possible value.

Comparing the efficiency of UV/ZrO_2 and $UV/H_2O_2/ZrO_2$ in removing dissolved COD

In this stage, the effects of the two primary processes studied herein were investigated in terms of dissolved COD generated by furfural under optimal conditions. According to Fig. 7, it can be clearly seen that $UV/H_2O_2/ZrO_2$ outperforms UV/ZrO_2 in its efficiency of dissolved COD removal and the COD removal efficiency of $UV/H_2O_2/ZrO_2$ and UV/ZrO_2 processes for a furfural concentration of 500 mg/L was 54% and 27% within a 60-min reaction time.

Fig. 7 Comparing the efficiency of $UV/H_2O_2/ZrO_2$ and UV/ZrO_2 processes in removing dissolved COD

Conclusion

The results of the present study indicated that the highest efficiencies were, respectively, obtained for $UV/H_2O_2/ZrO_2$, UV/H_2O_2, ZrO_2/H_2O_2, UV irradiation and UV/ZrO_2. UV alone, inter alia the performed stages, was found to have a small effect in furfural removal, and ZrO_2/H_2O_2 process efficiency was very trivial. The highest removal output, about 99%, was obtained by $UV/H_2O_2/ZrO_2$ process for a furfural concentration of 20 mg/L. The removal efficiency was somewhat increased with the increase in the dosage of the photocatalyst used as well as the increase in the H_2O_2 added which subsequently followed by a reduction; thus, the optimal conditions of the experiments were considered as stated in the following words: pH 7, H_2O_2 0.75 mg/L and catalyst dosage 0.25 g/L. The relationship between pH and furfural concentration in UV/ZrO_2 process and the relationship between the reaction time and removal efficiency in UV/ZrO_2 and $UV/H_2O_2/ZrO_2$ process were found statistically significant. It can be finally stated that the hydrogen peroxide in combination with zirconium nanoparticle can be utilized as an effective method in furfural removal. Of course, it has to be pointed out that there is a need for pretreatment in higher furfural concentrations of industrial wastewaters before employing the above-described method so as to decrease furfural concentration in which case the aforementioned processes can promise a high efficiency in furfural removal.

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