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RESEARCH

Photochemical oxidation of methyldiethanolamine (MDEA) in aqueous solution by UV/$K_2S_2O_8$ process

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Abstract Methyldiethanolamine (MDEA) as an organic material is a hazardous contaminant in the aquatic environment because of its adverse effects on aquatic life, environment, and humans. In this study, a batch reactor of ultraviolet (UV) light and peroxydisulfate was performed to investigate the degradation of MDEA in aqueous media. The effect of different experimental parameters such as UV irradiation, peroxydisulfate concentration, MDEA concentration, temperature, and solution pH on removal of MDEA was evaluated precisely. No significant degradation was observed with a separate UV light. Adding peroxydisulfate to the solution increased the removal performance more than 75 %.

Keywords Peroxydisulfate · MDEA · Advanced oxidation processes · Wastewater treatment

Introduction

Raw natural gas includes some acidic gases such as H_2S and CO_2. These acidic gases are very corrosive and toxic to the environment, and therefore required to be removed. Different alkanolamines such as monoethanolamine (MEA), diethanolamine (DEA), methyldiethanolamine (MDEA) and diisopropanolamine (DIPA) are used for the removal of acidic gases in the sweetening gas units [1]. In addition *N*-methyldiethanolamine (MDEA) as metal-MDEA complexes have as propertysignificant ultraviolet (UV) absorption. New photosensitive precursors was prepared as thin films by *N*-methyldiethanolamine complex [2, 3]. Usually during cleaning, protecting and scheduled control of absorption and desorption column, high concentration of alkanolamine is generated into the wastewater [4]. Nevertheless, due to its toxicity the conventional biological treatment cannot be used for this wastewater [5].

During recent two decades, advanced oxidation processes (AOPs) have been considered as popular techniques to treat the high concentration of organic contaminant in the wastewater [6]. AOPs are of the most alternative techniques for destruction of many other organic matters in wastewater and effluents. These processes generally involve UV/H_2O_2, UV/O_3, UV/$S_2O_8^{2-}$ or UV/Fenton's reagent for degradation of contaminants [7–9].

A large number of experimental works have been performed on the application of AOPs to treat wastewater. The Fenton' reagent in the AOPs was used to degrade MEA [10], DEA [11], *N*,*N*-diethyl-*p*-phenylenediamine [12] and DIPA [13]. Also, the use of UV/H_2O_2 in the AOPs for degradation of MEA and MDEA [4, 14] and ozonation for degradation of DEA [15] have been studied.

Because of its high reactivity of UV/$S_2O_8^{2-}$ process, high solubility, relatively low cost of peroxydisulfate and benign end products, recently the application of UV/$S_2O_8^{2-}$ in wastewater treatment was investigated in numerous studies [16]. Peroxydisulfate ($S_2O_8^{2-}$) is a strong oxidant ($E_0 = 2.05$ V) which has been used widely in the petroleum industry for the treatment of hydraulic fluids or as a reaction initiator [17].

It has also been reported to be effective for degrading organic matters in hazardous wastewaters in acidic or basic

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media through direct chemical oxidation, where peroxydisulfate is used as a sacrificial reagent [17–19]. However, since the reactions of peroxydisulfate are generally slow at normal temperature. The thermal or photochemical activated decomposition of $S_2O_8^{2-}$ ion to $SO_4^{\circ-}$ radical has been proposed as a method of accelerating the process [19, 20], as summarized in the following reactions (Eqs. 1–12):

$$S_2O_8^{2-} \xrightarrow{h\nu/\text{heat}} 2SO_4^{\circ-} \quad (1)$$

$$SO_4^{\circ-} + RH_2 \rightarrow SO_4^{2-} + H^+ + RH^\circ \quad (2)$$

$$RH^\circ + S_2O_8^{2-} \rightarrow R + SO_4^{2-} + H^+ + SO_4^{\circ-} \quad (3)$$

$$SO_4^{\circ-} + RH \rightarrow R^\circ + SO_4^{2-} + H^+ \quad (4)$$

$$2R \rightarrow RR(\text{dim}\,er) \quad (5)$$

$$SO_4^{\circ-} + H_2O \rightarrow HSO_4^- + OH^\circ(500 \pm 60\,S^{-1}) \quad (6)$$

$$HSO_4^- \rightarrow H^+ + SO_4^{2-} \quad (7)$$

$$OH^\circ + S_2O_8^{2-} \rightarrow HSO_4^- + SO_4^{\circ-} + \frac{1}{2}O_2 \quad (8)$$

$$SO_4^{\circ-} + OH^\circ \rightarrow HSO_4^- + \frac{1}{2}O_2 \quad (9)$$

$$2OH^\circ \rightarrow H_2O_2 \text{ (expect in alkaline solution)} \quad (10)$$

$$H_2O_2 \rightarrow H_2O + \frac{1}{2}O_2 \text{ (mostly in acidic solution)} \quad (11)$$

$$S_2O_8^{2-} + H_2O_2 \rightarrow 2H^+ + 2SO_4^{2-} + O_2 \quad (12)$$

As can be seen in the above reactions, the oxidation process is begun by production of the sulfate and hydroxyl radicals (Eqs. 1 and 2). These radicals are powerful oxidizing agents which may attack the organic matters (*R*) in the contaminated water. It causes, ultimately, complete decomposition of toxic and bioresistant compounds to harmless species (like CO_2, H_2O, etc.). Sulfate ion will be generated as the end product, which is practically inert and is not considered to be a pollutant. It is worth to mention that the United States Environmental Protection Agency (USEPA) has listed SO_4^{2-} under the secondary drinking water standards. A maximum concentration of sulfate ion is 250 mg l^{-1} (1.43 mM), based on sanitary reasons such as taste and odor [16, 21].

The peroxydisulfate is normally available as a salt associated with ammonium, sodium, or potassium. The comparative performance of $K_2S_2O_8$ (KPS) and $(NH_4)_2S_2O_8$ (APS) as an oxidant under the irradiation of UV light for removal of butylated hydroxyanisole [19], the dye Reactive Yellow 84 [22] and tylosin [23] was investigated. The results have indicated that KPS provides a more rapid photooxidative removal than APS at neutral pH. The difference in the removal efficiency is apparently due to the presence of the ammonium ion. The aqueous ammonium can undergo photooxidation leading to nitrate and/or nitrite by the available oxidants in the solution, such as, $S_2O_8^{2-}$, and its related intermediates H_2O_2 or O_2 [19, 20]. Furthermore, the reaction of NH^{4+}/NH_3 with UV/$S_2O_8^{2-}$ process is proved to be able to convert it to nitrate under the 254 nm photolysis [24], thereby making the ammonium as a competitor of the organic pollutants. In view of this, and the general unsuitability of adding ammonia to waters, APS is not recommended to be used in the UV/peroxydisulfate oxidation process. Therefore, the UV/KPS combination was chosen for further investigation throughout this study.

It has been proven that UV/$S_2O_8^{2-}$ and UV/H_2O_2 (the most common process) have similar reaction rate constants [25]. Moreover, peroxydisulfate advantages UV/H_2O_2 and other similar approaches by the following reasons: (1) Peroxydisulfate ions seem to be more useful when the process is not well controlled, for example when overdosing occurs because of the potential quenching effect of using H_2O_2 [25]. (2) Peroxydisulfate would be more applicable for industrial uses in comparison to liquid oxidants such as H_2O_2, because it is a solid oxidant. (3) Peroxydisulfate salts are much cheaper than other oxidants like hydrogen peroxide and ozone [26–28].

In this study, using UV/$S_2O_8^{2-}$ process the destruction of MDEA as an amine pollutant from contaminated water was investigated. Moreover, effect of different experimental parameters such as UV irradiation, peroxydisulfate concentration, MDEA concentration, Temperature, and pH was evaluated.

Experimental

Reagents

Potassium peroxydisulfate ($K_2S_2O_8$), sulfuric acid and sodium hydroxide were of laboratory reagent grade (Merck Co., Germany) and used without further purification. The synthetic wastewater for which treatment process was performed contains methyldiethanolamine (MDEA).

General procedure

Methyldiethanolamine (MDEA) degradation experiments were conducted in a photoreactor. For UV/peroxydisulfate process, irradiation was carried out with a 125 and 250 W (UV-C) mercury lamp (Philips, the Netherland), which was put above a batch photoreactor. The distance between the solution and UV source was adjusted according to the experimental conditions. The volume of sample was 500 ml, and total time of experiment was 60 min. The

reactor had a water-flow jacket for regulating the temperature by means of an external circulating flow of a thermostat. Since the photocatalysis is sustained by a ready supply of dissolved oxygen, air was supplied to the reaction system at a constant flow rate using a micro-air compressor.

Dilute solutions of sodium hydroxide and sulfuric acid were used for pH adjustment and the initial pH values were measured by Metrohm 827 pH/LF portable pH/conductivity-meter, Schott Instruments GmbH, Mainz, Germany. A prepared solution was transferred to the reactor and after adjusting the temperature, the UV lamp was switched on to initiate the process. During the experiments a mild aeration was kept for mixing the content and saturation with O_2. Samples (6 ml) were taken at regular time intervals. A maximum total sampling volume of 24 ml was withdrawn during each experimental run which is not significant, compared with the solution volume. Chemical oxygen demand (COD) was measured by a closed reflux and titrimetric method. Using this method, the degradation efficiency (X) was obtained at any time, according to [28]:

$$X = \frac{C_0 - C}{C_0}$$

where C_0 and C are initial and appropriate concentrations of MDEA at any time.

Peroxydisulfate concentration varied from 5 to 25 mM, while MDEA concentration varied from 500 to 1500 ppm and temperature varied from 30 to 50 °C. The effect of pH was evaluated in the range of 2–10. Figure 1 shows a schematic view of the reactor and its belongings.

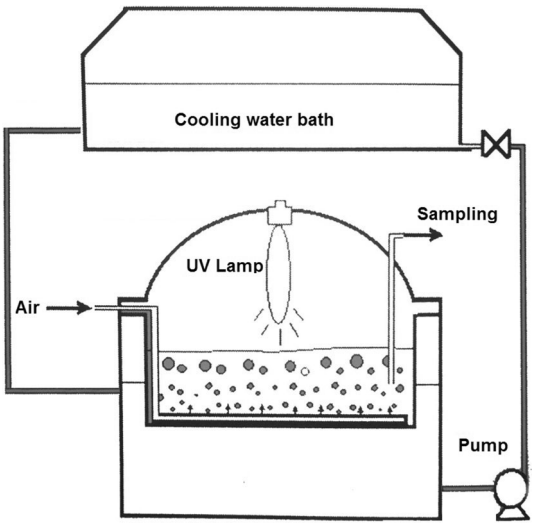

Fig. 1 The schematic view of the photo reactor set-up

Results and discussion

Effect of peroxydisulfate and UV irradiation on degradation of MDEA

Degradation of MDEA was investigated with UV irradiation only, $S_2O_8^{2-}$without UV radiation, and UV radiation plus $S_2O_8^{2-}$ with concentrations of 25 mM peroxydisulfate, 500 ppm MDEA and pH = 8.5. In the first case without any peroxydisulfate, we found no degradation, while using peroxydisulfate without UV irradiation, approximately, the degradation of 6 % was attained. However, UV plus peroxydisulfate made the two mentioned cases more efficient notably by 75 % removal (Fig. 2). In fact, UV irradiation improved the reaction of peroxydisulfate through intensifying the formation of hydroxyl and sulfate radicals in comparison with slow activity at normal temperature. Therefore, as summarized in Eqs. 1–5, thermo-photochemical treatments activated decomposition of $S_2O_8^{2-}$ ion to SO_4^{-} radical which has been proven by acceleration of the process [29]. A rapid attack can be produced on any oxidizable agent including organic pollutants (e.g., MDEA) once $SO_4^{\circ-}$ is formed [30]. Also, available oxidants in the solution and their corresponding intermediates are showed in Eqs. 6–12. Similar observations have been reported by others [31]. Chan et al showed that the removal of iopromide with photo-activated potassium peroxydisulfate was 90 % and it was achieved in 30 min under a light intensity of 6 lamps.

Both $SO_4^{\circ-}$ and OH° are possibly cause of the degradation of organic contaminants. Meanwhile either radical may predominate over the other depending on pH conditions, and react with organic compounds commonly by three mechanisms: hydrogen abstraction, hydrogen addition, and electron transfer.

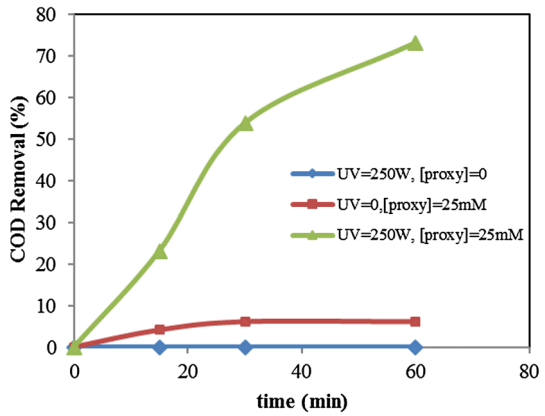

Fig. 2 Effect of peroxydisulfate and UV irradiation on degradation of MDEA (MDEA concentration, 500 ppm; temperature, 30 °C)

Sulfate radicals show a higher standard reduction potential than hydroxyl radicals at neutral pH, and both radicals show similar reduction potentials under acidic conditions [27]. In general, $SO_4^{\circ-}$ is more likely to participate in electron transfer reactions, whereas OH° is more likely to participate in hydrogen abstraction or addition reactions [32].

Effect of initial MDEA concentration

The initial MDEA concentration has a remarkable effect on degradation of MDEA in the UV/$S_2O_8^{2-}$ process. Investigations were made by varying the concentration of MDEA from 500 to 1500 ppm at fixed initial $S_2O_8^{2-}$ concentration of 15 mM, pH = 8.5, irradiation of 125 W and temperature of 30 °C. Degradation of 50 % is observed in 500 ppm, while only 19 % of degradation is achieved in 1500 ppm concentration of MDEA. The effect of initial MDEA concentration on photooxidation efficiency has been depicted in Fig. 3. As shown in Fig. 3, the higher concentration of MDEA was, the lower the degradation rate would be. This is because the rising in MDEA concentration induces an inner filter effect and hence the solution becomes more and more impermeable to UV radiation [33]. Also, the increase in MDEA concentration decreases the ratio of hydroxyl radical to MDEA and percentage of degradation reduces in result.

Effect of initial peroxydisulfate concentration

The initial concentration of peroxydisulfate was found to be an important parameter for the photooxidative degradation of MDEA in the UV/$S_2O_8^{2-}$ process. The effect of initial $S_2O_8^{2-}$ concentration on photooxidation efficiency has been expressed in Fig. 4.

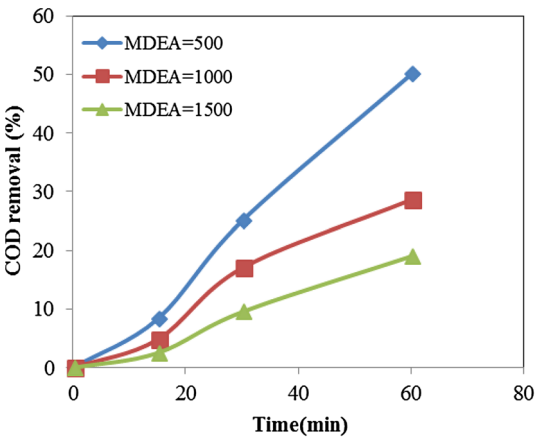

Fig. 3 Effect of the initial MDEA concentration on the oxidative degradation of MDEA (peroxydisulfate concentration, 15 mM; temperature, 30 °C; UV, 125 W)

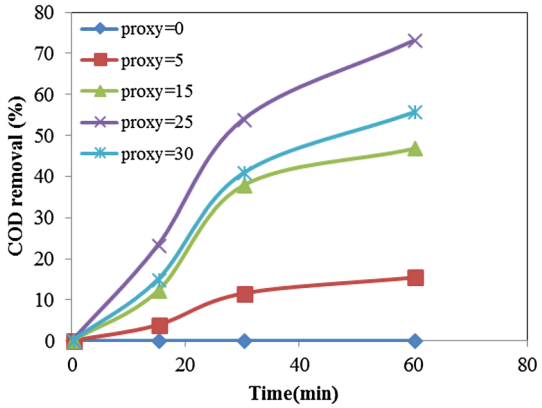

Fig. 4 Effect of the initial peroxydisulfate concentration on the degradation of MDEA (MDEA concentration, 500 ppm; temperature, 30 °C; UV, 250 W)

Investigations were made using varying the concentration of $S_2O_8^{2-}$ from 5 to 25 mM at fixed initial MDEA concentration of 500 ppm, pH = 8.5, irradiation of 250 W and temperature of 30 °C. Studies have revealed that increase in amount of $S_2O_8^{2-}$ from 5 to 25 mM would enhance degradation of the MDEA [34]. These observations can be explained by the fact that the increase in concentration of peroxydisulfate results in higher generation of hydroxyl and sulfate radicals and improves the photooxidative degradation of the MDEA consequently. It is likely because of excessive generation of hydroxyl radicals (Eqs. 1 and 6) that would be recombined to less reactive form of H_2O_2 (Eq. 10), which is a known quencher of OH° radical (Eq. 11). Therefore, the destruction of MDEA was slightly slowed down at higher $S_2O_8^{2-}$ dosages. However, such a recombination effect of the radical was likely not very effective due to the low steady-state concentrations of the radicals; higher decay rates of MDEA at higher $S_2O_8^{2-}$ dosages are still expected [20, 34]. For example, Lin et al. investigated the degradation of ciprofloxacin by UV/peroxydisulfate process. They described the removal efficiency increased with an increase for $S_2O_8^{2-}$ [35]. They described the degradation efficiency of ciprofloxacin increased with time, reaching 95 % after 30 min.

The first-order reaction kinetics was used to study the degradation kinetics of MDEA by UV/$S_2O_8^{2-}$ process. The individual expression was presented as below:

First-order reaction kinetics

$$\frac{dC}{dt} = -k_1 C \tag{13}$$

where C_t the concentration of MDEA at reaction time t (mM), C_o the initial concentration of the MDEA (mM), k_1 the rate constant of the first-order kinetic equation (min^{-1}).

Regression analysis based on the apparent first-order reaction kinetics for the degradation of MDEA in UV/$S_2O_8^{2-}$ was conducted and the results are shown in Fig. 5. The apparent first-order rate constant ranged from 0.003 to 0.0224 min^{-1} (Table 1). k_1 was increased with increasing initial potassium peroxydisulfate concentration. Similar results were also obtained by former studies [36–38]. Khataee et al. investigated the decolorization of basic blue by UV/peroxydisulfate treatment. They described the decolorization rate constant increased with an increase in the amount of $S_2O_8^{2-}$ [39].

Effect of UV irradiation

Investigations were made by varying the irradiation of UV from 0 to 250 W at fixed initial MDEA concentration of 500 ppm, initial concentration of $S_2O_8^{2-}$, pH = 8.5 and temperature of 30 °C. The effect of UV light intensity on the degradation of MDEA is shown in Fig. 6. The figure clearly shows that the removal increases through increasing UV irradiation intensity. This increase is due to the enhanced production of sulfate and hydroxyl radicals. At low UV light intensity the rate of photolysis of $S_2O_8^{2-}$ is limited hence degradation increases [20, 40].

Effect of the initial pH

The effect of initial pH was investigated in the range of 2–10 with constant concentration of MDEA of 500 ppm, initial peroxydisulfate concentration of 15 mM, irradiation of 250 W and temperature of 30 °C. The effect of initial pH on photooxidation efficiency has been depicted in Fig. 7. The pH = 5–7 was found to be the most effective level of pH in degradation of MDEA. However, effective photodegradation of MDEA was observed at all pH levels, which reveals the efficiency of this method in treatment of wastewater in different regions. The destruction performance increased from low to initial neutral pH levels, but it started to reduce when basic pH was performed. The decreasing photo decay at pH ≥ 7 can be explained by the following reasons: (a) the instability of H_2O_2 at high pH level, (b) relatively higher amounts of OH° and $SO_4^{\circ-}$ generated catalytically in alkaline conditions, which

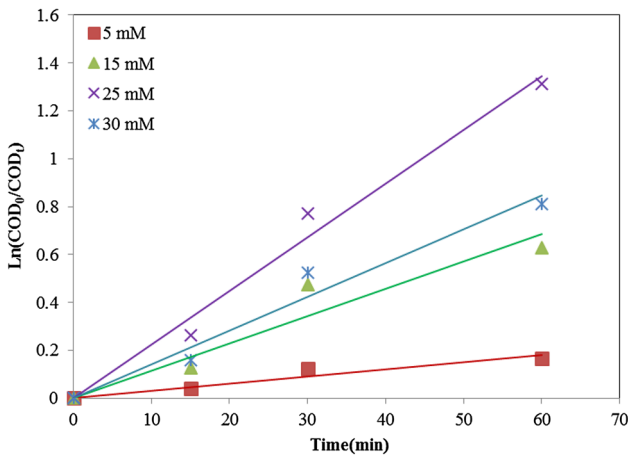

Fig. 5 First-order reaction kinetics for the degradation of MDEA (MDEA concentration, 500 ppm; temperature, 30 °C; UV, 250 W)

Table 1 Rate constants of UV/peroxydisulfate oxidation of MDEA at 30 °C

Sample number	$[S_2O_8^{2-}]_0$ (mM)	UV (W)	k_1 (min^{-1})
1	5	250	0.0030
2	15	250	0.0114
3	25	250	0.0224
4	30	250	0.0141

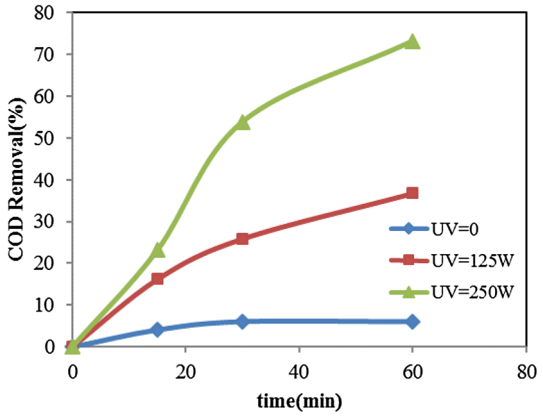

Fig. 6 Effect of UV light irradiation on the degradation of MDEA (MDEA concentration, 500 ppm; peroxydisulfate concentration, 15 mM; temperature, 30 °C)

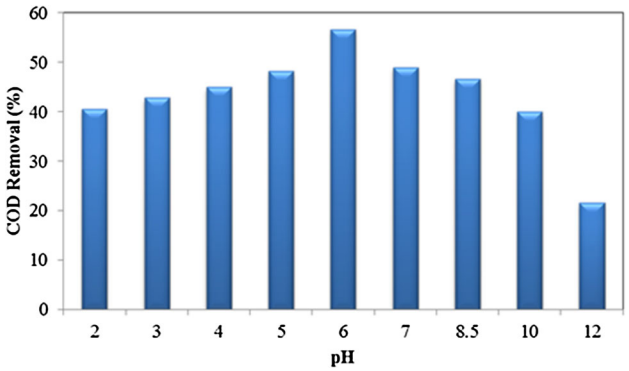

Fig. 7 Effect of pH on the oxidative degradation of MDEA (MDEA concentration, 500 ppm; peroxydisulfate concentration, 15 mM; temperature, 30 °C; UV, 250 W)

induced recombination of these two radicals (Eq. 9), though this could be minor [19].

Under acidic conditions, more $SO_4^{\circ-}$ can be generated according to Eqs. 14 and 15 [41], which can enhance the degradation efficiency of MDEA. Under alkaline conditions, $SO_4^{\circ-}$ may react with OH^- to form OH° [41, 42], according to Eq. 16. When the pH value exceeded 7, a base conversion of $SO_4^{\circ-}$ to OH° made OH° the dominant radical species [41]. OH° may have a very poor reactivity with MDEA in aqueous solutions. Accordingly, under alkaline conditions, OH^- may play a role of free radicals scavenger and, resulted in a decrease in the degradation of MDEA. Additionally, carbon oxide formed from the degradation of MDEA could lead to the formation of bicarbonate and carbonate ions under alkaline conditions, which may inhibit the degradation of MDEA [43].

$$S_2O_8^{2-} + H^+ \rightarrow HS_2O_8^- \quad (14)$$

$$HS_2O_8^- \rightarrow SO_4^{\circ-} + SO_4^{2-} + H^+ \quad (15)$$

$$SO_4^{\circ-} + OH^- \rightarrow SO_4^{2-} + OH^\circ. \quad (16)$$

Effect of the temperature

Temperature influence on the used processes was investigated in the range of 30–50 °C. The results for the UV/$S_2O_8^{2-}$ in constant concentration of MDEA of 500 ppm, initial peroxydisulfate concentration of 15 mM, irradiation of 250 W and pH = 8.5 are shown in Fig. 8.

Temperature helps the degradation reaction to compete more effectively according to the Arrhenius equation; however, at the same time, it reduces the oxygen solubility in water which is not desirable [44].

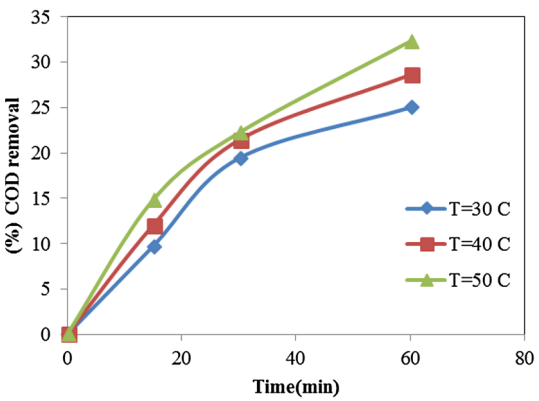

Fig. 8 Effect of the temperature on the oxidative degradation of MDEA (MDEA concentration, 1000 ppm; peroxydisulfate concentration, 15 mM; UV, 125 W)

Conclusions

In this work degradation of MDEA by peroxydisulfate along with UV irradiation is known an effective and safe method was studied. The following conclusions were reached:

- Almost no MDEA removal was achieved using UV irradiation alone and using peroxydisulfate alone removal percentage was obtained about 7 %. Finally, more than 75 % of MDEA concentration removed using UV irradiation and peroxydisulfate simultaneously.
- Increase in MDEA concentration would decrease the degradation. For example, the degradation of 50 % was observed in 500 ppm, while only degradation of 19 % was obtained in 1500 ppm concentration of MDEA.
- In terms of the changed of peroxydisulfate concentration, i.e., 0 to 25 mM, the COD removals increased.
- The COD removal increased as temperature and UV irradiation intensity increased.
- The solution pH had a major influence on MDEA degradation in the UV/$K_2S_2O_8$ process. The range of optimum solution pH was 6–7; however, all levels of pH demonstrate satisfactory removal but these ranges can be better for industrial conditions.
- Increase in the temperature would increase the degradation according to the Arrhenius equation. Increasing UV irradiation intensity increases removal by improving production of sulfate and hydroxyl radicals.
- The optimum operating conditions, which showed that the initial MDEA concentration of 500 ppm, UV irradiation of 250 W, initial peroxydisulfate concentration of 25 mM, and a temperature of 30 °C were the best conditions. Under the optimized conditions, the maximum degradation of MDEA was 75 %.

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