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Application of Response Surface Method in the Degradation of Wastewater Containing MDEA using UV/H_2O_2 Advanced Oxidation Process

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Abstract: Methyldiethanolamine (MDEA) solution is frequently used for the removal of acidic gases from raw natural gas. During maintaining, cleaning and scheduled inspection of absorption and desorption column of sweetening gas plant, a high concentration MDEA generated in the wastewater. This wastewater is toxic to bacteria and usually can not be treated using conventional biological treatment. Popular technique called Advanced Oxidation Processes (AOP's) such as UV/H_2O_2, Fenton's treatment and UV/Ozone has been used to treat the high concentration of organic contaminant in the wastewater. This study involves the UV/H_2O_2 advanced oxidation process to treat wastewater containing MDEA. Response Surface Method (RSM) was used to optimize the factor condition on MDEA degradation process. Approximately 85% of Total Organic Carbon (TOC) was removed. The biodegradability of partially degraded MDEA after UV/H_2O_2 was determined by calculating of the BOD_5/COD ratio. The ratio increased after UV/H_2O_2 process.

Key words: Methyldiethanolamine, advanced oxidation processes, UV/H_2O_2, total organic carbon, response surface methodology

INTRODUCTION

Raw natural gas contains acidic gases such as H_2S and CO_2. These acidic gases required to be removed since these gases are corrosive and toxic to the environment, especially H_2S. Alkanolamine such as monoethanolamine (MEA), diethanolamine (DEA), methyl-diethanolamine (MDEA) and diisopropanolamine (DIPA) are commonly used for the removal of acidic gas in the sweetening gas plant (Kohl and Niielsen, 1997). During cleaning, maintaining and scheduled inspection of absorption and desorption column, high concentration of alkanolamine are generated into the wastewater. This wastewater is toxic to bacteria and usually can not be treated using conventional biological treatment. In this recent two decades, some popular techniques have been used to treat the high concentration of organic contaminant in the wastewater called Advanced Oxidation Processes (AOP's). The most famous of AOP's are UV/H_2O_2, Fenton's treatment and UV/Ozone. These techniques involve a reactive species called hydroxyl radical ($^\bullet$OH). The hydroxyl radical will degrade the contaminant into the less toxic species. According to the UV/H_2O_2 process, generation of hydroxyl radical follows the following (Jones, 1999; Koprivanac and Kusic, 2007; Oppenlander, 2003; Tang, 2004):

$$H_2O_2+hv\rightarrow 2\bullet OH \quad (1)$$

As reported by Furhacker *et al.* (2003), MDEA is non-biodegradable for the test period of 28 days in the batch bioreactor. Therefore, this study was conducted to introduce the UV/H_2O_2 advanced oxidation process for the treatment of wastewater containing MDEA. This method was selected due to its advantages such as: no formation of sludge during the treatment, high ability to produce hydroxyl radical ($^\bullet$OH) and also applicable on the wide range of pH. Laboratory works were conducted to study the effect of parameters involved in the degradation of MDEA. The factor parameters studied are initial concentration of contaminant, initial concentration of hydrogen peroxide (H_2O_2), initial pH condition, temperature, irradiation period and UV intensity. Experimental results indicated that the oxidation was influenced by all factor parameters. Therefore, statistic software was used to determine the most influential factor parameter in the oxidation. Response Surface Methodology (RSM) was used during optimization. This RSM method was able to verify the optimum point of the oxidation process condition which was hidden on the experimental works.

A large number of experimental works on the application of AOP's to treat high concentration of waste have been reported in the literature. The use of Fenton' reagent in the AOP's was conducted to degrade MEA (monoethanolamine) (Harimurti *et al.*, 2008; Harimurti *et al.*, 2010; Sominidevi *et al.*, 2007), DEA

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(diethanolamine) (Dutta *et al.*, 2010), salfolen (Khamarudin *et al.*, 2008) and DIPA (diisopropanolamine) (Omar *et al.*, 2010). Meanwhile, the use of UV/H_2O_2 in the advanced oxidation process had been also examined. Ariff *et al.* (2008) conducted the degradation of MEA. The effectiveness of UV/H_2O_2 to decolorize azo-dye was studied by Malik and Sanyal (2004) and Muruganandham and Swaminathan (2004). The degradation of 4-nitrophenol using UV/H_2O_2 was reported by Daneshvar *et al.* (2007). Lopez *et al.* (2000) successfully degraded 4-chloro-3,5-dinitrobenzoic acid (CDNBA) in aqueous solution. Degradation and detoxification of formaline wastewater using UV/H_2O_2 was greatly achieved by Kajitvichyanukula *et al.* (2006). Mamane *et al.* (2007) studied inactivation of microbiocidal (*E. coli* and *B. subtilis* spores) and virucidal (MS2, T4 and T7 phage) using UV/H_2O_2. Riga *et al.* (2007) investigated the effect of the presence of inorganic salt on advanced oxidation.

This study involved the use of Response Surface Method (RSM) in the optimization of the oxidation process condition of MDEA by using UV/H_2O_2 advance oxidation process. Experimental work had been conducted and the optimum process conditions had been determined as well. Moreover, this study also explained the biodegradability of MDEA after UV/H_2O_2 treatment compared to the un-treatment MDEA.

MATERIALS AND METHODS

Reagent: MDEA, potassium permanganate ($KMnO_4$), sulfuric acid (H_2SO_4) and hydrogen peroxide (H_2O_2) were obtained from Merk (Germany). Sodium hydroxide (NaOH) was obtained from RM chemicals (Malaysia). Synthetic wastewater was prepared by dissolving MDEA with desired concentration in distilled water. Effluent containing MDEA was obtained from local refinery in Malaysia.

Experiment: The experiments were conducted in 400 mL stirred jacketed glass reactor to monitor the progress degradation. The reaction zone is a cylindrical borosilicate glass tube of 14 inch long with internal diameter 2 inch. The photo reactor is equipped with a low pressure Hg vapour lamp, a currents voltage control unit and a hole for collecting the sample. A solution with known concentration of amine was taken in the glass reactor. A required amount of H_2O_2 was added in to the amine solution. Total volume of amine solution and H_2O_2 was 400 mL. The pH adjustment was made using NaOH/H_2SO_4. The temperature was maintained by circulating cooling water through the jacket. During the process, samples of the liquid were withdrawn from time to time and the TOC of the samples were measured using TOC analyzer (Shimadzu), while the H_2O_2 concentration was measured using $KMnO_4$ titration (Mendham *et al.*, 2000). Furthermore, the BOD_5 was measured following the HACH method for BOD_5 determination (HACH-BODTrak^{TM}). Seed of bacteria for BOD_5 determination was using an activated sludge which was collected from Wastewater Treatment Plant (WWTP) Universiti Teknologi PETRONAS, Malaysia. This seed was selected because it was a very common bacteria. Therefore, no special or adapted bacteria needed to reproduce the result. The Chemical Oxygen Demand (COD) was measured by using COD Test' N Tube (HACH) and the value of COD reading was measured using spectrophotometer DR 5000 (HACH). Moreover, the biodegradability was calculated from the BOD_5/COD value.

Statistical design experiment: RSM is a method used in modelling and analysis of problem in which a response of importance is influenced by several factor. The objective of this method is to optimize this response. The most of response surface represented graphically where the contour plot is often drawn to visualize the shape of the response surface (Daneshvar *et al.*, 2007). When, the process is close up to optimum, the second order model that incorporates curvature is usually required to approximate the response:

$$y = \beta_o + \sum_{i=1}^{k}\beta_i x_i + \sum_{i=1}^{k}\beta_{ii} x_i^2 + \sum_{i<j}\sum \beta_{ij} x_i x_j + \varepsilon \quad (2)$$

where, y is the predicted response, β_0 the constants term, β_i the linear effect, β_{ii} the squared effect and β_{ij} represents the interaction effect. After conducting the screening of factor with the factorial design, a response surface analysis was employed to optimize the highest TOC removal of the waste. The results of experimental design was analyzed using Portable Statgraphics Centurion 15.2.11.0 statistical software to approximate the response of dependent response variable and to find the effects, coefficients, standard deviation of coefficients as well as other parameter of the model. The optimum conditions was obtained from contour plot graphically and also by solving the polynomial regression equation. Quality of fit was expressed by coefficient of determination R^2 and statistical significance was analyzed in Analysis of Variance (ANOVA) in which the level of significance at 5% probability level was given as value of $p<0.05$ (Lim *et al.*, 2009).

RESULTS

Screening of independent factor affecting the TOC removal was carried out according to the Table 1 in duplicate with 2 blocks of experiment and each block containing 1 center point. In order to understand and evaluate of the effect of factors and the interactions between the factors, where any value is larger than an absolute value is considered significant. Pareto chart of standardize effect at $p = 0.05$ (Fig. 1) shows that the factor parameter of temperature has no significance for the contribution of TOC removal. Furthermore, determination of the optimum conditions for the oxidation process carried out by using Response Surface Methods (RSM). Experiments carried out in accordance with Table 2 and 3 in the duplication of the full 3^2 factorial with 1 center point. Range and level of factors was based on initial screening results which indicated the presence of curvature in the regions relatively close to the optimum. Fitting of experimental data into the second order polynomial model (Eq. 2) resulted a curvature as depicted in Fig. 2-5. The 3D of curvature can be seen in Fig. 3 and 5. Based on the regression analysis, a second order model is expressed in Table 4. Optimum condition of oxidation process for "Pure" MDEA and local refinery effluent are similar. However, the local refinery effluent requires lower pH and higher H_2O_2 initial concentrations compared to that for "Pure" MDEA. The optimum condition of oxidation process for the "Pure" MDEA and local refinery waste by using UV/H_2O_2 are summarized in Table 5.

Table 1: The input factors of H_2O_2/temperature/pH system

Block	H_2O_2 (M)	Temp (°C)	pH	TOC removal (%)
1	0.24	30	5	47.36
1	0.12	50	5	38.48
1	0.12	30	5	35.28
1	0.12	50	9	43.92
1	0.24	50	5	46.42
1	0.12	30	9	48.59
1	0.18	40	7	68.01
1	0.24	30	9	82.45
1	0.24	50	9	75.61
2	0.24	30	5	50.57
2	0.12	50	5	41.32
2	0.12	30	5	34.76
2	0.12	50	9	43.92
2	0.24	50	5	46.40
2	0.12	30	9	43.47
2	0.18	40	7	67.94
2	0.24	30	9	85.74
2	0.24	50	9	75.80

	Levels	
Factors	Low level	High level
A: H_2O_2 (M)	0.12	0.24 M
B: Temperature (°C)	30	50
C: pH	5	9

The biodegradability studies were conducted by calculating the ratio between the BOD_5 value and COD value. Figure 7 and 8 show the result of biodegradability evaluation. The biodegradability was increased after UV/H_2O_2 oxidation process.

DISCUSSION

In this study, screening of the independent factors was conducted for concentration of H_2O_2, pH and temperature while other factors such as period of irradiation, UV intensity and initial waste concentration were keep constant. The influence factor was determined by measuring the percentage of TOC removal. Figure 1 shows that temperature is not significant in the

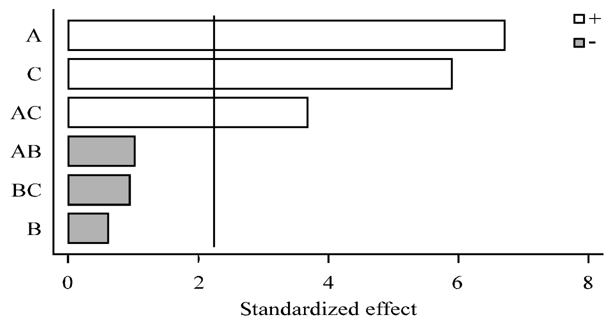

Fig. 1: Pareto chart of the standardized effect for percentage TOC removal, A: H_2O_2, B: Temperature, C: pH

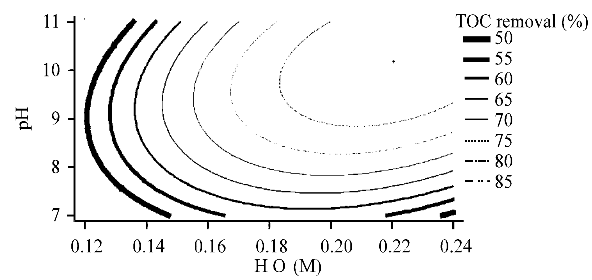

Fig. 2: Contour plots of TOC removal for pure MDEA

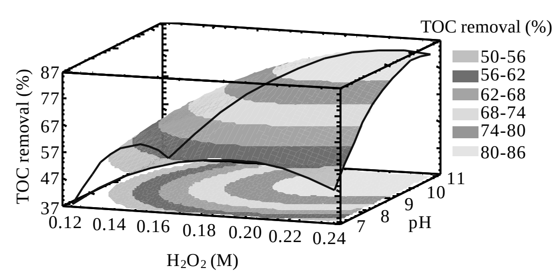

Fig. 3: 3D Contour plots of TOC removal for pure MDEA

Table 2: The input factors of H_2O_2 and pH system for pure MDEA

Block	H_2O_2 (M)	pH	TOC removal (%)
1	0.18	7.0	59.04
1	0.18	9.0	76.94
1	0.24	7.0	47.37
1	0.12	9.0	49.36
1	0.24	9.0	79.22
1	0.18	9.0	76.94
1	0.12	11.0	38.63
1	0.12	7.0	37.31
1	0.24	11.0	83.52
1	0.18	11.0	74.30
2	0.18	7.0	59.00
2	0.18	9.0	76.07
2	0.24	7.0	47.36
2	0.12	9.0	49.29
2	0.24	9.0	79.84
2	0.18	9.0	76.94
2	0.12	11.0	37.91
2	0.12	7.0	37.21
2	0.24	11.0	80.49
2	0.18	11.0	73.63

	Levels	
Factors	Low level	High level
A: H_2O_2 (M)	0.12	0.24
B: pH	7	11

Table 3: The input factors of H_2O_2 and pH system for local refinery effluent

Block	pH	H_2O_2 (M)	TOC removal (%)
1	9.0	0.18	76.47
1	11.0	0.30	65.47
1	11.0	0.24	75.46
1	9.0	0.24	82.87
1	9.0	0.30	72.68
1	7.0	0.30	42.32
1	9.0	0.24	83.39
1	11.0	0.18	67.35
1	7.0	0.18	55.55
1	7.0	0.24	56.67
2	9.0	0.18	77.36
2	11.0	0.30	64.98
2	11.0	0.24	76.69
2	9.0	0.24	82.87
2	9.0	0.30	74.03
2	7.0	0.30	42.13
2	9.0	0.24	83.39
2	11.0	0.18	69.50
2	7.0	0.18	55.65
2	7.0	0.24	56.64

	Levels	
Factors	Low level	High level
A: pH	7	11
B: H_2O_2 (M)	0.18	0.30

Table 4: The regression coefficients of second order model (Eq. 2) for TOC Removal of pure MDEA and local refinery effluent

Coefficient	Estimate (pure MDEA)	Estimate (local refinery effluent)
Constant	-256.379	-376.674
A: H_2O_2 (M)	1002.31	857.123
B: pH	45.2796	76.2414
AA	-3887.28	-2299.82
AB	70.0625	21.2056
BB	-2.98192	-4.2626
R^2 (%)	99.52	99.36
Adjusted for R^2 (%)	99.35	99.13

Table 5: The optimum condition for oxidation of pure MDEA and local refinery effluent by using UV/H_2O_2

Factor	Optimum condition (pure MDEA)	Optimum condition (local refinery effluent)
H_2O_2 (M)	0.22 M	0.23 M
pH	10.18	9.52
Maximum value of TOC removal, predicted	84.82%	84.73%
Maximum value of TOC removal, experimental	85.74%	85.99%

The initial concentration of TOC: 1000 ppm, Total volume: 400 mL, UV light intensity: 12.06 mW cm^{-2} and temperature: 30°C

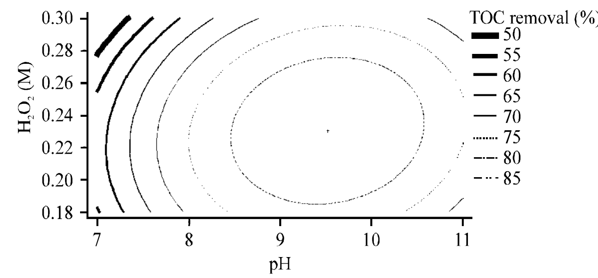

Fig. 4: Contour plots of TOC removal for local refinery effluent

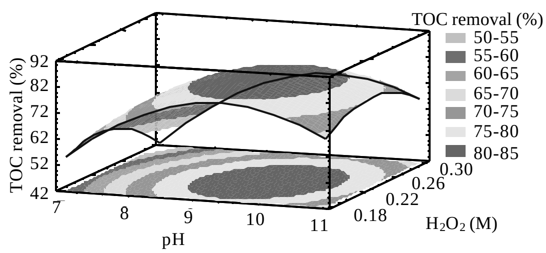

Fig. 5: 3D Contour plots of TOC removal for local refinery effluent

contribution of TOC removal. Hence, the temperature factor was eliminated for this study and the further experiments were performed using 30°C as the temperature adjustment.

The optimum response and relationship between the factors and response were obtained by using response surface method. The quadratic regression model (Eq. 2) for the percentage TOC removal of pure MDEA and local refinery effluent are given in the Table 4.

In order to examine the effect of factors towards the response, a graphical representation known as contour plots of model regression from Table 4 were used and are shown in Fig. 2-5. The 3D of curvature can be seen in Fig. 3 and 5. As illustrated in Fig. 3 and 5, increasing concentrations of H_2O_2 to a certain level causes an increase in TOC removal. This is due to the increasing sources of hydroxyl radicals. As a result, the processes of

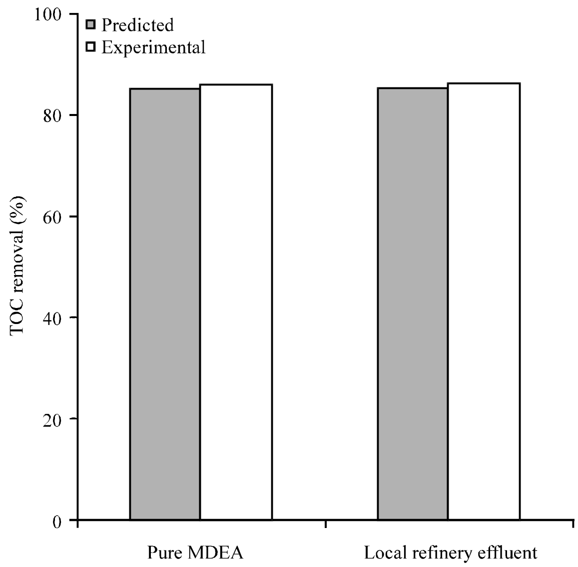

Fig. 6: TOC removal predicted compare to observe experimental

oxidation increase as well. Therefore, TOC removal increased. Further increase of H_2O_2 will decrease the TOC removal. The decrease of TOC removal related to the scavenging effect of H_2O_2 itself (Eq. 3-6). Hence, it will decrease the H_2O_2 concentration in the system. In addition, by increasing the pH to a certain level will increase the removal of TOC. This condition is due to hydroxyl radical oxidation mechanism for compounds containing nitrogen atoms is better at high pH (Klare *et al*., 2000). But at still higher pH conditions would reduce the TOC removal. It is associated with the decomposition of H_2O_2 at high pH. The observed experimental value by using condition at the maximum point of TOC removal was found to be 85.74% for 'pure" MDEA and 85.99% for the local refinery effluent which were in satisfactory agreement with the predicted value (Fig. 6). The optimum conditions that obtained for oxidation are given in Table 5. The difference of the optimum process condition of oxidation of pure MDEA and effluent from the refinery may be due to the different composition of the effluent. Effluent from the refinery may contain many other contaminants except MDEA which are generated during the sweetening process of natural gas:

$$H_2O_2 \leftrightarrow HO_2^- + H^+ \quad (3)$$

$$^\bullet OH + H_2O_2 \rightarrow HO_2^\bullet + H_2O \quad (4)$$

$$^\bullet OH + HO_2^- \rightarrow HO_2^\bullet + HO^- \quad (5)$$

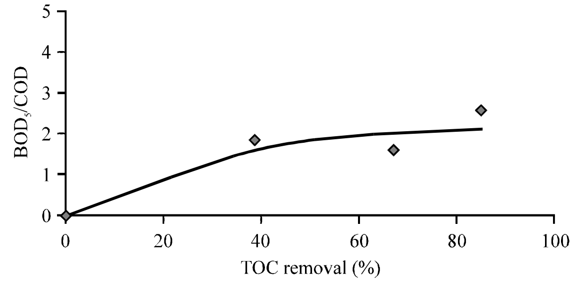

Fig. 7: The biodegradability evaluation of pure MDEA

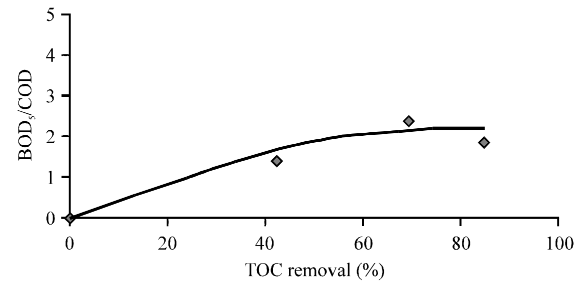

Fig. 8: The biodegradability evaluation of local refinery effluent

$$HO_2^\bullet + ^\bullet OH \rightarrow H_2O + O_2 \quad (6)$$

The degradation experiment of MDEA by UV/H_2O_2 oxidation showed 85% of TOC removal. Complete TOC removal could not be achieved even with a high concentration of hydrogen peroxide. In fact, UV/H_2O_2 treatment is suitable for fractional degradation of organic compounds. This partially degraded compound has low toxicity fragments compared to the pure compound. Hence, it will be easy to degrade by micro-organism.

The biodegradability evaluation of partially degraded effluent after UV/H_2O_2 treatment was evaluated through the BOD_5/COD ratio (EUR-Lex, 2005; Berto *et al*., 2009; Chun and Yizhong, 1999; Eckenfelder *et al*., 1995; Kim *et al*., 2007; Rajeswari and Kanmani, 2009). BOD_5 is a measure for the dissolve oxygen consumption during biological oxidation of organic contaminant, while the COD is a measure for the oxygen that is required to oxidize chemical in the sample. According to the EU (Europe Union) regulation, readily biodegradable means the BOD_5/COD ratio is = 0.5 (EUR-Lex, 2005). The UV/H_2O_2 process could increase the ratio of BOD_5/COD. Result of biodegradability evaluation is depicted in Fig. 7 and 8. The higher ratio of BOD_5/COD means that partially degraded MDEA is more easily degraded by biological oxidation. It can be concluded that the UV/H_2O_2 oxidation can break down the MDEA structure and convert the non-biodegradable compound to biodegradable compound.

CONCLUSION

UV/H_2O_2 process is able to remove the TOC of wastewater containing MDEA to a certain level. The optimum condition for degradation of waste containing MDEA (local refinery effluent) was at initial concentration of contaminant equal with 1000 ppm TOC, 400 mL in total volume, 12.06 mW cm^{-2} of UV intensity, temperature at 30°C, pH of 9.52 and 0.23 M of initial H_2O_2 concentration with TOC removal after 180 minute oxidation time was 85.99%. Response Surface Method (RSM) was applicable for the optimization of the degradation study of MDEA using UV/H_2O_2. The biodegradability of partially degraded MDEA was increased after UV/H_2O_2 treatment.

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