Conditional Optimization for Treatment of Wastewater from Ethanol Production Process Using Ozonation

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Abstract

This study aimed to determine the optimal conditions for removing COD and color from ethanol production effluent using the ozonation process. The investigation employed Response Surface Methodology (RSM) with a Central Composite Design (CCD) model, focusing on two key variables: ozone dosage rate (900, 1,200, and 1,500 mg/hr) and contact time (60, 90, and 120 minutes). The results showed that the highest COD and color removal efficiencies—63.32±1.28% and 75.65±1.42%, respectively—were achieved at an ozone dosage of 1,500 mg/hr and a contact time of 120 minutes. Based on the ANOVA statistical analysis of the treatment results, the Quadratic Model and the Linear Model were found to be suitable for describing the relationship between the variables (ozone dosage rate and contact time) and the COD and color removal efficiencies of effluent from a UASB tank, which is the wastewater after preliminary treatment, using the ozonation process. According to the model, the optimal conditions for maximum treatment efficiency were an ozone dosage rate of 1,500 mg/hr and a contact time of 116.881 minutes, which predicted COD and color removal efficiencies of 63.99% and 75.79%, respectively. To validate these findings, a series of experiments was conducted under the recommended optimal conditions. The average COD removal efficiency observed was 62.99±0.68%, which closely aligns with the model prediction, with a deviation of only 1.60%. Similarly, the average color removal efficiency was 73.22±0.62%, showing a minor deviation of 3.51% from the predicted value. These results confirm the reliability and suitability of the CCD model in optimizing the ozonation process for treating effluent from ethanol production.

Introduction

The ethanol industry is a sector that requires a large amount of water in its production process, starting from the preparation of raw materials, which involves washing and cleaning before being processed into a starch solution. The process then moves on to boiling, fermentation, and distillation, re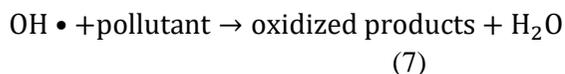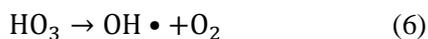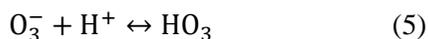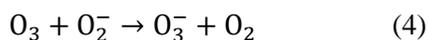spectively. The wastewater generated from production contains high levels of COD, BOD, and color, while having a low pH (high acidity), making it difficult to treat [1]. This leads to negative impacts on surrounding communities, such as the discharge of wastewater into rivers or ethanol wastewater overflowing into residential areas. The overflowing wastewater is characterized by a brown color, an unpleasant odor, and chemical contamination. These issues not only affect local communities but also pose significant environmental concerns [2].

Currently, the ethanol industry often adopts anaerobic wastewater treatment systems. These systems help reduce costs by eliminating the need for aeration and generating less sludge, thereby lowering sludge disposal expenses. Additionally, anaerobic treatment can produce biogas, mainly composed of methane gas, which can be used as fuel or an alternative energy source within the factory, leading to further energy cost savings [3]. Nevertheless, although anaerobic systems are widely utilized in ethanol wastewater treatment, the treated water often has a brown color and high COD levels that do not meet discharge standards. There are also issues related to unpleasant odors. To address these problems, various treatment methods have been explored, including membrane filtration, activated carbon adsorption, ozonation, and the Fenton process. Each method has its limitations depending on the treatment process. Previous studies have shown that ozonation is effective in removing color and organic substances from ethanol production wastewater [4]. However, prior research lacks an in-depth study on determining the optimal conditions of ozone dosage and contact time using Response Surface Methodology (RSM) specifically for treating wastewater from the ethanol production process. Therefore, this research was conducted to study the optimal conditions for treating wastewater from the ethanol production process using the ozonation method, by comparing the water quality before and after treatment.

Ozonation is currently used in wastewater treatment, as it does not produce toxic byproducts in the treated water. This process involves removing pollutants in the form of biologically non-degradable organic compounds through a chemical reaction using ozone. Ozone is a powerful oxidizing agent capable of breaking down complex organic molecules. Additionally, ozone is an unstable gas that can decompose into oxygen, releasing various radicals such as hydroxyl radicals ($OH \bullet$) and superoxide anion ($O_2^{\bullet-}$). These radicals are highly reactive and act as strong oxidizing agents, readily reacting with various substances. The use of ozone in degrading organic matter in water reduces the toxicity of these compounds and enhances their biodegradability [5, 6]. Furthermore, COD and many colored and complex organics are broken into smaller, less colored or colorless compounds. Ozone is also a powerful disinfectant, making it highly effective in eliminating pathogens [7]. The key reactions in the ozonation process for wastewater treatment occur through both direct and indirect oxidation mechanisms. In direct oxidation, ozone reacts directly with certain organic and inorganic compounds via electrophilic attack, particularly targeting functional groups such as double bonds, activated aromatic rings, or amines, as illustrated in Equation 1. In indirect oxidation, ozone decomposes in water, especially under alkaline conditions, to generate hydroxyl radicals ($OH \bullet$), which are highly reactive and non-selective oxidants (Equations 2–6). Once formed, these hydroxyl radicals can rapidly oxidize a wide range of organic pollutants, often more efficiently than ozone itself, as shown in Equation 7 [8].

$$O_3 + \text{pollutant} \rightarrow \text{oxidized products} + \text{byproducts} \tag{1}$$

$$O_3 + OH^- \rightarrow HO_2^- + O_2 \tag{2}$$

$$HO_2^- \leftrightarrow O_2^- + H^+ \tag{3}$$

$$O_3 + O_2^- \rightarrow O_3^- + O_2 \qquad (4)$$

$$O_3^- + H^+ \leftrightarrow HO_3 \qquad (5)$$

$$HO_3 \rightarrow OH \bullet + O_2 \qquad (6)$$

$$OH \bullet + pollutant \rightarrow oxidized\ products + H_2O \qquad (7)$$
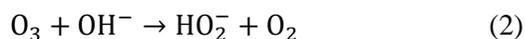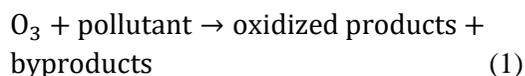
This study focused on two primary variables influencing pollutant removal efficiency: ozone dosage rate and contact time. To evaluate their effects and interactions, the Central Composite Design (CCD) approach was utilized through the Design Expert software. This statistical method enabled a structured assessment of how these factors impact treatment performance. Moreover, the integration of advanced analytical tools and computational techniques has significantly improved the ability to determine the most effective treatment parameters. Design Expert's Response Optimizer feature further aids in forecasting and confirming the optimal conditions for achieving maximum removal efficiency.

Methodology

This research emphasizes determining the optimal conditions for treating wastewater from the ethanol production process using the ozonation method. The study commenced with the collection of wastewater samples from the effluent discharge point after the UASB (Upflow Anaerobic Sludge Blanket) reactor of an ethanol production plant in Ubon Ratchathani province. Wastewater samples were collected every two weeks from November to December

2023. The collection, preservation, and handling of wastewater samples were performed in compliance with Standard Methods for the Examination of Water and Wastewater [9]. An experimental design was then developed based on the principles of RSM. Treated water samples were analyzed to evaluate treatment efficiency and validate the accuracy of the mathematical model generated by the software. The CCD experimental design was executed as a batch experiment, with each experimental run performed in duplicate to determine the mean and standard deviation. The characteristics of the wastewater sample were analyzed according to standard methods, as displayed in **Table 1** [10]. Based on primary research, two key process variables, ozone dosage rate and contact time, were adjusted to values of 900, 1,200, and 1,500 mg/hr, and 60, 90, and 120 minutes, respectively [4, 11]. The ozone dosage rate generated by the ozone generator was analyzed following the standard method [10].

The optimization process using RSM involves designing a structured set of experiments where key variables are systematically adjusted. The experimental data are then used to develop a mathematical model that illustrates the relationship between the independent variables (factors) and the dependent variable (response). This model enables the prediction of the response outcome based on different combinations of factor levels. Additionally, it plays a crucial role in determining the optimal conditions for enhancing contaminant removal, reducing chemical oxygen demand (COD), and improving the color and odor quality of the treated wastewater [12-14]. The RSM model is typically represented by the following equation [15].

Table 1 Parameters and methods for analyzing characteristics of wastewater from ethanol production

Parameter	Analysis method
pH	pH Meter
COD (mg/L)	Closed Reflux Colorimetric Method
Color	ADMI Method

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum \sum_{i<j} \beta_{ij} x_i x_j + \varepsilon \tag{8}$$

Where y is the model response, β_0 is the intercept, β_i are the linear coefficients for each independent variable, β_{ii} are the quadratic coefficients for the squared terms of each independent variable, β_{ij} are the interaction coefficients between the variables X_i and X_j, X_i and X_j are the independent variables, k is the number of independent variables, and ε is the error term. This equation is essential for understanding the impact of changes in process variables on the response and plays a crucial role in process optimization.

Results and Discussion

The preliminary analysis of the wastewater samples revealed that the effluent from the ethanol production process contains relatively high levels of pollution, particularly in terms of COD and color, as shown in **Table 2**. This indicates a significant load of organic and inorganic substances in the wastewater [11]. The effluent appears as dark brown water with black sludge, as illustrated in **Figure 1**.

Treatment of wastewater sample from ethanol production using ozonation

The study on the efficiency of COD and color removal from ethanol production wastewater using the ozonation process showed that color could be reduced to 173±15.6 ADMI, and COD to 1,772±82.5 mg/L. The resulting color value meets the industrial effluent standards set by the Ministry of Industry, while the COD value exceeds the permissible limit. Additionally,

experimental results revealed an increase in the pH of the treated water, as shown in Table 2. The rise in pH is attributed to the formation of hydroxide ions (OH⁻) during the reaction, leading to a condition where the concentration of hydroxide ions [OH⁻] is greater than that of hydronium ions [H_3O^+], resulting in a more basic solution and, consequently, a higher pH after treatment [17]. **Figure 2** illustrates the physical characteristics of the wastewater sample and treated water. As seen from the figure, the color of the water sample changed from dark brown to lightly cloudy white.

A comparison between the experimental results and the predicted values for COD and color removal efficiencies obtained from the Design-Expert software is demonstrated in **Table 3**. It should be noted that the "Prediction" values in the table are calculated using the derived Quadratic and Linear equations (Equations 9 and 10), respectively. The results indicate that the predicted COD removal efficiencies closely match the experimental data. Similarly, the color removal efficiencies obtained from both the experimental results and the Design-Expert predictions are also comparable, as shown in **Table 3**, further confirming the model's accuracy. The highest COD and color removal efficiencies were observed at an ozone dosage rate of 1,500 mg/hr and a contact time of 120 minutes, achieving 62.93% and 75.92% removal efficiencies, respectively. In contrast, the lowest removal efficiencies were found at an ozone dosage rate of 900 mg/hr and a contact time of 60 minutes, with COD and color removal rates of 11.86% and 62.54%, respectively.

Table 2 Wastewater characteristics from ethanol production

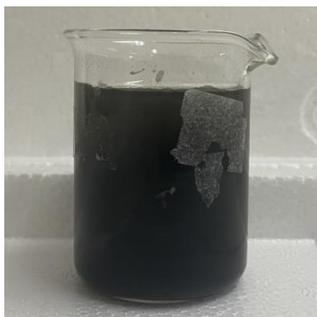| Parameters	Average value before treatment	Average value after treatment	Standard values of effluent discharge [16]
pH	7.59±0.2	8.68±0.3	5.5 – 9.0
COD	4,830±247.5 mg/L	1,772±82.5 mg/L	≤ 120 mg/L
Color	708±7.1 ADMI	173±15.6 ADMI	≤ 300 ADMI

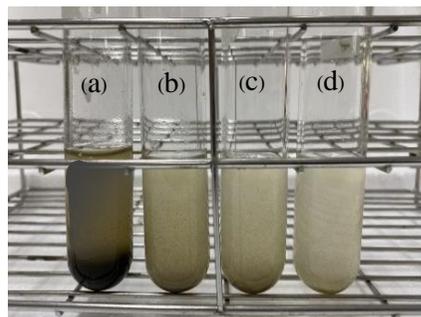**Figure 1** Wastewater sample from ethanol production

Figure 2 Physical characteristics of the wastewater sample and treated water at different contact times (a) 0 min, (b) 60 min (c) 90 min (d) 120 min

Table 3 Comparison between experimental and predicted values of removal efficiency

No.	Ozone dosage rate (mg/hr)	Contact time (min)	Removal efficiency (%)			
			COD		Color	
			Experiment	Prediction	Experiment	Prediction
1	900	60	14.14±3.07	11.86	62.37±1.90	62.54
2	900	90	40.63±4.29	42.21	70.52±0.14	70.20
3	900	120	51.59±1.26	53.10	72.96±1.41	73.11
4	1200	60	22.74±1.78	29.19	63.74±0.22	63.29
5	1200	90	56.52±0.73	53.34	72.96±0.69	73.11
6	1200	90	54.39±5.32	53.34	72.91±3.44	71.17
7	1200	90	55.06±6.89	53.34	71.15±3.59	71.17
8	1200	90	55.48±2.07	53.34	69.75±0.17	71.17
9	1200	90	52.20±6.73	53.34	68.22±1.99	71.17
10	1200	120	59.14±3.31	58.02	74.73±0.19	74.31
11	1500	60	50.70±5.02	46.53	64.18±4.39	64.46
12	1500	90	59.09±1.01	64.46	73.12±1.88	72.57
13	1500	120	63.32±1.28	62.93	75.65±1.42	75.92

Statistical analysis

Based on the experimental results using the RSM, the data from Table 3 were used to identify the most appropriate mathematical model for predicting COD and color removal efficiencies in ethanol production wastewater treated by the ozonation process. The model summary statistics indicated that the quadratic model, with an adjusted R^2 value of 0.9209, was selected to describe the relationship between the process variables (ozone dosage rate and contact time) and COD removal efficiency. The linear model was suggested for the prediction of color removal efficiency. Statistical analysis revealed that the equation for the percentage of COD removal had an F-value of 41.02 and a P-value of < 0.0001. Since the P-value is less than 0.05, this indicates that the model is statistically significant

and suitable for explaining the relationship between COD removal efficiency and the studied variables in the ozonation process. Furthermore, the coefficient of determination (R^2) was found to be 0.9535, as presented in Table 4, indicating that 95.35% of the experimental data can be explained by the predicted values from the mathematical model [18]. A high R^2 value signifies high accuracy in the model's predictive capability. Similarly, the adjusted R^2 (Adj R^2) was 0.9303, which is higher than the minimum acceptable value of 0.64 [10, 18-19], suggesting that the model is well-suited for predicting COD removal. The predicted R^2 (Pred R^2) was 0.7701, with a difference of only 0.1602 from the Adj R^2. Since this difference is less than 0.2, it implies that the model has no significant problems regarding prediction adequacy [19-20]. Furthermore, the coefficient of variation (C.V.) was 8.40%, which is below the 10% threshold [10, 18], indicating that the predictive model has good repeatability and precision. Additionally, the adequacy precision was 21.9545, as shown in **Table 4**. Since this value is greater than 4 [10, 18], it demonstrates that the model is reliable [10].

For color treatment, the statistical analysis revealed that the equation for the percentage of color removal had an F-value of 41.56 and a P-value of < 0.0001, which is less than 0.05. This indicates that the model is statistically significant and suitable for explaining the relationship between the color removal efficiency and the investigated factors in the ozonation process. When examining the coefficient of determination (R^2), a value of 0.9205 was obtained, as shown in **Table 5**, indicating that 92.05% of the experimental data can be explained by the mathematical model's predictions [17]. A high R^2 value suggests high accuracy in the model's ability to predict outcomes, which is essential in mathematical

modeling for experimental design. In general, R^2 values range from 0 to 1, and a good model should have an R^2 value close to 1. Likewise, the adjusted R^2 (Adj R^2) was found to be 0.8637, which exceeds the minimum acceptable value of 0.64 [10, 18-19], confirming the model's suitability. The predicted R^2 (Pred. R^2) was 0.8560, differing from the Adj R^2 by only 0.0077. Typically, this difference should not exceed 0.2, indicating that the model has no significant issues regarding prediction reliability [19-20]. Moreover, the coefficient of variation (C.V.) was only 2.28%, which is well below the 10% threshold [10, 18]. A lower C.V. implies better reliability and precision in the model's reproducibility. The adequacy precision value was 12.3015, as shown in Table 5, which is greater than 4 [10, 18], further demonstrating the model's reliability [10]. Additionally, the statistical analysis indicated that both factors had a significant impact on the removal efficiency of COD and color.

A comparison of the experimental results with other related research confirms the robustness of the findings. The maximum COD removal efficiency of 63.32±1.28% and color removal of 75.65±1.42% in this study are higher than the 43.51±1.86% COD removal reported for ozonation treatment of rubber wastewater [11]. However, the observed color removal was lower than that reported in the dye wastewater study, where efficiencies reached 98% under optimal conditions [14]. The high R^2 values of the models (0.9535 for COD and 0.9205 for color) are also consistent with the statistical accuracy found in optimization research for landfill leachate and industrial wastewater decolorization processes [12-13]. These results reinforce the potential of the ozonation process as a highly effective technology for treating this type of wastewater.

Table 4 Fit statistical results for COD removal using the ozonation process

Std. Dev.	3.86	R^2	0.9535
Mean	48.91	Adjusted R^2	0.9303
C.V. %	8.40	Predicted R^2	0.7701
Adequacy precision			21.9545

Table 5 Fit statistical results for color removal using the ozonation process

Std. Dev.	1.60	R^2	0.9205
Mean	70.17	Adjusted R^2	0.8637
C.V. %	2.28	Predicted R^2	0.8560
Adequacy precision			12.3015

The experimental data can be used to construct a mathematical model (Actual Equation) as shown in Equations 9 and 10 for COD and color removal efficiencies, respectively. Note that the last two terms in Equation 10 can be omitted, thus making Equation 10 a linear equation.

$$\%R.E.COD = -196.48857 + 0.099158A + 3.25477B - 0.000690AB - 0.010815B^2 \quad (9)$$

$$\%R.E.\ Color = 34.54805 - 0.003865A + 0.629209B + 0.0000024AB + 2.33716*10^{-6}A^2 \quad (10)$$

Where A is the ozone dosage rate (mg/hr), and B is the contact time (min).

Conditional optimization for the COD and color removal using ozonation

From the conditional optimization for treating wastewater from the ethanol production process using ozonation and based on the CCD model, a total of seven conditions were identified. However, the model recommended the optimal condition for both COD and color removal to be at an ozone dosage rate of 1,500 mg/hr and a contact time of 116.88 minutes. Under these conditions, the COD removal efficiency was 63.99%, and the color removal efficiency was 75.79%, with an overall desirability score of 0.995, as shown in **Figure 3**. Since this value is close to 1.00, it indicates that the optimal condition for treating ethanol production wastewater using the ozonation process, derived from the mathematical model, is highly reliable [21].

A set of experiments was conducted to validate the optimal treatment conditions obtained from the model. The experiment was performed in triplicate using the conditions recommended by the model, an ozone dosage rate of 1,500 mg/hr and a contact time of 117 minutes. The average COD removal efficiency was found to be 62.99 ± 0.68%. This experimental result was very close to the model's predicted value, with only a 1.602% deviation. The average color removal efficiency was 73.22 ± 0.62%, also closely matching the model's prediction, with a 3.51% deviation. These results confirm that the CCD model is both reliable and suitable for determining the optimal conditions for treating wastewater from the ethanol production process using ozonation, as the deviation between the experimental results and the mathematical model was less than 10% [10]. It is important to emphasize that the resulting model is constructed only from data within the specified experimental range (900-1,500 mg/hr and 60-120 minutes) and should not be used for prediction outside this range, as treatment efficiency may not always follow the predicted trend. Furthermore, while the absolute optimal condition found is highly accurate for the batch tested, the characteristics of wastewater from ethanol production can vary across different production batches. Therefore, the application of the developed CCD model serves as an effective tool for rapidly and accurately adjusting the optimal conditions to accommodate this wastewater variability.
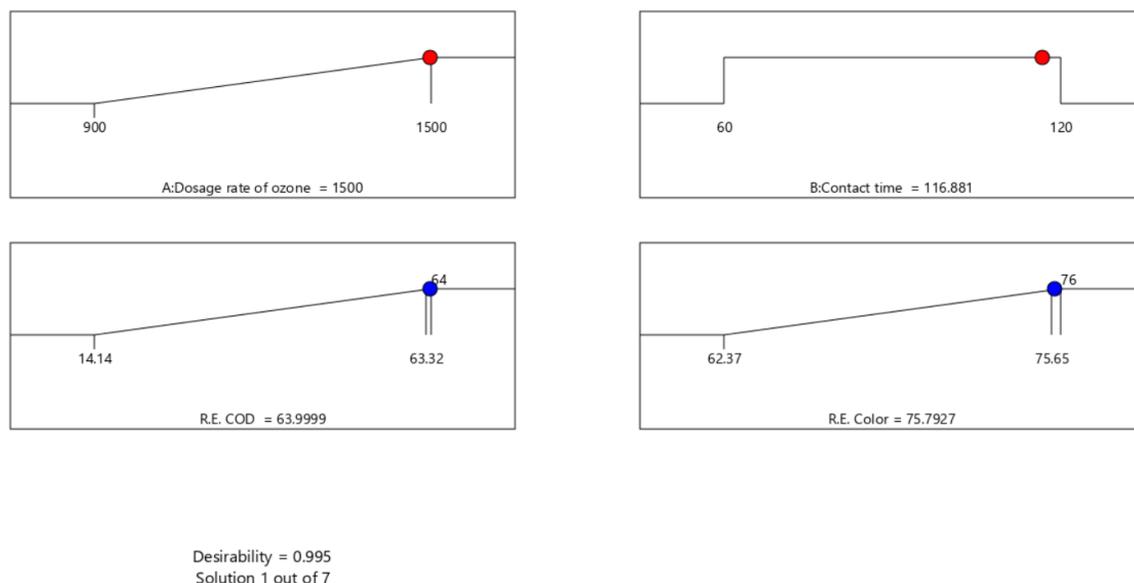
Figure 3 Desirability ramps for COD and color removal using ozonation at the optimal conditions

Conclusions

This research focuses on the treatment of wastewater from ethanol production using the ozonation process. Two key factors affecting the treatment efficiency were considered: the ozone dosage rate and contact time. It was found that an ozone dosage rate of 1,500 mg/hr and a contact time of 120 minutes resulted in the highest removal efficiencies of COD and color, which were $63.32 \pm 1.28\%$ and $75.65 \pm 1.42\%$, respectively.

Statistical analysis revealed that both factors significantly influenced COD and color removal efficiencies. A quadratic model was found to be suitable for predicting COD removal efficiency, while a linear model was appropriate for predicting color removal efficiency. Based on conditional optimization, the model recommended the optimal conditions for COD and color removal at an ozone dosage rate of 1,500 mg/hr and a contact time of 116.88 minutes, yielding a COD removal efficiency of 63.99% and a color removal efficiency of 75.79%, with a desirability of 0.995, indicating a high level of suitability. The findings from this research can be applied to wastewater treatment in ethanol production plants or other processes with similar wastewater characteristics.

Acknowledgement

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