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Optimization of micro-bubble aeration process for magnesium sulfite oxidation in desulphurization wastewater with response surface methodology

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ABSTRACT

In the magnesium-based exhaust gas cleaning system (Mg-EGCS) on board, a large amount of magnesium sulfite is generated in the washing water. In order to reduce the COD and suspended solids (SS), micro-bubbles were produced by using a porous tube in combination with an agitator stirring for the purpose of miniaturization of the bubbles and prolonging the residence time in the water to oxidize the magnesium sulfite to magnesium sulfate. The response surface methodology (RSM) was used to optimize the key parameters of the aeration process. The experimental results showed that 92.1% of the magnesium sulfite in the desulphurization wastewater was oxidized to magnesium sulfate in 54.5 min with pH of 7.5, the stirring speed of 1384 rpm and the aeration pressure of 5.44 bar.

Keywords: Magnesium-based exhaust gas cleaning system (Mg-EGCS); Magnesium sulfite; Micro-bubble aeration; Response surface methodology

1. Introduction

A novel magnesium-based exhaust gas cleaning system (Mg-EGCS), which adopts magnesium oxide or magnesium hydroxide as absorbent, has been reported in our previous studies and proven to meet the IMO sulfur emission requirements after a long time operation on board [1,2]. However, a large amount of byproduct magnesium sulfite is generated in washing water. The quality of the wastewater produced by the Mg-EGCS has been analyzed [3] and the results show that sulphite, nitrite, PAHs and total oil content are the main factors affecting the COD in the wastewater. The main ingredients in suspended solid (SS) include magnesium hydroxide, magnesium sulfite and silicon dioxide. In order to reduce the COD and SS, magnesium sulfite should be oxidized to magnesium sulfate.

As reported by the literatures, the oxygen diffusion rate is a rate-determining step of the oxidation of magnesium sulfite, and the total reaction rate is controlled by the mass transfer of oxygen from the gas phase to the liquid phase [4,5]. In the previous study on the oxidation of magnesium sulfite, fan or air compressor was often used to produce bubbles, but the size of the bubbles was normally beyond 50 μm [6]. In reference [6], an air compressor was used to study the oxidation of magnesium sulfite. The discharge pressure of the air compressor was 1.6 MPa and the power was 1.5 kW. The result showed that the oxidation rate could reach 25% with a running time of 2 h. Thus, the efficiency of oxidation was low and power consumption was high. Some researchers also used cavitating Venturi to produce bubbles, but when the flow rate increased, the size of the bubbles also increased, thus the efficiency decreased [7].

Compared with ordinary bubble aeration technology, micro-bubble aeration has a strengthening effect on the mass transfer of oxygen [8]. Although some micro-bubble aerators have been developed [9], few application of micro-bubble technology has been reported for the oxidation of the magnesium sulfite until now.

Micro-bubbles can be generated via several methods, such as acoustic cavitation, micro-fluidic oscillation, porous membranes and hydrodynamic cavitation [10,11]. Similar with porous membranes, porous medium tube is prepared based on phase separation of primary Na_2O-CaO-MgO-Al_2O_3-B_2O_3-SiO_2 type glasses and subsequent acid leaching. Micro-bubble aeration by using a porous tube promotes oxy-

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gen mass transfer [12]. The porous tube has uniform-sized tortuous pores which together form a three-dimensional interconnected network. Through the gas dispersion process, the gas phase at high pressure is forced through a porous tube into the liquid phase to form micro-bubbles. The use of the porous tube produces micro-bubbles of small and uniform size. Another advantage of this technique is that the resultant bubble size and void fraction are mainly determined by the pore size. This indicates that bubble size and void fraction can be optimized for large-scale applications.

According to the characteristics of the desulphurization wastewater and the special requirements of the ship wastewater treatment, in this work we proposed an aeration oxidation device using porous tube to produce micro-bubbles combined with an agitator stirring to make magnesium sulfite be oxidized to magnesium sulfate, and the parameters of the process was optimized with response surface methodology.

2 Materials and methods

2.1. Desulphurization wastewater

The desulphurization wastewater was obtained from the container ship "*binghe*" where Mg-EGCS was installed. The wastewater used in the experiments was from the same batch and the concentration of SO_3^{2-} was 345 mg/L.

2.2. Experimental devices

The desulfurization wastewater treatment process is the first to conduct aeration oxidation, and then solid-liquid separation, and finally the treated water is discharged to meet the emission standards [13]. The aeration oxidation process and experimental devices are shown in Fig. 1.

The micro-bubble aeration unit included an oxidation reactor, a porous tube, an air compressor, a drain pump, an agitator and a pH meter. The size of the oxidation reactor was 800 × 800 × 750 mm^3. The parameters of the porous tube are shown in Table 1. The discharge air pressure, the power and the gas flow rate of the air compressor was 0.8 MPa, 0.75kW and 40 L/min, respectively. The paddle of the agitator was PTU type, with the paddle angle of 45 degrees, the diameter of 125 mm and the height of 125 mm.

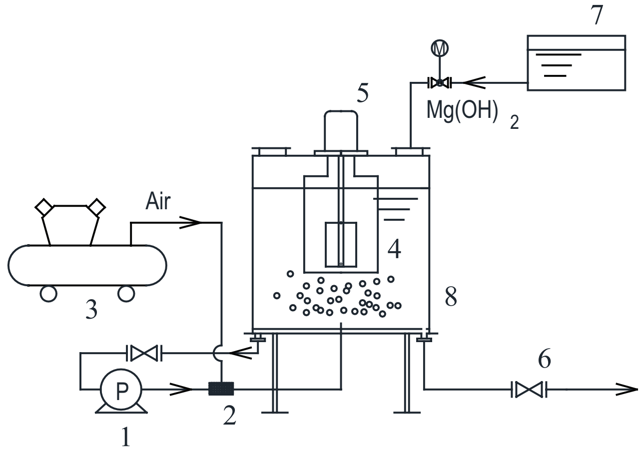

Fig. 1. Aeration oxidation process and experimental devices. 1) Drain pump; 2) Porous tube; 3) Air compressor; 4) Agitator; 5) Motor; 6) Drain valve; 7) Magnesia slurry tank; 8) Oxidation reactor.

The experiments were carried out in batch mode. In the experiment, ran the drain pump to circulate the water inside the reactor, ran the air compressor to provide air, and a large number of bubbles in the water were produced through the porous tube. In the role of agitator, the bubble was cut into smaller bubbles. Due to enhanced turbulence of water, the residence time of the bubbles in the water increased. The air compressed by an air compressor was divided into micro-bubbles through the porous tube, and then mixed with the wastewater into the oxidation reactor. There was an agitator in the oxidation reactor. With the agitator stirring, magnesium sulfite in the wastewater was oxidized to magnesium sulfate by the oxygen from the micro-bubbles. The speed of the agitator was changed by a converter. The pH of the wastewater in the oxidation reactor was adjusted by addition of slurry of magnesia.

2.3. Analytical methods

The water samples were obtained from the oxidation reactor in different experiment conditions. The samples were sampled for analysis after every experiment. Three replicates were tested: the final results came from the average thereof. The sulfite and sulfate were analyzed according to the standard methods [14]. According to the content of sulfite and sulfate in the wastewater before and after the experiments, the oxidation rate was calculated.

2.4. Variables evaluation

The oxidation of magnesium sulfite depends on many variables. Five major factors, including pH, aeration pressure, stirring speed, running time and temperature, affect the rate of magnesium sulfite oxidation, according to previous studies and literatures [15–18].

In this study, the pH of the wastewater, aeration pressure, stirring speed and running time were selected as the independent and most critical operating factors for the following reasons. In the experiment, the other variables were kept constant.

1. Aeration pressure is the main factor affecting the oxidation rate of magnesium sulfite. According to

Table 1
Parameters of porous tube

Item	Value
External diameter, mm	20
Compressive strength, MPa	0.5–1.5
Porosity, %	28–50
Filtering accuracy, µm	0.22–100
Length, mm	300

the two-film theory, with the increase of the aeration pressure, the oxygen mass transfer from the gas phase to the liquid phase increases obviously. However, further increase of the aeration pressure results in a decrease in the rate of oxidation due to the formation of larger bubbles.

A number of reports have described the dispersed gaseous phase flux in relation to bubble formation from a porous nozzle [15–17]. According to Anagbo’s report [16], the dispersion patterns of bubbles from a round porous nozzle in a water bath can be classified into three types with respect to the gaseous phase flux, i.e., low, middle, and high gas flow rate regimes. At low gas flow rates small bubbles of several millimeters in diameter are generated without coalescence or disintegration, and at moderate flow rates some of the bubbles gather and coalesce into large bubbles. In addition, at high gas flow rates, bubbles formed at the nozzle outlet are merged into a large package. These results are similar to our preliminary studies.

2. Stirring speed is also an important factor affecting the oxidation rate of magnesium sulfite. Obviously, the increase of stirring speed improves the gas-liquid contact area and reaction time, which enhances the oxidation rate of magnesium sulfite. However, if the stirring speed is too high, the bubbles collapse rapidly and the retention time decreases, which reduces the rate of oxidation.
3. The pH of the wastewater is another important factor in the oxidation of magnesium sulfite. Sulfite oxidation is believed to depend on the concentration of bisulfite [18]. The ratio of sulfite concentration to bisulfite concentration increases with increasing pH, so that the sulfite oxidation rate increases with the increase of pH value. However, when the pH value rises to 8 or more, due to the impact of increased oxygen, bisulfite concentration decreases rapidly, which results in lower oxidation rate.
4. The temperature of the waste water in the desulfurization tower is in the range of 50–60°C. Thus, a given temperature of the oxidation reactor is maintained at about 55°C.
5. Obviously, the increase of the running time resulted in higher oxidation rate of magnesium sulfite. The purpose of the study is that we want more magnesium sulfite to be oxidized to magnesium sulfate in as short a time as possible.

2.5. Experimental design

Response surface methodology (RSM) may be summarized as a collection of statistical tools and techniques for exploring an approximate functional relationship between a response variable and a set of design variables [19]. A four-factor at five-level design in the software of RSM (Design Expert 8.06) was applied in the study of the oxidation of magnesium sulfite. Each independent variable was coded at five levels between −2 and +2 at the ranges determined by the preliminary experiments, where the independent variables were pH 6–8, stirring speed 800–1400 rpm, aeration pressure 4.5–6 bar and running time 25–65 min. Table 2 shows the levels of original and coded factors.

In the optimization process, the responses can be simply related to the chosen factors by linear or quadratic models. A quadratic model, which also includes the linear model, is given as:

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{i} \sum_{j} \beta_{ij} x_i x_j + \varepsilon \qquad (1)$$

where y is the response (viz., the oxidation rate of magnesium sulfite), x_i and x_j are variables (viz., pH of wastewater, stirring speed, aeration pressure and running time), β_0 is the constant coefficient, β_i, β_{jj} and β_{ij} (i and j = 1–4) are the interaction coefficients of linear, quadratic and the second-order terms, respectively, and ε is the error.

3. Results and discussion

3.1. Evaluation of experimental results with RSM

Table 3 shows the central composite design (CCD) with pH of the wastewater, aeration pressure, stirring speed and running time for thirty experimental trials when the experimental conditions were kept as the wastewater volume of 0.3 m^3 and the temperature of 55°C.

The results obtained from RSM experiments were analyzed by multiple regression analysis and the relationship between the response and independent variables had been expressed by a second-order polynomial equation. The final models obtained in terms of coded factors are given below:

$$\begin{aligned} y = {} & 83.81 + 0.84x_1 + 3.27x_2 + 1.88x_3 + 8.4x_4 - 0.22x_1^2 \\ & -0.55x_2^2 - 1.05x_3^2 - 3.07x_4^2 - 0.001x_1x_3 + 0.13x_1x_4 \\ & +0.001x_2x_3 - 1.03x_2x_4 - 0.8x_3x_4 \end{aligned} \qquad (2)$$

Statistical adequacy of the quadratic response surface model was analyzed further using Fisher’s statistical test (*F*-test) [20]. The analysis of variance (ANOVA) results for the prescribed RSM model were summarized in the following Table 4. The results showed that the quadratic model was highly significant, implied by the high *F*-test values (3468.2) with low probability values (“Prob > *F*” <

Table 2
Level and code of experimental variables

Factor	Original factor (x)	Coded factor (X)				
		–2	–1	0	1	2
pH of wastewater	x_1	6	6.5	7	7.5	8
Stirring speed (rpm)	x_2	800	950	1100	1250	1400
Aeration pressure (bar)	x_3	4	4.5	5	5.5	6
Running time (min)	x_4	25	35	45	55	65

Table 3
CCD experimental design for the oxidation rate of magnesium sulfite by aeration

Run	x_1 [pH]	x_2 [Stirring speed] (rpm)	x_3 [Aeration pressure] (bar)	x_4 [Running time] (min)	y [Oxidation rate] (%)
1	7	1100	5	45	83.78
2	6.5	950	5.5	35	68.23
3	7	1100	4	45	75.94
4	7.5	1250	5.5	55	91.64
5	6.5	1250	5.5	55	89.71
6	7	1100	5	45	83.85
7	7	1100	5	45	83.81
8	6.5	950	4.5	55	83.01
9	7	1100	5	45	83.82
10	7	1100	5	25	54.63
11	6	1100	5	45	81.23
12	7	1400	5	45	88.07
13	7	800	5	45	75.11
14	7	1100	5	65	88.41
15	7.5	1250	4.5	55	89.44
16	6.5	1250	4.5	55	87.51
17	7	1100	5	45	83.84
18	7.5	1250	4.5	35	72.87
19	8	1100	5	45	84.61
20	6.5	950	4.5	35	62.83
21	7.5	950	4.5	55	84.93
22	7	1100	5	45	83.75
23	6.5	1250	5.5	35	76.89
24	7.5	1250	5.5	35	78.27
25	7	1100	6	45	83.25
26	7.5	950	5.5	35	69.63
27	7.5	950	4.5	35	64.23
28	6.5	950	5.5	55	85.21
29	6.5	1250	4.5	35	71.47
30	7.5	950	5.5	55	87.13

0.0001). The correlation coefficient (R^2) value of the model was 0.9984, indicating a satisfactory fitting of the quadratic model to experimental data. Besides, the value of the adjusted R^2 for the model was 0.9921, which could also confirm the accuracy of the quadratic model.

However, checking R^2 is not enough to evaluate the "Over-Fitting" problem. Generally, learning algorithms are called over-fitting if they are more accurate in fitting known data (hindsight) but are less accurate in predicting new data (foresight) [21]. The predicted R^2 can detect whether the model is over-fitted by determining the extent to which the model is generalized to other data sets [22,23]. The predicted R^2 (R_{Pred}^2) calculated by the software was 0.9891, which confirmed the validity of the model.

Table 4
Analysis of variance (ANOVA) for the developed RSM model

Statistical parameter	Value
R^2	0.9984
R_{adj}^2	0.9921
R_{Pred}^2	0.9891
F value	3468.2
Prob > F	<0.0001
LOF-F	4.72
LOF-P	0.0503

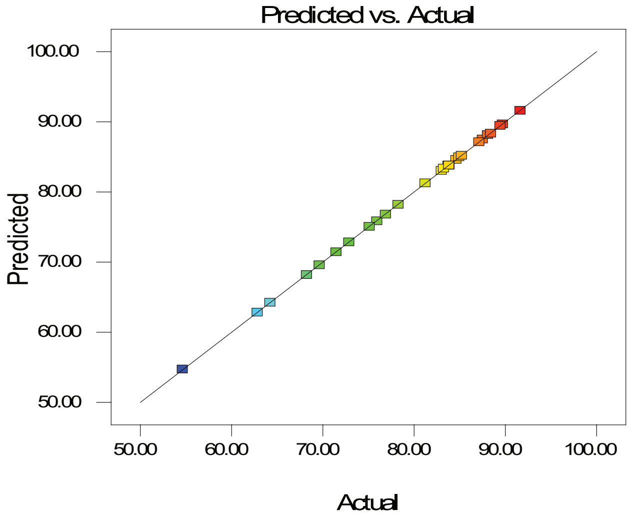

Fig. 2. Predicted *vs.* actual values plot for the oxidation rate of magnesium sulfite by aeration.

The lack of fit (LOF) F-test described the variation of the data around the fitted model. In this study, the "LOF F-value" of 4.72 was non-significant, implied a 5.03% chance that a "LOF F-value" could occur due to noise.

Fig. 2 illustrates the diagnostic plot of predicted response versus actual experimental data. The observed points on the plots revealed that the actual values were distributed quite close to the diagnostic plots line, indicating an adequate accuracy of the predicted responses *vs.* the experimental data.

3.2. Evaluation of operational parameters through response surface plot

Fig. 3 shows the effect of pH of the wastewater and aeration pressure on the oxidation rate at a constant running time of 45 min and a stirring speed of 1100 rpm. The oxidation rate increased slowly with an increase of pH at aeration pressure of 4.0 bar. The oxidation rate increased rapidly with increasing aeration pressure until the aeration pressure was 5.5 bar. When the aeration pressure more than 5.5 bar, the oxidation rate decreased slightly. The reason is that the aeration pressure is too high, resulting in shortened residence time of the air in the water, thereby reducing the oxidation rate. Thus, the maximum oxidation rate for magnesium sulfite at 45 min was 85.3% at a pH of 8.0 and aeration pressure of 5.45 bar.

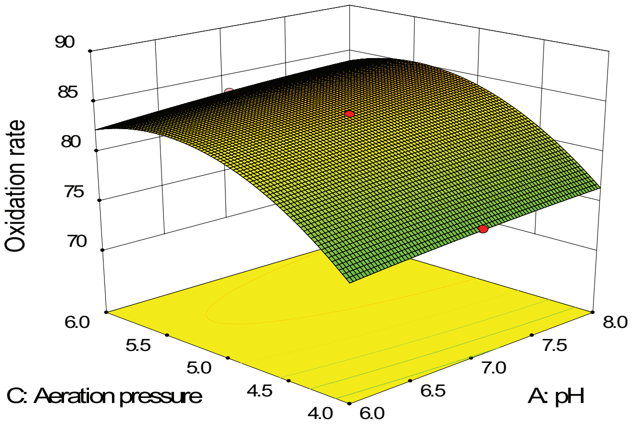

Fig. 3. Effect of pH and aeration pressure on oxidation rate.

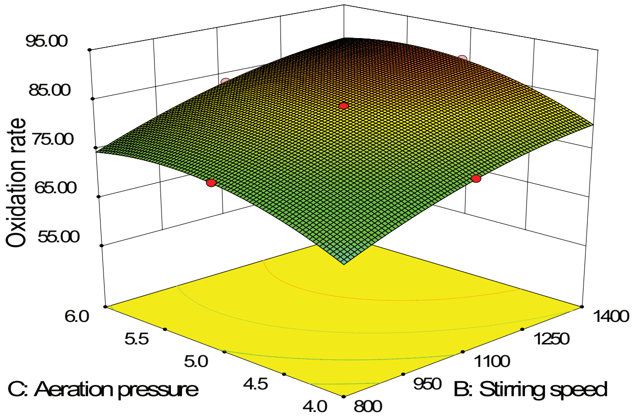

Fig. 5. Effect of stirring speed and aeration pressure on oxidation rate.

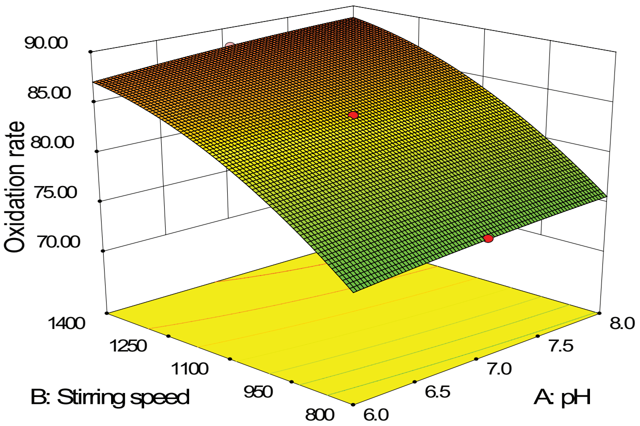

Fig. 4. Effect of pH and stirring speed on oxidation rate.

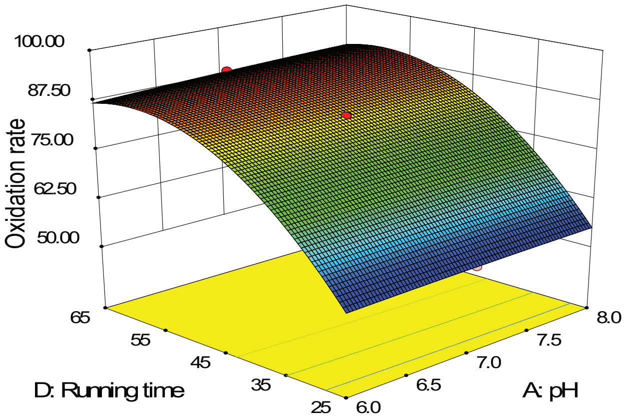

Fig. 6. Effect of running time and pH on oxidation rate.

Another important operating parameter in the aeration process is the stirring speed. As an example, Fig. 4 shows the effect of stirring speed and pH on the oxidation rate with aeration pressure of 5.0 bar and running time of 45 min. When the stirring speed increased, the oxidation rate increased significantly, but when the stirring speed reached 1350 rpm, the oxidation rate increased slightly. This means that an increase in the stirring speed contributes to improvement of the gas-liquid contact area and the reaction time; however, there is a limit to the improvement of the stirring speed of the oxidation of magnesium sulfite. Therefore, at a pH of 7.9, a stirring speed of 1386 rpm and an aeration pressure of 5.0 bar, the maximum oxidation rate was 88.7% for 45 min.

Fig. 5 shows the effect of aeration pressure and stirring speed on the oxidation rate, when pH is 7.0 and running time is 45 min. It can be observed that the oxidation rate increases strongly with increasing aeration pressure and stirring speed. However, when the aeration pressure reached 5.45 bar and the stirring speed reached 1350 rpm, the oxidation rate slightly increased. This is because oxygen mass transfer from the air to the liquid phase increases significantly with increasing aeration pressure and agitation speed, but an increase of stirring speed will result in the wastewater becoming more turbulent. Therefore, the maximum oxidation rate was 89.1% at 45 min with a pH of 7.0, a stirring speed of 1350 rpm, and aeration pressure of 5.45 bar.

The plots shown in Figs. 6–8 indicate the influence of the running time on the oxidation rate. From the figures, it can be found that the oxidation rate is increased with increasing running time at any value of pH, stirring speed and aeration pressure. The increasing of running time means more micro-bubbles are introduced to the reactor, which increase the oxidation rate of magnesium sulfite [4]. However, it is noticed that increasing running time beyond 55 min shows no significant effect on the oxidation rate. This shows that the oxidation reaction may reach equilibrium at the moment.

3.3. Optimization and validation of experimental conditions

The aeration parameters including pH of wastewater, aeration pressure, stirring speed and running time were optimized by RSM. In the optimization process, the pH value of the wastewater should be within the range of 6.5–9 according to the regulation conducted by IMO, the aeration pressure does not exceed the output pressure of the air compressor, and the stirring speed should be set under the rated speed of the motor.

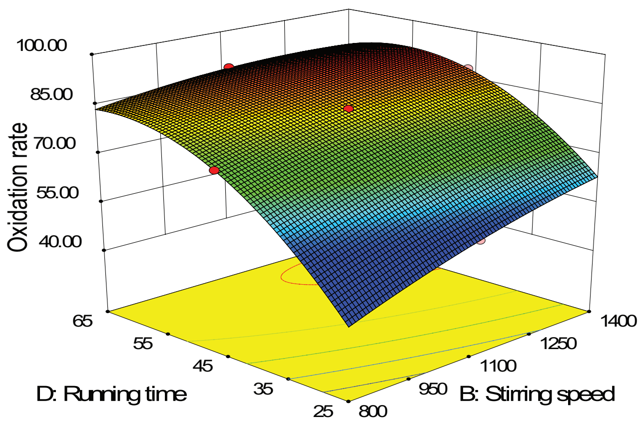

Fig. 7. Effect of running time and stirring speed on oxidation rate.

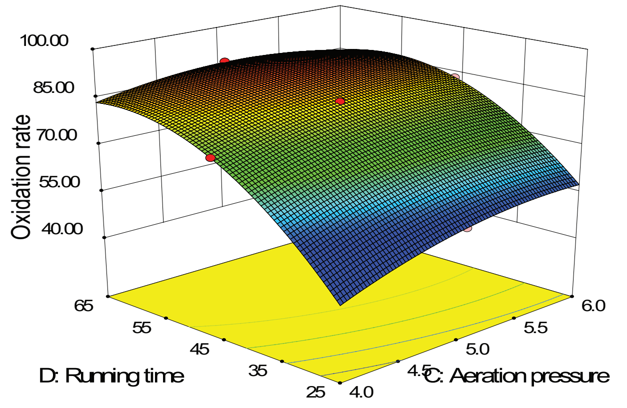

Fig. 8. Effect of running time and aeration pressure on oxidation rate.

According to the study performed by RSM under the model fitted by Eq. (2), the optimum parameters were pH 7.5, aeration pressure of 5.44 bar, stirring speed of 1384 rpm and running time of 54.5 min, and the maximum oxidation rate of magnesium sulfite was 92.3% at this time. In order to validate the optimization results, a specific batch run was carried out under the optimum conditions. The other experimental conditions were kept as the wastewater volume of 0.3 m^3 and the wastewater temperature of 55°C. In this process, the oxidation rate of magnesium sulfite was 92.1%. Thus, the model method has good prediction ability.

4. Conclusions

In this work, a novel micro-bubble aeration device using a porous tube in combination with an agitator stirring was proposed, and the key parameters of aeration process were optimized with response surface methodology (RSM). Based on the experimental results, a relationship between the oxidation rate and independent variables was obtained and expressed by the hierarchical second-order Eq. (2). The effect of experimental parameters on the oxidation rate of magnesium sulfite was established by the response surfaces of the developed model. As determined by RSM, the optimum parameters were pH 7.5, aeration pressure of 5.44 bar, stirring speed of 1384 rpm and running time of 54.5 min, and the maximum oxidation rate of magnesium sulfite was 92.3%. Meanwhile, the experimental result was 92.1%, which are consistent with the model predictions. In conclusion, this study provides an alternative to oxidize magnesium sulphite in desulphurized wastewater to magnesium sulphate.

Acknowledgements

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