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Reaction Kinetics for a Novel Flue Gas Cleaning Technology

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Abstract

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Disciplines

Biomechanical Engineering | Complex Fluids | Other Mechanical Engineering | Process Control and Systems

Comments

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Reaction Kinetics for a Novel Flue Gas Cleaning Technology

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This paper studies the kinetics of the reaction between NaClO_3 , FeSO_4 , and NaHSO_3 , which can potentially be used as an alternative to the conventional lime-limestone process for flue gas desulfurization. The key for the establishment of a kinetic model of the reaction is to find a way to determine concentrations of reactants or products during the reaction. The generation rate of Cl^- during the reaction was monitored using a Dionex Series 4000i ion chromatograph. Based on the changes of Cl^- concentrations at the designed initial reaction conditions, reaction orders for each reactant were derived. The reaction orders were determined to be 1.1 for NaClO_3 , 1.1 for FeSO_4 , and 1.4 for NaHSO_3 . The global rate coefficients of the reaction at temperatures ranging from 40 to 80 °C were determined. Furthermore, the preexponential factor and the activation energy in the empirical Arrhenius form of the reaction were derived from the relationship between temperature and its corresponding observed global rate coefficient.

Introduction

The harmful effects on the environment of SO_2 produced from fossil fuel consumption are becoming a problem worldwide. For a long time, treatment with lime or limestone as an absorbent was the major technique for SO_2 removal from flue gas. The disadvantages of this conventional technique include the discharge of a large amount of waste slurry containing calcium sulfate, which either needs to be stored or further treated, along with the generation of large amounts of the greenhouse gas carbon dioxide (CO_2) into the air. Furthermore, this technique has led to huge financial burdens for many coal-based power plants. Therefore, less environmentally intrusive and cost-effective flue gas cleaning technologies need to be developed (1–5). An important goal for new SO_2 removal technologies is the production of value-added products.

We have conducted research on the absorption of SO_2 , a coal combustion byproduct, with a solution of FeSO_4 , an

inexpensive and readily available byproduct of several industrial processes (3–5). This SO_2 removal method has several advantages: it has high SO_2 removal efficiency; it occurs at low temperature; it does not add harmful byproducts to the environment; and it produces polymeric ferric sulfate (PFS), an attractive coagulant for water and wastewater treatment (6, 7).

The production of PFS from SO_2 with FeSO_4 as an adsorption agent basically consists of three steps. The first step is to oxidize Fe^{2+} into Fe^{3+} under an acidic environment provided by bubbling SO_2 into the reaction system. Another step is to hydrolyze Fe^{3+} into $[\text{Fe}_x(\text{OH})_y]^{(3x-y)+}$ species. Finally, $[\text{Fe}_x(\text{OH})_y]^{(3x-y)+}$ and SO_4^{2-} will combine and form stable, polymeric ferric sulfate characterized by a high basicity.

The toxicity of PFS synthesized from SO_2 in a simulated flue gas has been evaluated (7). The experimental results have shown that water treated with PFS produced from simulated flue gas had no significant effect on body weight gain or the food and water intakes of tested rats and that no significant difference between the clinical chemistry profile of the PFS-treated rat group and that of control group was found (7). Actually, the SO_2 cleaning system studied in this research will be built downstream of the electrostatic precipitator (ESP), where most flue gas desulfurization (FGD) systems are presently located. In most coal-fired plants, ESPs have a particulate removal efficiency greater than 99.5% (8). Therefore, to affect the quality of polymeric ferric sulfate synthesized from flue gas, almost all fly ash containing trace contaminants is captured before reaching the reaction vessels. Also, most sulfate salts of trace contaminants are insoluble or only very slightly soluble in liquid PFS and can be filtered from PFS product. Furthermore, the PFS synthesized from flue gas is used primarily for wastewater treatment, in which the standards for coagulants are less stringent than those for treating drinking water. Therefore, PFS synthesized from flue gas has good prospects for use in wastewater treatment.

Despite the attractiveness of the integration of air pollution control with water treatment in this SO_2 removal technique (3–5), more research needs to be done before it is developed into a commercial process. For instance, the kinetics of the process, an essential element in optimizing reaction conditions, need to be investigated. The central reactions to this new sulfur removal technology are oxidation of HSO_3^- and Fe^{2+} by ClO_3^- . The oxidation process leads to production of $\text{Fe}_2(\text{SO}_4)_3$, which is followed by hydrolysis of Fe^{3+} to produce PFS. This paper investigates reaction kinetics between NaClO_3 , FeSO_4 , and NaHSO_3 .

It is noted that the dissolved O_2 can potentially oxidize Fe^{2+} and SO_2 during PFS production. However, it is well-known that NaClO_3 is much stronger than O_2 under acidic reaction conditions. Therefore, the effects of dissolved O_2 on the oxidation of Fe^{2+} and SO_2 as well as the resulting PFS production can be neglected.

Methods

Theory. SO_3^{2-} and HSO_3^- may simultaneously exist due to the presence of SO_2 in the reaction system. However, the oxidation of Fe^{2+} requires the presence of H^+ . Since SO_3^{2-} is unrelated to the oxidation of Fe^{2+} , only HSO_3^- can meet this requirement. Consequently, only the reaction between NaClO_3 , FeSO_4 , and NaHSO_3 needs to be considered for the production of PFS when the pH of the reaction system is less than 7, which can be realized by continuously bubbling SO_2 into the reactor.

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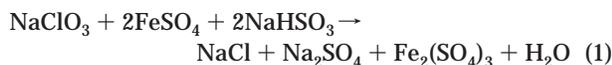
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The reaction between NaClO₃, FeSO₄, and NaHSO₃ can be expressed as follows:



Assuming that $c_{\text{NaClO}_3,0}$, $c_{\text{FeSO}_4,0}$, and $c_{\text{NaHSO}_3,0}$ represent the initial mole concentrations of reactants NaClO₃, FeSO₄, and NaHSO₃, respectively, initial concentration of NaCl is 0, and c_{Cl^-} is the concentration of NaCl or Cl⁻ generated during the reaction or the quantity of NaClO₃ or ClO₃⁻ consumed, then the concentrations of all the reactants at any time, t , should be $c_{\text{NaClO}_3,0} - c_{\text{Cl}^-}$, $c_{\text{FeSO}_4,0} - 2c_{\text{Cl}^-}$, and $c_{\text{NaHSO}_3,0} - 2c_{\text{Cl}^-}$, respectively, and the increasing rate of c_{Cl^-} can be expressed as follows

$$\frac{dc_{\text{Cl}^-}}{dt} = k(c_{\text{NaClO}_3,0} - c_{\text{Cl}^-})^{n_1}(c_{\text{FeSO}_4,0} - 2c_{\text{Cl}^-})^{n_2}(c_{\text{NaHSO}_3,0} - 2c_{\text{Cl}^-})^{n_3} \quad (2)$$

where k represents the global rate coefficient and exponents n_1 , n_2 , and n_3 are the orders with respect to reactants NaClO₃, FeSO₄, and NaHSO₃. Reaction orders n_1 , n_2 , and n_3 are not necessarily integers because reaction 1 is not an elementary reaction.

Special initial reactant concentrations need to be designed in order to derive reaction orders n_1 , n_2 , and n_3 . For example, when $c_{\text{NaClO}_3,0}$ and $c_{\text{FeSO}_4,0}$ are much larger than $c_{\text{NaHSO}_3,0}$, $c_{\text{NaClO}_3,0}$, and $c_{\text{FeSO}_4,0}$ at any time t of the reaction 1 can be considered as constants, or

$$c_{\text{NaClO}_3} = c_{\text{NaClO}_3,0} - c_{\text{Cl}^-} \approx c_{\text{NaClO}_3,0} \quad (3)$$

$$c_{\text{FeSO}_4} = c_{\text{FeSO}_4,0} - 2c_{\text{Cl}^-} \approx c_{\text{FeSO}_4,0} \quad (4)$$

and then eq 2 can be simplified as

$$\frac{dc_{\text{Cl}^-}}{dt} = k_3(c_{\text{NaHSO}_3,0} - 2c_{\text{Cl}^-})^{n_3} \quad (5)$$

where

$$k_3 = kC_{\text{NaClO}_3,0}^{n_1} \cdot C_{\text{FeSO}_4,0}^{n_2} \quad (6)$$

Taking the logarithm of eq 5 yields

$$\log \frac{dc_{\text{Cl}^-}}{dt} = n_3 \log(c_{\text{NaHSO}_3,0} - 2c_{\text{Cl}^-}) + \log k_3 \quad (7)$$

The c_{Cl^-} at any time, t , of the reaction was measured with a Dionex Series 4000i ion chromatograph, which leads to the establishment of the relationship of c_{Cl^-} vs time. Consequently, the reaction order with respect to NaHSO₃, n_3 , can be derived by curve fitting $\log dc_{\text{Cl}^-}/dt \sim \log(c_{\text{NaHSO}_3,0} - 2c_{\text{Cl}^-})$. It is noted that the value of dc_{Cl^-}/dt at time t was obtained from $c_{\text{Cl}^-} - t$ curve by using a tangential approach (8). Similarly, when $c_{\text{NaClO}_3,0}$ and $c_{\text{NaHSO}_3,0}$ are much larger than $c_{\text{FeSO}_4,0}$, n_2 can be obtained by curve fitting $\log dc_{\text{Cl}^-}/dt \sim \log(c_{\text{FeSO}_4,0} - 2c_{\text{Cl}^-})$, which is based on the relationship of $c_{\text{Cl}^-} \sim t$ attained through experimental data. In addition, when the initial concentrations of the reactants FeSO₄ and NaHSO₃, $c_{\text{FeSO}_4,0}$ and $c_{\text{NaHSO}_3,0}$, are much larger than $c_{\text{NaClO}_3,0}$, the reaction order with respect to NaClO₃, n_1 , can be inferred by curve fitting the experimental $c_{\text{Cl}^-} \sim t$ relationship and its corresponding $\log dc_{\text{Cl}^-}/dt \sim \log(c_{\text{NaClO}_3,0} - c_{\text{Cl}^-})$ plot.

Furthermore, the initial reactant concentrations with the relationship of $2c_{\text{NaClO}_3,0} = c_{\text{FeSO}_4,0} = c_{\text{NaHSO}_3,0}$ were designed to derive global rate coefficient k at a known temperature. Given initial concentrations of three reactants, eq 2 can be

reorganized as

$$\frac{dc_{\text{Cl}^-}}{dt} = k(c_{\text{NaClO}_3,0} - c_{\text{Cl}^-})^{n_1} \cdot 2^{n_2} \cdot (c_{\text{NaClO}_3,0} - c_{\text{Cl}^-})^{n_2} \cdot 2^{n_3} (c_{\text{NaClO}_3,0} - c_{\text{Cl}^-})^{n_3} = 2^{n_2+n_3} \cdot k \cdot (c_{\text{NaClO}_3,0} - c_{\text{Cl}^-})^{n_1+n_2+n_3} \quad (8)$$

Integration of eq 8 with the boundary condition ($c_{\text{Cl}^-,0} = 0$) results in

$$\int_0^{c_{\text{Cl}^-}} \frac{dZ}{(c_{\text{NaClO}_3,0} - Z)^{n_1+n_2+n_3}} = \int_0^t 2^{n_2+n_3} \cdot k dt \quad (9)$$

$$(c_{\text{NaClO}_3,0} - c_{\text{Cl}^-})^{1-(n_1+n_2+n_3)} - c_{\text{NaClO}_3,0}^{1-(n_1+n_2+n_3)} = 2^{n_1+n_3} \cdot k \cdot (n_1 + n_2 + n_3 - 1)t \quad (10)$$

or

$$k = \frac{(c_{\text{NaClO}_3,0} - c_{\text{Cl}^-})^{1-(n_1+n_2+n_3)} - c_{\text{NaClO}_3,0}^{1-(n_1+n_2+n_3)}}{2^{n_2+n_3}(n_1 + n_2 + n_3 - 1)t} \quad (11)$$

Therefore, the global rate coefficient k at a specified temperature can be determined using eq 11 given that n_1 , n_2 , and n_3 have been obtained, and c_{Cl^-} at time t is measured.

The temperature range, 40–80 °C, important for the FeSO₄-based sulfur removal approach, is reasonably narrow, consequently variations of the preexponential factor and the activation energy of reaction 1 with temperature can be ignored (9, 10). Therefore, the relationship between the global rate constant (k) and reaction temperature (T) can be expressed in the empirical Arrhenius form below

$$k = A \exp\left(-\frac{E}{RT}\right) \quad (12)$$

where A is the preexponential factor, E is the activation energy of reaction 1, and R is the ideal gas constant. The plot of $\log k$ versus $1/T$ can be used to extract values for A and E in Arrhenius form (eq 12), since the slope and interception of such a plot are $-E/2.303R$ and $\log A$, respectively.

Experimental Section

Apparatus. The reactions between NaClO₃, FeSO₄, and NaHSO₃ were conducted in a 1.0 L double-wall glass reactor with three inlets on the top. A stir set through the central inlet of the reactor was used to mix the reactants during the reaction. A thermometer passed through another inlet to measure the temperature in the reactor. The prepared reactant solutions were added through the additional inlet. A DL-501 thermostat was used to control the temperature in the reactor by circulating water around the reactor. Water and solutions of reactants NaClO₃, FeSO₄, and NaHSO₃ were prepared in advance and stored in separate vessels. All the chemicals used for the reactions and analysis of this study were analytical grade.

Measurement of Cl⁻ Concentration. The concentrations of Cl⁻ during the reaction at different times were measured for monitoring the extent of reaction. To this end, the reactions in the reactor need to be stopped immediately after a desired period of reaction time. Sodium hydroxide solution (0.1 M) was prepared and used for that purpose, since the reaction between NaClO₃, FeSO₄, and NaHSO₃ stops when the pH of reaction 1 is higher than 7 (11). The pH was monitored using a Fisher Scientific model 310 pH meter. Change of c_{Cl^-} was monitored for 240 s in each test. Rates for most nonzero and positive order reactions are higher at early times than a later period; accordingly, a shorter sampling interval, 5 s, was chosen for the first 30 s of c_{Cl^-} monitoring time. This increased the density of the data points in the

TABLE 1. Cl^- Concentrations (c_{Cl^-}) at 150 s and Global Rate Coefficients Calculated for Reaction 1 under the Different Temperature ($C_{\text{NaClO}_3,0} = 14.75 \text{ mM}$, $C_{\text{FeSO}_4,0} = 29.5 \text{ mM}$, and $C_{\text{NaHSO}_3,0} = 29.5 \text{ mM}$)

temperature ($^{\circ}\text{C}$)	40	50	60	70	80
c_{Cl^-} (mM)	4.73	7.04	9.02	10.5	11.6
k ($\text{mol}^{-2.6}\cdot\text{dm}^{2.6}\cdot\text{s}^{-1}$)	17.3	43.3	102.3	230.0	494.0

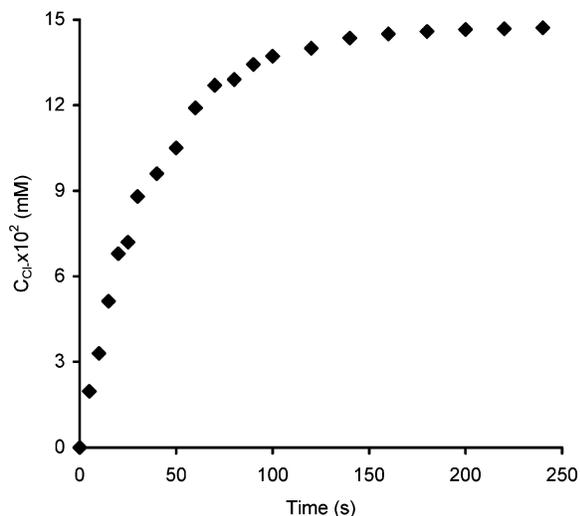


FIGURE 1. Change of Cl^- concentration with time ($C_{\text{NaClO}_3,0} = 0.1475 \text{ mM}$, $C_{\text{FeSO}_4,0} = 29.5 \text{ mM}$, and $C_{\text{NaHSO}_3,0} = 29.5 \text{ mM}$, $T = 60^{\circ}\text{C}$).

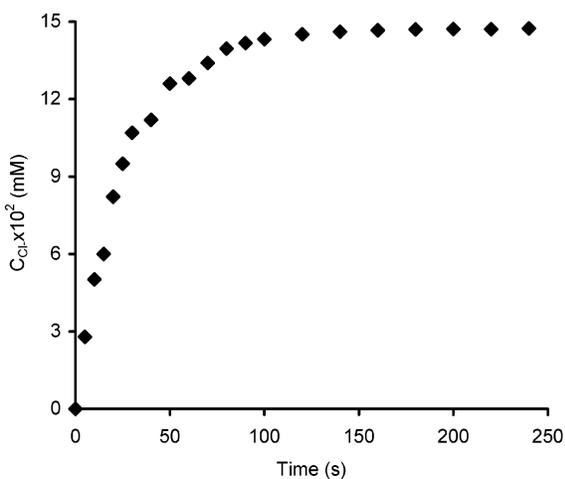


FIGURE 2. Change of Cl^- concentration with time ($C_{\text{NaClO}_3,0} = 14.75 \text{ mM}$, $C_{\text{FeSO}_4,0} = 0.295 \text{ mM}$, and $C_{\text{NaHSO}_3,0} = 29.5 \text{ mM}$, $T = 60^{\circ}\text{C}$).

initial period of $c_{\text{Cl}^-} \sim t$ plots, where the $c_{\text{Cl}^-} \sim t$ curves typically appear steep, and thus improved the accuracy of curve fitted $c_{\text{Cl}^-} \sim t$ relationship. Ten seconds was used as the time interval for the remaining sampling period. Samples were taken using pipets. The value of c_{Cl^-} was determined using a Dionex Series 4000i ion chromatograph. The instrument was calibrated with a set of NaCl standard solutions at concentrations of 0, 50, 100, 150, and 200 ppm.

Preparation of the Solution for the Determination of Reaction Orders. All the tests designed for the determination of reaction orders were conducted at 60°C . As previously mentioned, the initial concentration of the reactant for which the reaction order is to be determined should be much smaller than other reactants. Accordingly, 0.4425 mM NaClO_3 , 88.5 mM FeSO_4 , and 88.5 mM NaHSO_3 solutions were prepared and used for the determination of the reaction order of NaClO_3 , 44.25 mM NaClO_3 , 0.885 mM FeSO_4 , and 88.5 mM

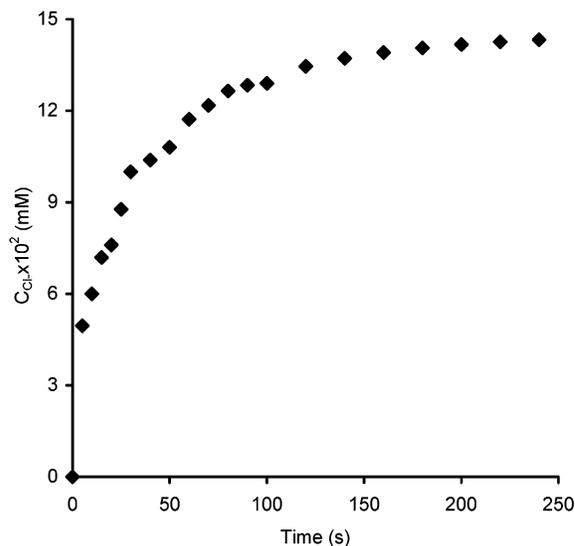


FIGURE 3. Change of Cl^- concentration with time ($C_{\text{NaClO}_3,0} = 14.75 \text{ mM}$, $C_{\text{FeSO}_4,0} = 29.5 \text{ mM}$, and $C_{\text{NaHSO}_3,0} = 0.295 \text{ mM}$, $T = 60^{\circ}\text{C}$).

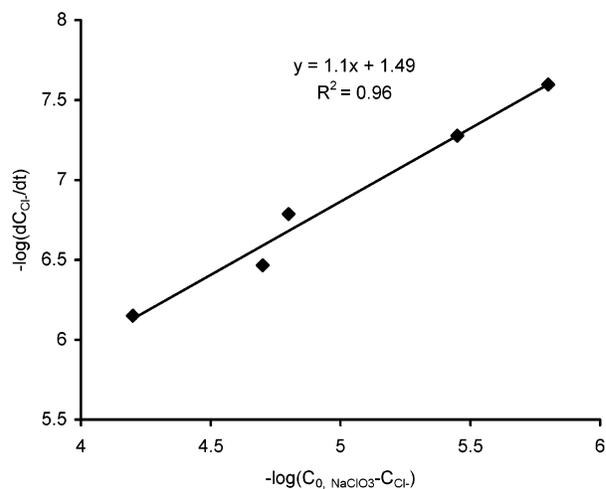


FIGURE 4. Determination of reaction order of NaClO_3 ($C_{\text{NaClO}_3,0} = 0.1475 \text{ mM}$, $C_{\text{FeSO}_4,0} = 29.5 \text{ mM}$, and $C_{\text{NaHSO}_3,0} = 29.5 \text{ mM}$, $T = 60^{\circ}\text{C}$).

NaHSO_3 solutions for the determination of reaction order of FeSO_4 , and 44.25 mM NaClO_3 , 88.5 mM FeSO_4 , and 0.885 mM NaHSO_3 solutions for the determination of reaction order of NaHSO_3 . The same volume (150 mL) of each prepared reactant solution (i.e., NaClO_3 , FeSO_4 , and NaHSO_3) was used in each reaction. Therefore, the real initial concentration of each reactant in each reaction was one-third of that prepared before mixing.

Preparation of Solutions for the Determination of Arrhenius Form. Rate constants were calculated at different temperatures in order to determine the Arrhenius equation for reaction 1. To obtain the data needed for these purposes, 44.25 mM NaClO_3 , 88.5 mM FeSO_4 , and 88.5 mM NaHSO_3 solutions ($2C_{\text{NaClO}_3,0} = C_{\text{FeSO}_4,0} = C_{\text{NaHSO}_3,0}$) were prepared and used for reactions at 40°C , 50°C , 60°C , 70°C , and 80°C . The concentrations of Cl^- generated, c_{Cl^-} , were measured 180 s after the reactions started. Rate constants at different temperatures were calculated using eq 11.

Results and Discussions

$c_{\text{Cl}^-} \sim t$ Relationships. The results of the tests on the change of c_{Cl^-} with time under different combinations of initial reactant concentrations are shown in Figures 1–3. The trends of the curves in Figures 1–3 indicate that none of the reaction orders for NaClO_3 , FeSO_4 and NaHSO_3 were zero, since none

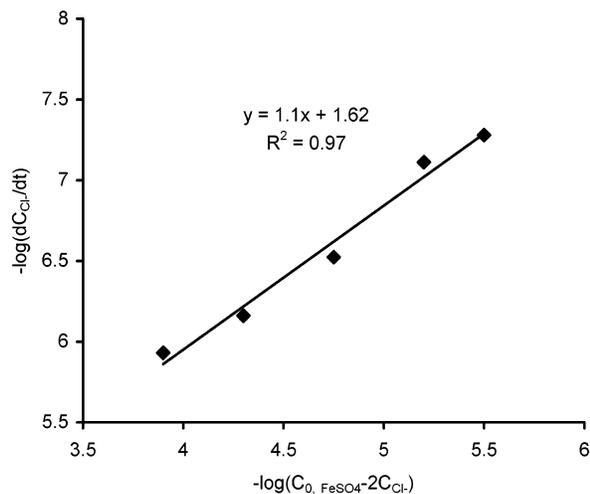


FIGURE 5. Determination of reaction order of FeSO_4 ($C_{\text{NaClO}_3,0} = 14.75$ mM, $C_{\text{FeSO}_4,0} = 0.295$ mM, and $C_{\text{NaHSO}_3,0} = 29.5$ mM, $T = 60$ °C).

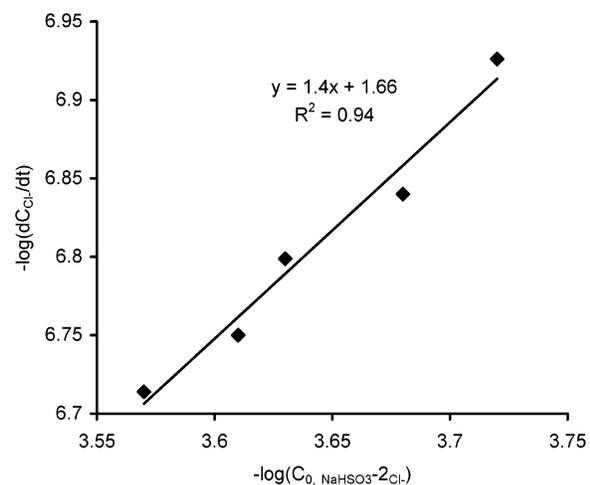


FIGURE 6. Determination of reaction order of NaHSO_3 ($C_{\text{NaClO}_3,0} = 14.75$ mM, $C_{\text{FeSO}_4,0} = 29.5$ mM, and $C_{\text{NaHSO}_3,0} = 0.295$ mM, $T = 60$ °C).

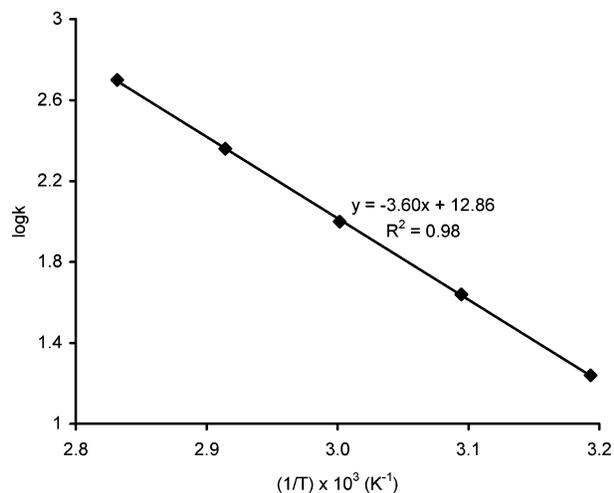


FIGURE 7. Determinations of activation energy and the preexponential factor in Arrhenius form ($C_{\text{NaClO}_3,0} = 14.75$ mM, $C_{\text{FeSO}_4,0} = 29.5$ mM, $C_{\text{NaHSO}_3,0} = 29.5$ mM, and $t = 150$ s).

of those plots is linear. All three of the curves, shown in Figures 1–3, rise steeply at the beginning of the reaction and become flat after about 100 s; however, each has a different initial rate (within the first 5 s). From this, one can determine

that the reaction rates of NaClO_3 , FeSO_4 , and NaHSO_3 are closely correlated with initial reactant concentrations.

Derivation of Reaction Orders. The data points in Figures 4–6 were derived numerically from Figures 1–3 using a tangential method (9). The resulting linear plots, Figures 4–6, each have a regression coefficient higher than 0.95. The slopes of the lines are the reaction orders of reactants NaClO_3 , FeSO_4 , and NaHSO_3 and are approximately 1.1, 1.1, and 1.4, respectively. Therefore, eq 2 can be expressed as follows:

$$\frac{dc_{\text{Cl}^-}}{dt} = k(c_{\text{NaClO}_3,0} - c_{\text{Cl}^-})^{1.1}(c_{\text{FeSO}_4,0} - 2c_{\text{Cl}^-})^{1.1}(c_{\text{NaHSO}_3,0} - 2c_{\text{Cl}^-})^{1.4} \quad (13)$$

Establishment of Arrhenius Form. The average concentrations of Cl^- in the reaction system at 150 s under temperature 40 °C, 50 °C, 60 °C, 70 °C, and 80 °C are shown in Table 1. Global rate coefficient k at 40 °C, 50 °C, 60 °C, 70 °C, and 80 °C, calculated with the c_{Cl^-} values in Table 1 and eq 11, are also listed in Table 1. From these global rate coefficients, it is evident that rate increases with temperature. The global rate coefficient increases by approximately 100% when temperature increases 10 °C in the temperature range tested, which is consistent with van't Hoff rule (12).

Based on the results in Table 1, the relationship between $\log k$ and $1/T$ was determined as demonstrated in Figure 7. A regression coefficient of 0.98 in Figure 7 indicates that a good linear relationship between $\log k$ and $1/T$ exists. Therefore, the Arrhenius form of reaction 1 can be expressed as

$$k = 7.2 \times 10^{12} \cdot e^{-\frac{8291}{T}} \quad (\text{mol}^{-2.6} \cdot \text{dm}^{2.6} \cdot \text{s}^{-1}) \quad (14)$$

In conclusion, the reaction between NaClO_3 , FeSO_4 , and NaHSO_3 , potentially used for sulfur removal from flue gas, was characterized. A kinetic model, including reaction orders and activation energy, has been established by tracking the change of the concentration of the chloride ion with time during the reactions with different initial reactant concentrations. This study is essential in understanding the underlying chemical mechanism of the new sulfur removal technique, which can provide the basis for the design and optimization of the process.

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