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Removal of Dilute Nitric Oxide using Cobalt Diethylenetriamine Solution under Aerobic Condition

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Abstract: A novel process has been developed to remove NO from flue gas using a basic solution of cobalt(II) diethylenetriamine. The cobalt(II) diethylenetriamine cation can not only bind NO but also activate oxygen in aqueous solution. When only cobalt(II) diethylenetriamine solution was used, NO was oxidized to soluble nitrite and nitrate by active oxygen. So O₂ in the gas phase is favorable for the absorption of NO as O₂ concentration is less than 6%. When hydrazine hydrate were added to the solution, NO was reduced to N₂ and almost no nitrate was found in the spent scrubbing liquor. O₂ reacted with active Co^{II} to inactive Co^{III}. Thus the removal efficiency of NO decreased with increasing O₂ concentration. The effects of liquid-to-gas ratio, cobalt(II) diethylenetriamine, (NH₂)₂·H₂O concentration, and temperature on NO removal efficiency were also examined under aerobic condition. Results showed that cobalt(II) diethylenetriamine was a promising metal chelate for NO removal.

Keywords: Absorption, cobalts, diethylenetriamines, hydrazine hydrates, Nitric oxides

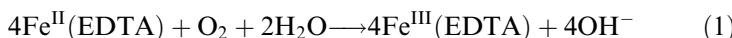
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INTRODUCTION

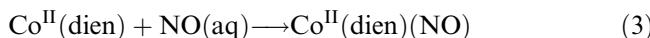
The removal of nitrogen oxides (NO_x) from flue gases has received considerable attention over the years because of its various deleterious effects to all forms of lives. Among the various physico-chemical wet and dry methods (1–6), wet method is considered to be the simplest method for gas scrubbing with very high removal efficiency. However, nitric oxide (NO), the main component of NO_x (7), has a very low solubility in aqueous solutions, which leads to difficulty when attempting to remove NO by conventional methods (8). It is shown that metal chelates can bind NO to form metal nitrosyl chelates. Therefore, the use of metal chelate additives in wet flue gas denitrification systems has been widely reported (9–15).

Besides NO_x , various other components are also present in flue gas, which can interfere with the metal chelate absorption process and give rise to undesired side reactions. One of the major components is molecular oxygen, which is typically present in amounts between 2 and 10 vol%. It has been reported that oxygen can react with the iron chelate to form Fe^{III} (EDTA) (eq. (1)) which is not able to bind NO (16–20).



To achieve high NO removal efficiencies in the process, the oxidation reaction should be suppressed as much as possible. However, using cobalt chelate solution as the absorbent, oxygen in the gas phase is favorable to the absorption of nitric oxide from flue gases (21–24). The dissolved oxygen can oxidize nitric oxide to soluble nitric dioxide quickly. So the presence of oxygen in flue gas affects NO absorption process complicatedly.

In this paper, the authors describe the use of $\text{Co}^{\text{II}}(\text{dien})$ (dien stands for diethylenetriamine) for the removal of NO under aerobic condition. Dien is another promising chelating agent to Co^{II} for NO removal. When NO containing gas was flowed through the aqueous solution of $\text{Co}^{\text{II}}(\text{dien})$, the nitric oxide reacted with the reagent and was removed:



The main objective is to investigate the effect of oxygen on NO removal by $\text{Co}^{\text{II}}(\text{dien})$ with/without hydrazine hydrate regeneration and to determine the parameters influencing the process under aerobic condition.

EXPERIMENTAL

Materials

Cobalt (II) chloride hexahydrate ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$, AR) and diethylenetriamine ($\text{C}_4\text{H}_{13}\text{N}_3$, CP) were purchased from Sinopharm Chemical Reagent Co., Ltd, China. 80% Hydrazine hydrate ($(\text{NH}_2)_2 \cdot \text{H}_2\text{O}$, AR) was the product of the Wenli Fine Chemical Plant. 1000 ppm NO in N_2 was obtained from Oxygen Co., Ltd. of WISCO, China. Reverse osmosis water was applied to prepare the cobalt chelate solution.

Experimental Setup

The schematic diagram of the experimental set-up for the removal of NO is shown in Fig. 1. A glass column (20 mm in diameter, 1000 mm in height) and a holding tank with a working volume of 400 ml were used for NO removal. The column was packed with glass rings with a nominal size of 4 mm. The absorber temperature was controlled with the use of a jacket, through which water from a thermostatic bath was circulated.

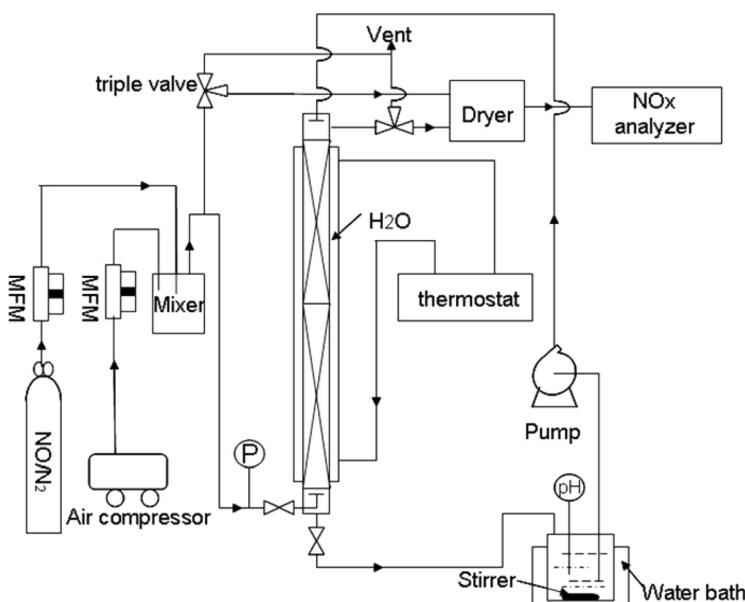


Figure 1. A schematic diagram of the experimental apparatus.

Flow rates of air and 1000 ppm NO were controlled by two mass flow meters (MFM). The simulated gas continuously flowed through the system. The column was operated batchwise with respect to the absorbing liquid which was prepared by adding certain proportions of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ and dien to distilled water. The liquid was collected under the column in a holding tank from where it was pumped to the top of the column. The regeneration of the absorption solution was carried out under a stirring speed of 250 rpm. A valve at the liquid outlet from the column assured that only liquid and no gas left the column through the liquid outlet.

Analytical Methods

The concentration of NO was measured by Delta2000-CD-IV flue gas analyzer (York Instrument Ltd.). In order to protect the flue gas analyzer, the simulated gas must be dried before entering into the analyzer. The incoming NO concentration was measured just before the gas inlet and the outlet concentration was measured just after the gas outlet. Three-way valves regulate gas stream which should be analyzed. Liquid samples were taken from the holding tank. The ions were analyzed both quantitatively and qualitatively on an ion chromatograph (Dionex ICS 2500).

Data Analysis

The removal efficiency of NO could be obtained according to eq. 4.

$$E_f(\text{NO}) = (C_{\text{NO}(i)} - C_{\text{NO}(o)})/C_{\text{NO}(i)} \times 100\% \quad (4)$$

Where $E_f(\text{NO})$ is the efficiency of nitric oxide removal, C_{NO} is the concentration of the nitric oxide and the subscripts (i) and (o) represent the inlet and the outlet conditions, respectively.

RESULTS AND DISCUSSION

Effect of Inlet Oxygen Concentration on NO Removal Efficiency without Hydrazine Hydrate Reduction

The effect of varying inlet O_2 concentrations on NO removal efficiency from simulated flue gas is shown in Fig. 2. Under aerobic condition,

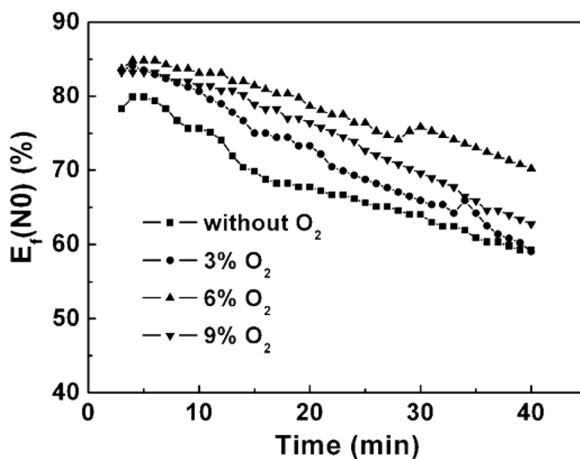
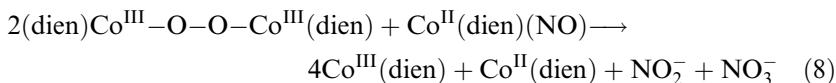
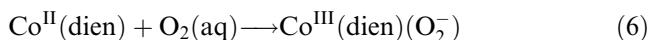


Figure 2. Effect of inlet oxygen concentration on NO removal efficiency without hydrazine hydrate reduction. (0.01 mol/L Co^{II}(dien); liquid-to-gas ratio = 66.5 L/m³; 50°C).

the dissolved oxygen (Eq. (5)), in equilibrium with oxygen in the feed gas, activated by Co^{II}(dien) solutions (eqs. (6) and (7)), oxidizes nitric oxide to soluble nitrite NO_2^- and nitrate NO_3^- quickly (Eq. (8)). Therefore, NO removal efficiency increases with O₂ concentration when O₂ concentration is varied from 0 to 6%.



Ion chromatographic analyses of the scrubbing liquor depicted in Fig. 3 also demonstrate that the NO absorbed has been converted to a mixture of nitrite and nitrate, which leads to the decrease of pH (from 7.91 to 7.20). Due to the reduction of nitrite in alkaline solution, more nitrate (11.485 mg/L) than nitrite (1.575 mg/L) is found in the spent scrubbing liquor. However, Co^{II}(dien) in the solution was readily

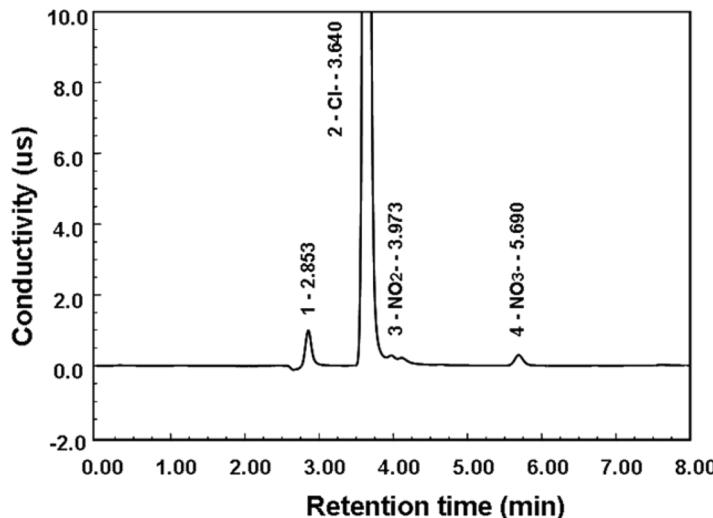


Figure 3. Ion Chromatograms of scrubbing liquor from the NO removal experiment with 0.01 mol/L Co^{II} (dien), 6% O_2 .

oxidized to Co^{III} (dien) under the condition of a high level of oxygen (Eq. (9)).



The higher the amount of O_2 in the flue gas, the faster the reaction of $\text{Co}^{\text{II}}(\text{dien})$ with O_2 . Thus, the concentration of active $\text{Co}^{\text{II}}(\text{dien})$ in the solution decreases at the beginning of absorption as a result of too much oxygen presence. Subsequently, NO removal efficiency decreases as O_2 concentration increases further above 6%.

Effects of Hydrazine Hydrate on NO Removal Efficiency

The effect of hydrazine hydrate on NO removal by $\text{Co}^{\text{II}}(\text{dien})$ solution was also performed for comparative purposes. The results are depicted in Fig. 4. When only $\text{Co}^{\text{II}}(\text{dien})$ solution is used to absorb NO, $\text{Co}^{\text{III}}(\text{dien})$ in the solution cannot be reduced to regenerate $\text{Co}^{\text{II}}(\text{dien})$. Thus, the concentration of $\text{Co}^{\text{II}}(\text{dien})$ decreases quickly, resulting in a decrease in NO removal efficiency. After about 45 min of absorption, the NO removal efficiency was about 58% with inlet O_2 concentration

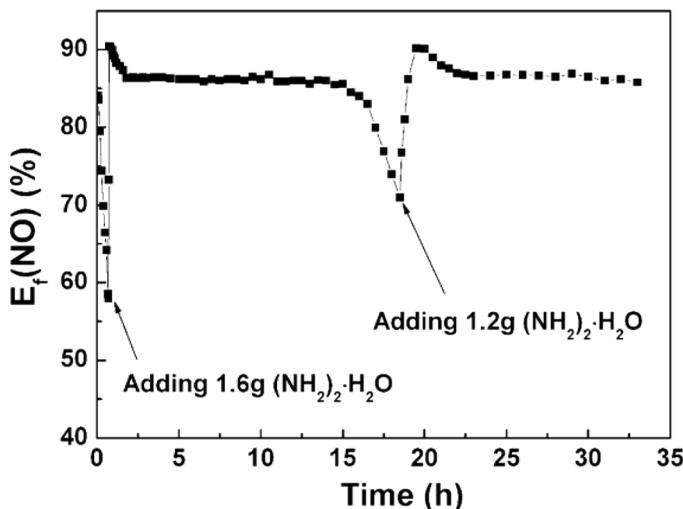


Figure 4. Effects of hydrazine hydrate on NO Removal efficiency. (0.01 mol/L Co^{II}(dien); liquid-to-gas ratio = 66.5 L/m³; 50°C; 3% O₂).

of 6%. When 1.6 g and 1.2 g hydrazine hydrate were added into Co^{II}(dien) scrubbing solution, the NO removal efficiency increases and stabilizes at about 86%. This trend may be attributed to the reductions of Co^{III}(dien) and Co^{II}(dien)NO (eqs. (10) and (11)). The experimental results show that the regeneration method works effectively.

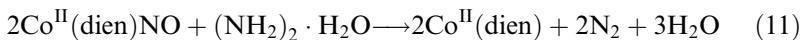


Figure 5 reveals that there is almost no nitrate existing in the scrubbing solution. The pH value of the scrubbing solution change little, only from 9.40 to 9.28. These results indicate that no secondary pollution is generated by hydrazine hydrate reduction.

Effect of Liquid-To-Gas Ratio on NO Removal Efficiency

Figure 6 presents the removal efficiencies of NO by Co^{II}(dien) solution at various liquid-to-gas ratios. The results demonstrate that NO removal efficiency increases with reaction time and reaches steady-state values

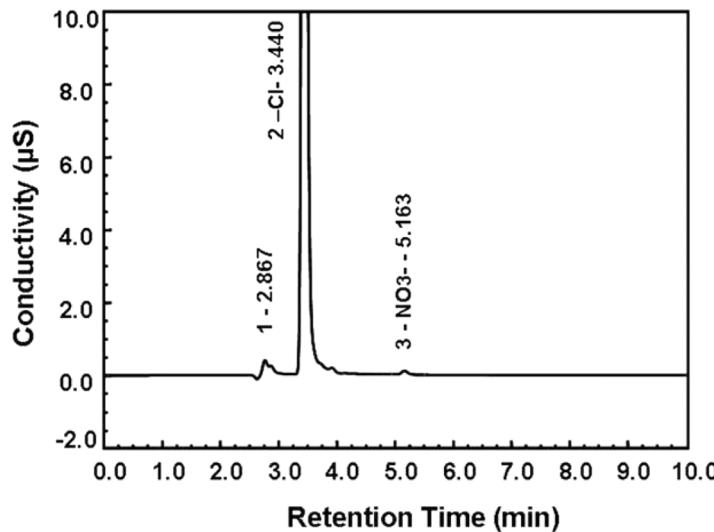


Figure 5. Ion chromatographic of scrubbing liquor from the NO removal experiment with hydrazine hydrate reduction.

of 86.7%, 92.8%, 95.6%, and 96.8% for liquid-to-gas ratios of 10.6, 23.3, 44.6, and 66.5 L/m^3 , respectively. Namely, the removal efficiency of NO increases with the liquid-to-gas ratio. This result is due to higher

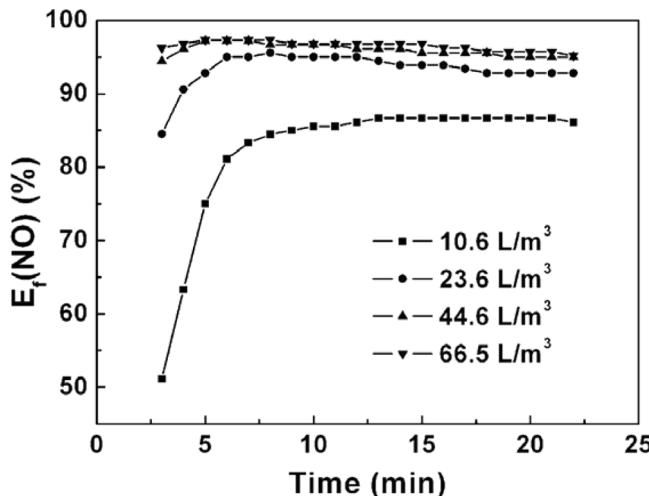


Figure 6. Effect of liquid-to-gas ratio on NO removal efficiency. (0.01 mol/L Co^{II}(dien); 0.075 mol/L $(\text{NH}_2)_2 \cdot \text{H}_2\text{O}$; 50°C; 3% O₂).

L/G ratios increasing the gas-liquid contacting surface area and mass transfer. It can be seen that the NO removal efficiency is satisfactory at a liquid-to-gas ratio of 44.6 L/m^3 . Therefore, the authors lowered the liquid-to-gas ratio to 23.3 L/m^3 in the following experiments.

Effect of the Initial $\text{Co}^{\text{II}}(\text{dien})$ Concentration on NO Removal Efficiency

The NO removal efficiency affected by varying $\text{Co}^{\text{II}}(\text{dien})$ concentrations is shown in Figure 7. It is clear that the removal efficiency of NO increases with the increasing $\text{Co}^{\text{II}}(\text{dien})$ concentration since the absorption capacity of NO is dependent upon the concentration of $\text{Co}^{\text{II}}(\text{dien})$. For instance, when 0.0025 mol/L $\text{Co}^{\text{II}}(\text{dien})$ is present in the beginning, just 68.6% NO was removed, while for 0.01 mol/L $\text{Co}^{\text{II}}(\text{dien})$, 95% NO was removed. It also can be seen that when $\text{Co}^{\text{II}}(\text{dien})$ concentration is very low, the NO removal efficiency is increased rapidly by increasing $\text{Co}^{\text{II}}(\text{dien})$ concentration. However, when $\text{Co}^{\text{II}}(\text{dien})$ concentration increases further above 0.01 mol/L , the NO removal efficiency increases little, which can be concluded that the NO absorption into the $\text{Co}^{\text{II}}(\text{dien})$ solution changes from two-film controlling to gas-film controlling. So it is unnecessary to increase $\text{Co}^{\text{II}}(\text{dien})$ concentration further above its critical concentration.

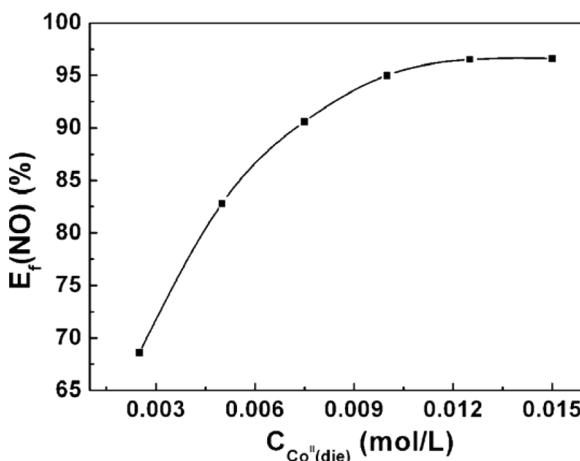


Figure 7. Effect of the initial $\text{Co}^{\text{II}}(\text{dien})$ concentration on NO removal efficiency. ($0.075 \text{ mol/L} (\text{NH}_2)_2 \cdot \text{H}_2\text{O}$; liquid-to-gas ratio = 23.3 L/m^3 ; 50°C ; 3% O_2 , 30-min reaction time).

Effect of $(\text{NH}_2)_2 \cdot \text{H}_2\text{O}$ Concentration on NO Removal Efficiency

The experimental results for various $(\text{NH}_2)_2 \cdot \text{H}_2\text{O}$ concentrations are shown in Fig. 8. It can be seen that the NO removal efficiency increased with the $(\text{NH}_2)_2 \cdot \text{H}_2\text{O}$ concentration increased. When the $(\text{NH}_2)_2 \cdot \text{H}_2\text{O}$ concentration was over 0.064 mol/L, a dynamic equilibrium could be realized between the regeneration and consumption of $\text{Co}^{\text{II}}(\text{dien})$ and the NO removal efficiency could be kept basically constant.

Effect of Temperature on NO Absorption Rate

A group of experiments was performed to investigate the effect of temperature (20–70°C) on removal efficiency of NO. The NO removal efficiencies for a reaction of 30 min are given in Fig. 9. It can be seen that the NO removal efficiency increases with temperature when the temperature is below 50°C. However, it decreases as the temperature increases further above 60°C. As the temperature of the solutions changes from 50 to 60°C, the NO removal efficiency changes little and remains at about 86%.

Dynamically, the reduction rates of $\text{Co}^{\text{III}}(\text{dien})$ and $\text{Co}^{\text{II}}(\text{dien})\text{NO}$ increase with temperature. Thus, the absorption of NO increases with temperature. On the other hand, a negative influence of raising the temperature on NO absorption performance should be mentioned.

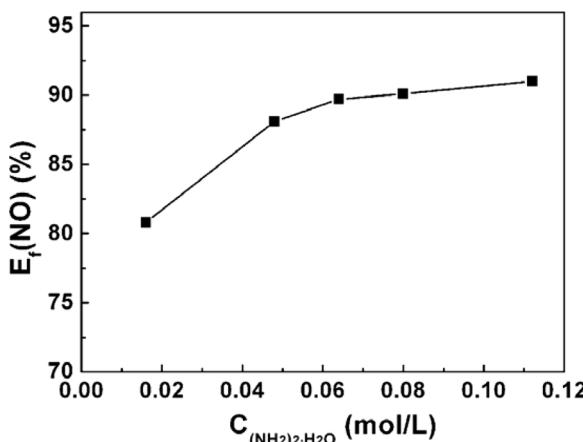


Figure 8. Effect of $(\text{NH}_2)_2 \cdot \text{H}_2\text{O}$ concentration on NO removal efficiency. (0.0075 mol/L $\text{Co}^{\text{II}}(\text{dien})$; liquid-to-gas ratio = 23.3 L/m³; 50°C; 3% O₂, 30-min reaction time).

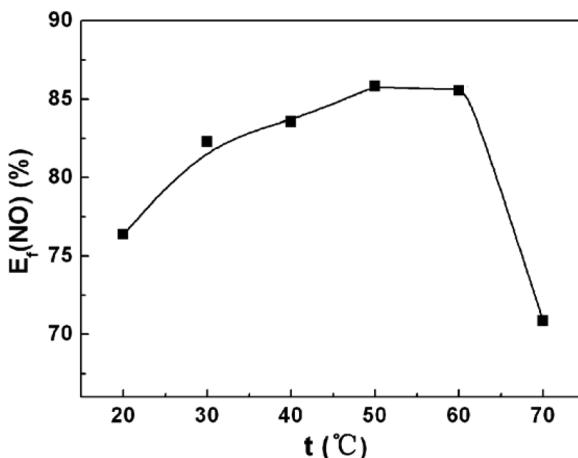


Figure 9. Effect of temperature on NO absorption rate. (0.0075 mol/L $\text{Co}^{\text{II}}(\text{dien})$; 0.064 mol/L $(\text{NH}_2)_2 \cdot \text{H}_2\text{O}$; liquid-to-gas ratio = 23.3 L/m³, 3% O_2 , 30-min reaction time).

Namely, the physical solubility of NO in aqueous solution decreases as the temperature is increased. When the temperature is below 50°C, the NO removal is governed mainly by the reduction step, so the NO removal efficiency increases with rising temperature. After the temperature is above 60°C, the physical solubility of NO controls mainly the NO removal. Therefore, the higher the temperature, the lower the NO removal efficiency. So, there is an optimal temperature range for the absorption of NO by $\text{Co}^{\text{II}}(\text{dien})-(\text{NH}_2)_2 \cdot \text{H}_2\text{O}$ solutions.

Effect of Inlet Oxygen Concentration on NO Removal Efficiency with Hydrazine Hydrate Reduction

A group of experiments was performed to investigate the effect of oxygen concentration on NO removal efficiency with $(\text{NH}_2)_2 \cdot \text{H}_2\text{O}$ reduction. The results are showed in Fig. 10. As stated earlier, the dissolved oxygen, activated by $\text{Co}^{\text{II}}(\text{dien})$ solutions, can oxidize nitric oxide to soluble nitrite and nitrate. However, hydrazine hydrate, prior to nitric oxide, present in solution will first react with active oxygen (Eq. (12)). So just a few nitric oxides can be oxidized by active oxygen. Moreover, although hydrazine hydrate can reduce $\text{Co}^{\text{III}}(\text{dien})$ and $\text{Co}^{\text{II}}(\text{dien})(\text{NO})$ to $\text{Co}^{\text{II}}(\text{dien})$, the presence of oxygen can also oxidize active Co^{III} to inactive Co^{III} as eq. (9). The higher the amount of O_2 , the faster the reaction of oxidation. Thus, the NO removal efficiency decreases with

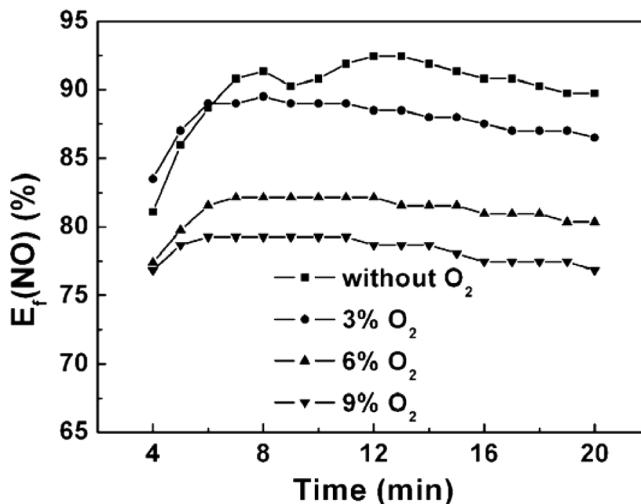
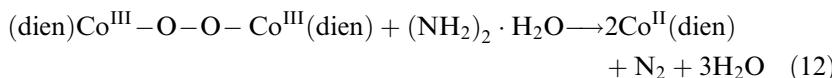


Figure 10. Effect of inlet oxygen concentration on NO removal efficiency with hydrazine hydrate reduction. (0.0075 mol/L Co^{II}(dien); 0.064 mol/L (NH₂)₂ · H₂O; liquid-to-gas ratio = 23.3 L/m³; 50°C).

increasing O₂ concentration.



CONCLUSION

The use of Co^{II}(dien) chelate for absorption with hydrazine hydrate regeneration to control NO emissions has been demonstrated in this paper. In our regenerative system, Co^{II}(dien) chelate serves to absorb NO while hydrazine hydrate keep the NO removal efficiency at a higher constant value than that would exist without it. Though molecular oxygen in flue gas activated by Co^{II}(dien) solutions can oxidize nitric oxide to soluble nitrite and nitrate, its presence also, adversely, oxidizes Co^{II} rapidly. Without hydrazine hydrate regeneration, NO removal efficiency increases with O₂ concentration when O₂ concentration is varied from 0 to 6%. But the removal efficiency decreases as O₂ concentration is higher. Ion chromatographic alyses of the scrubbing liquor demonstrate that the NO absorbed is converted to a mixture of nitrite and nitrate. When with hydrazine hydrate regeneration, NO removal efficiency decreases with increasing O₂ concentration. Almost no secondary pollution is found

by ion chromatographic analysis. The authors have also found that the NO removal efficiency increases with increasing $\text{Co}^{\text{II}}(\text{dien})$ concentration. When $\text{Co}^{\text{II}}(\text{dien})$ exceeds 0.01 mol/l, the gas-liquid reaction becomes gas film controlling. The experimental results also depict that the NO removal efficiency increases with the increase of hydrazine hydrate concentration and liquid-to-gas ratio. The optimal temperature range for the absorption of NO is 50–60°C by $\text{Co}^{\text{II}}(\text{dien})\text{-}(\text{NH}_2)_2 \cdot \text{H}_2\text{O}$ solutions.

Based on the current results, this study may provide a basis for developing a new approach for removal of NO from flue gas.

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