International Journal of ELECTROCHEMICAL SCIENCE www.electrochemsci.org

Study on performance of flue gas denitrification by sodium hypochlorite and electrogenerated Ag (II)

Ling Tao^{1,2,*}, Dan Zheng^{1,2}, Jingyue Bao^{1,2}, Xiaoyan Zhu^{1,2}, Menghu Wang^{1,2}

¹ School of Environmental and Biological Engineering, Wuhan Technology and Business University, Wuhan, Hubei 430065, China
² Engineering and Technology Research Center of Hubei Province for Wastewater Reclamation, Wuhan Hubei 430065, China
*E-mail: taolingwtbu@163.com and taoling@wtbu.edu.cn

Received: 10 May 2022 / Accepted: 20 June 2022 / Published: 7 August 2022

This study was done on the performance of flue gas denitrification based on the electrolytic process in the electrochemical scrubbing method. Furthermore, the determination of the optimal condition for the removal of NO_x in scrubber and electro-scrubber systems and a comparison of their removal efficiencies were investigated. The experiments were carried out in a scrubber reactor for NO_x and SO₂ removal from 1000 ppm simulated gas at a flow rate of 1 l/min in NaClO solution, and the results showed that HClO was predominant oxidizing agent in aqueous solution to oxidize NO in the scrubber reactor, and the maximum removal efficiency was obtained at pH 6 and NaClO concentration of 1M. Study the temperature effect on NOx removal efficiency demonstrated that the NO oxidation rate enhanced with increasing the temperature which attributed to temperature effect on the dissolution, mass transfer and diffusion in scrubbing reactions. Investigation the coexisting CO₂, O₂ and SO₂ effects on NOx removal efficiency implied that flue gas denitrification is considerably dominated through a wet scrubbing absorption of NO₂. In addition, the efficiency of Ag(II)/Ag(I) redox mediator to oxidation of NO_x and SO₂ in electro-scrubber system was studied. The comparison between the NO_X and SO₂ removal efficiencies under scrubbing and electro-scrubbing processes indicated to significantly increase of removal efficiency under electro-scrubbing process due to contribution of Ag(II) as mediator. Therefore, the electro-scrubbing process can be used as a reused and cost-effective system for flue gas denitrification in industrial applications.

Keywords: Scrubber system; Electro-scrubber system; NO_x removal efficiency; Ag(II)/Ag(I) redox mediator; Cyclic voltammetry

1. INTRODUCTION

CO₂, CO, NO, NO₂, SO₂, and HC, as well as polycyclic aromatic hydrocarbons, make up the gas portion of diesel exhaust [1]. Light-headedness, headaches, coughs, and nausea can all be caused

by diesel pollution, which irritates the eyes, nose, throat, and lungs. These compositions contain organic carbon which causes cancer in humans and animals [2-4]. Lung cancer risk is increased when workers are exposed to diesel exhaust at work. Diesel cars also emit much more NO_x . The European Commission's scientists reported the share of NO_2 in the total NO_x emissions can reach 60% for diesel vehicles [5-7].

Furthermore, when an engine's air-to-fuel combination is excessively lean, substantial NOx emissions might result. This can happen when there is too much air in the gasoline or when there is not enough fuel put into the engine [8, 9]. This causes the mixture to burn hotter in either situation. The most harmful type of nitrogen oxides is NO_2 . NO_x can cause serious health damage to humans, including respiratory diseases [10]. Smog and the usual brown cloud that surrounds larger cities and causes poor air quality are also caused by NO_x . In the presence of sunshine, NO_x interacts with volatile organic molecules to generate ground-level ozone [11, 12]. The development of efficient denitrification methods to decrease the NOx emissions from industries, cars and ships is essential [13, 14].

Therefore, several research has been conducted for the removal of NO_x using catalytic and photocatalytic processes [15-17], chemical and physical absorptions [18, 19], electrochemical [20, 21], wet scrubbing and wet scrubber-electrochemical cell systems [22-25]. These methods are very expensive and time-consuming. As a result, more parameter studies and experimental tool modifications are required to achieve a cost-effective approach for denitrification technologies. Studies have shown that SO₂ and NO_x may be effectively removed in a wet scrubbing process utilizing hypochlorite-based oxidants, which are produced directly by electrolysis of a NaCl solution with a concentration similar to seawater using the anodic and cathodic reactions described below [26-28]: $2Cl^- \rightarrow Cl_2 + 2e^-$ (1)

 $2H_{2}O + 2e^{-} \rightarrow H_{2} + 2OH^{-}$ (2) $2Na^{+} + 2OH^{-} + Cl_{2} \rightarrow Na^{+}OCl^{-} + Na^{+}Cl^{-} + H_{2}O$ (3)

Therefore, utilization of seawater appears as a cost effective and more efficient solution to this problem. Accordingly, this study was conducted to determine the optimal condition for NO_x removal in scrubber and electro-scrubber systems and compare their removal efficiencies.

2. MATERIALS AND METHOD

2.1. Scrubber system

Denitrification gas was performed in the experimental setup which contained a system for gas production, a scrubber system and a gas analyzer system that is presented in Figure 1a. The system for generation the flue gas was typically consist of a rotary screw gas compressor (ES04A-F, Beijing Ouliyuan Machinery Co., Ltd., China), plug flow reactor (GSH0.5-50L, Weihai Global Chemical Machinery Mfg Co., Ltd., China), and pure N₂, NO, O₂, CO₂ and SO₂ (\geq 99.5%, Foshan Yangte Gas Company Limited., China) gas cylinders for preparation of the simulated flu gas that the flow rates of gases were controlled using mass flow controllers (MFC, Sierra Instrument, Monterey, CA.,USA), and

the gases were blended using on-line mixer. Then, the simulated flue gases passed through the reactor column and were introduced into the scrubber. A roller pump (293KC-ZL, Chongqing Jieheng Peristaltic Pumps Co., Ltd., China) used for pumping fluids from the bottom of a column at a flow rate of 1 l/min under the scrubbing cyclically. There was a typical stainless nozzle (1/8 FD1-SS, Guangdong Boyuan Spraving Technology Co., Ltd., China) at the top of the reactor which was used to spray the fluids with 1 L of 0.05-3M sodium hypochlorite (NaClO, 99.0%, Sigma-Aldrich) as an absorbent under the each cyclic scrubbing process. Each cycle of scrubbing lasted 15 minutes. The concentration of the flue gas at the outlet of the scrubbing process was analyzed at an interval of 15 minutes. The temperature of the solution was measured using water baths (Lagua, Horiba, Japan). The pH of the scrubbing solution was adjusted by adding 0.1 M H₂SO₄ (99.9%, Sigma-Aldrich) solution. The in-situ gas analyzer (GM32, SICK, Germany) was used for measurements of NO_X (the sum of NO and NO₂) concentrations in the inlet (C_{in}) and outlet (C_{out}) flue gases which were used to calculate NO_x removal efficiency (η) through the following relation, shown in Equation (4) [29]: (4)

 $\eta = \left[\frac{c_{in} - c_{out}}{c_{in}}\right] \times 100\%$

2.2. Electro-scrubber system

The experimental scheme of the electro-scrubber system is illustrated in Figure 1b. It is contained of two main sections: an electrochemical cell reactor and a wet scrubber column. The electrochemical cell was employed for electro-generation of Ag(II) that it was consisted of mesh type Pt-coated-Ti as anode and mesh type Ti (Hebei Chaochuang Metal Mesh Co., Ltd., China) as cathode with effective geometrical area of 400 cm² which separated by a Nafion membrane (324, Changzhou City Jianhui Trade and Business Co., Ltd., China). The anolyte and catholyte solutions were circulated continuously through the anode and cathode compartments of the electrochemical cell. The distance between the electrodes was maintained at 4 mm. A 5 M HNO₃ (99.5%, Sigma-Aldrich) containing 0.1 M AgNO₃ (≥99.0%, Sigma-Aldrich) solution as anolyte solution and 2 M H₂SO₄ as catholyte solution were placed in separate anolyte and catholyte tanks (601 PVC, Wuxi Ewater Water Treatment Co., Ltd., China), respectively. The active Ag(II) mediator was galvanostatically generated through electrolysis by applying alternating on/off (3 minute/3 minute) switch of constant current source of 30-80 mA/cm² from the current source (Hebei Huanhai Import & Export Trading Co., Ltd., China).

Two wet scrubber columns were used to evaluate the emission of the mists. The first column was contained an electrolytic liquid of 5 M HNO₃ containing 0.1 M AgNO₃ and was connected to an electrochemical cell and anolyte solution tank. The simulated gas was introduced at the bottom of the first column at flow rate of 50 N.m³/h. The second column was connected to the absorption solution tank with 1M NaClO so that it was exposed to the counter current of flue gas at a flow rate of 1 l/min. Oxidation reduction potential (ORP) measurements were used to determine the concentration of electro-generated Ag(II) in the anolyte through a potentiometric titration with 0.1 M FeSO₄ (≥99%, Merck, Germany) solution. Cyclic voltammetry (CV) measurements were performed using the CS310 Electrochemical Workstation (Xian Yima Optoelec Co., Ltd., China) which contained the Ag/AgCl, Pt plate and Pt wire as the reference, working and counter electrodes, respectively.



Figure 1. Schematic image of the experimental setup of (a) scrubber system and (b) electro-scrubber system.

3. RESULTS AND DISCUSSION

3.1 Determination the optimal condition in Scrubber system

Figure 2a shows the influence of initial pH on the removal efficiency of NO_x from a gas flue. As observed, the η value is slightly increased with increasing the initial pH of the NaClO solution from 3 to 6. The maximum value for η is obtained at a pH of 6, and for pH value higher than 6, the η is drastically decreased. NaClO aqueous solution releases active chlorine that is disinfectant, algaecide, fungicide and microbiocide. Namely, NaClO hydrolyzes to hypochlorous acid (HClO) in water so that HClO participates in equilibrium with chlorine as the following reactions [30-32]:

 $NaClO + H2O \leftrightarrow Na^{+} + HClO + OH^{-}$ (5) $HClO + H3O^{+} + Cl^{-} \leftrightarrow Cl_{2} + 2H_{2}O$ (6)

The ratio of Cl₂/HClO/ClO⁻ is pH dependent, which is indicated in Figure 2b [33-35]. As observed, HClO is predominant in the pH range of 4 to 5.5, whereas the hypochlorite anion predominates at pH >10. Chlorine can be present at pH < 4 only. The increase in η value in the pH range of 4 to 6 is associated with the presence of HClO as a stronger oxidizing agent than OCl⁻ in aqueous solution to oxidize NO. HClO can enhance the rate of NO_x removal through the chain reactions (equations (7) to (13)) which indicate oxidize NO to other soluble NOx species (NO₂, NO₃, N₂O₃, N₂O₄, and N₂O₅) [36-38]:

$NO + HClO \rightarrow NO_2 + HCl$	(7)
$3NO_2 + H_2O \leftrightarrow 2HNO_3 + NO$	(8)
$2NO_2 + H_2O \leftrightarrow HNO_3 + HNO_2$	(9)
$3HNO_2 + 3HNO_3 \rightarrow H_2O + 2NO$	(10)

$NO + NO_2 \rightarrow N_2O_3$	(11)
$2NO_2 \rightarrow N_2O_4$	(12)
$4NO_2 + O_2 \rightarrow 2N_2O_5$	(13)



Figure 2. (a) NO_x removal efficiency as a function of pH (gas flow: 1 l/min; concentration of NaClO: 1 M; inlet NO_x concentration: 1000 ppm; number of scrubbing cycle: 1; solution temperature: 20° C). (b) Chlorine speciation profile as a function of pH [33-35].

Figure 3 shows the effect of the concentration of NaClO on removal efficiency. As seen, the n value is increased with increasing the NaClO concentration to 1M due to the presence of more oxidizing agents to oxidize NO. The reaction (8) helps some of the outlet NO. The oxidation of NO with NaClO aqueous solutions depends on reactions (8) and (9). The oxidation rate of NO increases when the rate of reaction (5) is much greater than the rate of reaction (6). This is because a rapid chemical reaction in a liquid results in a significant increase in the mass transfer rate [39]. Reaction (7) also oxidizes much of the NO near the gas-liquid interface, which leads to the gradient for NO oxidation rate [39, 40]. Evidently, the reaction (7) is not rapid enough to effectively pretermit the resistance in the solution until the NaClO concentration reaches 1M [41, 42]. Therefore, the removal efficiency of NOx is predominantly affected by the absorption of NO₂ [8]. Furthermore, increasing the concentration of NaClO above 1M has no effect on removal efficiency because the wet scrubbing process for removing NOx from gas flues is currently dominated by gas-phase mass transfer [43-45]. Results indicate the maximum removal efficiency is obtained at pH 6 and a NaClO concentration of 1M. Consequently, these values of pH and concentration were selected for further studies. Figure 4 shows the removal efficiency of NO_x under successive cyclic scrubbing process under pH 6 and NaClO concentration of 1M. As seen, NO_x could be completely removed after 45 minutes of scrubbing reaction because of the significant oxidation of NO_x by NaClO (reactions (5) to (13)).



Figure 3. The NaClO concentration effect on NO_x removal efficiency (gas flow: 1 l/min; concentration of NaClO: 0.05-3M; pH: 6; inlet NO_x concentration: 1000 ppm; number of scrubbing cycle: 1; solution temperature: 20°C).



Figure 4. The removal efficiency of NO_x under successive cyclic scrubbing process (gas flow: 1 l/min; concentration of NaClO: 1 M; pH: 6; inlet NO_x concentration: 1000 ppm; number of scrubbing cycle: 0-4; solution temperature: 20° C).

The next set of experiments was carried out in the temperature range from 20° C to 60° C as the temperature range for industrial application for absorbents for the determination of the temperature effect on flue gas denitrification. Figure 5 shows that η value is increased from 72.06 to 83.33 % with increasing the temperature from 20° C to 60° C, demonstrating the NO oxidation rate enhanced with increasing the temperature which attributed to temperature effect on the dissolution, mass transfer and diffusion in scrubbing reactions [46, 47], and consequently, it can enhance the absorption rate of NO₂ into NaClO solutions [48, 49].



Figure 5. The NOx removal efficiency as a function of temperature (gas flow: 1 l/min; concentration of NaClO: 1 M; pH: 6; inlet NO_x concentration: 1000 ppm; number of scrubbing cycle: 1; solution temperature: 20-60°C).

Exhaust emissions from marine diesel engines comprise a mixture of CO_2 , O_2 , and SO_2 which can partially affect the oxidation of NO under certain conditions. Therefore, it is essential to evaluate the effect of CO_2 , O_2 , and SO_2 on NO_x removal efficiency. Figure 6a depicts the coexisting CO_2 effect on NO_x removal efficiency. It can be observed that η value is slightly increased with increasing the CO_2 in inlet flue gas from 0 to 10%, and for more content of CO_2 the η value is imperceptibly decreased. It is related to the acidic oxide characteristics of CO_2 and its ability to react with NO_x and absorbent through chlorine hydrolysis equilibrium in wet scrubber as following reactions [50, 51].

$CO_2 + H_2O \leftrightarrow CO_3^{2^-} + 2H^+$	(14)
$CO_2 + H_2O \leftrightarrow HCO_3^- + H^+$	(15)
$Cl_2 + H_2O \leftrightarrow HClO + H^+ + Cl^-$	(16)
$HClO \leftrightarrow ClO^- + H^+$	(17)

Figure 6b displays the O_2 content effect on NO_x removal efficiency. As seen, the removal efficiency is slightly increased with increasing the O_2 content during the scrubbing process, which is related to the oxidation of a little of NO into NO_2 under the introduction of O_2 as the following reaction [52]:

$$O_2 + NO \rightarrow 2NO_2$$
 (1)

The results indicate that flue gas denitrification is considerably dominated by the wet scrubbing absorption of NO₂ [53, 54].

8)



Figure 6. The effect of (a) CO_2 and (b) O_2 on NOx removal efficiency (gas flow: 1 l/min; concentration of NaClO: 1M; pH: 6; inlet NO_x concentration: 1000 ppm; number of scrubbing cycle: 1; solution temperature: 20°C).

Figure 7a exhibits the SO₂ and NO_x removal efficiency under a successive cyclic scrubbing process. It can be observed that the 100% removal of NO_x of SO₂ is obtained after the third and first cyclic scrubbing. The fast rate of removal of SO₂ is because of the great solubility of SO₂ and its appropriate adsorption in scrubbing solution [55, 56]. The mechanism for the removal of SO₂ is based on the following hydrolysis reactions [57-59]:

 $SO_{2} + H_{2}O \rightarrow HSO_{3}^{-} + H^{+}$ (19) $HSO_{3}^{-} \rightarrow SO_{3}^{2-} + H^{+}$ (20) $SO_{3}^{-} + HClO \rightarrow SO_{4}^{2-} + HCl$ (21)

In these chain reactions, the active chlorine species can rapidly oxidize the hydrolysis products (reaction (21)). Figure 7b shows the SO₂ effect on NO_x removal efficiency which indicates a slight decrease in NO_x removal efficiency is observed with increasing the SO₂ content in inlet gas, and the complete removal of NO_x with the addition of 5%, 10% and 15% SO₂ is obtained after 60, 75 and 90 minutes of scrubbing process. The decrease in removal rate of NO_x it attributed to competitive oxidation reactions between SO₂ and NO_x [29, 57, 60], which consume the oxidants during the oxidation-absorption and hydrolysis reactions of SO₂ in wet scrubber systems [29, 57, 58]. The results illustrate that NaClO solution can be employed for the simultaneous treatment of NO and SO₂ from exhaust gas.



Figure 7. (a) The removal efficiency of SO₂ (inlet concentration: 1000 ppm) and NO_x (inlet concentration: 1000 ppm) under successive cyclic scrubbing process; (b) SO₂ effect on NO_x removal efficiency (gas flow: 1 l/min; concentration of NaClO: 1 M; pH: 6; inlet NO_x concentration: 1000 ppm; number of scrubbing cycle: 0-6; solution temperature: 20°C).

3.2 study of electro-scrubber system

Figure 8 displays the CV curves of an anolyte solution before electrolysis and after 1 hour and 12 hours of electrolysis. There is not any peak associated with the presence of the Ag^{2+} in anolyte solution in the CV curves of Figure 8a. The observed peak in CV curve of Figure 8b is correlated with mediated electrochemical oxidation of high oxidation capacity of Ag^{2+} (E₀ = 1.98 V) which is generated anodically from Ag^+ in an aqueous HNO₃ solution in an electrochemical cell with a Nafion membrane separator between the anode and the cathode according to the following reactions [61-63]:

$Ag^+ \rightarrow Ag^{2+} + e^-$	(22)
$Ag^{2+} + NO_3^- \rightarrow Ag(II)NO_3^+$	(23)
$Ag^+ + NO_3^- \rightarrow Ag(II)NO_3^+ + e^-$	(24)
$HNO_3 + 2H^+ + 2 e^- \rightarrow HNO_2 + H_2O$	(25)
$3HNO_2 \rightarrow HNO_3 + H_2O + 2NO$	(26)
$HNO_3 + 3H^+ + 3e^- \rightarrow 2H_2O + NO$	(27)

Comparison between the CV curves in Figures 8c and 8b illustrates that there is a slight decrease in redox peak current and positive peak potential shift which indicates the small decrease in performance of the system to for the generation of Ag(II) due to increase the NO₃ content during the salvation of AgNO₃ under applying the alternating constant current source [64, 65].



Figure 8. CV curves of anolyte solution (a) before electrolysis and (b) after 1 hours and (c)12 hours electrolysis at scan rate of 50 mV/s.

In order to evaluate an effective approach to maintaining Ag(II) for industrial application, the current effect on the oxidation efficiency of Ag(I) was investigated over 12 hours, which is presented in Figure 9 in 5 M HNO₃ anolyte containing 0.1 M AgNO₃ circulation flow rate of 1 l/min. As observed, the oxidation efficiency at a current density of 70 mA/cm² exhibits an initial exponential increase to 30.10% Ag(II).



Figure 9. Current densities effect on oxidation efficiency of Ag(I) (current density: 30-80 mA/cm²; anolyte: 5 M HNO₃ containing 0.1 M AgNO₃; circulation flow rate: 1 l/min).

The oxidation efficiency is maintained at almost constant levels for 12 hours (28.47%). The small decrease of oxidation efficiency (>6%) indicates to great stability of the system to electrogenerated Ag(II). The causes of small decrease in Ag(II) generation can be attributed to slow electron transfer and water splitting by electrogenerated Ag(II) whose products can combine with Ag(II) [64]. Moreover, Figure 9 shows the current density can effectively change the oxidation rate in

an electrochemical cell. Increasing the current density to 70 mA/cm² leads to an increase in the generation efficiency of Ag(II), and the maximum and most stable generation efficiency of Ag(II) is observed at a current density of 70 mA/cm². For applied current density more than 70 mA/cm², the increase of water oxidation rate can decreases the concentration of electrogenerated Ag(II) [66, 67]. Therefore, the current density of 70 mA/cm² as the optimum current density was applied for the following experiments.Figure 10a demonstrates the comparison between the NO_X removal efficiency under scrubbing and electro-scrubbing processes. The results indicated to significantly increased NO_X removal efficiency under electro-scrubbing process due to the contribution of Ag(II) as mediator to oxidation of NOx as the following reactions [68]:

 $2NO + 5Ag(II) + 3H2O \rightarrow NO_2 + HNO_3 + 5Ag(I) + 5H^{+}$ (28) $NO_2 + Ag(II) + H_2O \rightarrow HNO_3 + Ag(I) + H^{+}$ (29) $HNO_2 + Ag(II) + H_2O \leftrightarrow HNO_3 + Ag(I) + 2H^{+}$ (30)

Figure 10b also shows the SO₂ effect on NO_x removal efficiency under electro-scrubbing process which demonstrated to enhance the simultaneous removal rate of SO₂ and NO_x by electrogenerated Ag(II) due to dissociation into H^+ and HSO₃⁻ for aqueous SO₂ [69, 70]. Therefore, the electro-scrubbing process can be used as reused and cost-effective system for flue gas denitrification of pollutants in industrial applications.



Figure 10. (a) NO_X removal efficiency under scrubbing and electro-scrubbing processes, (b) SO₂ effect on NO_x removal efficiency under electro-scrubbing process.

4. CONCULUSION

This study was conducted to determine the optimal conditions for the removal of NO_x in scrubber and electro-scrubber systems and to compare their efficiencies for flue gas denitrification. The experiments were carried out in the scrubber reactor for NO_x removal from 1000 ppm simulated gas at a flow rate of 1 l/min in NaClO solution, and results showed the maximum removal efficiency was obtained in pH 6 and NaClO concentration of 1M. HClO was the predominant oxidizing agent in

aqueous solution to oxidize NO in the scrubber reactor which can enhance the rate of NO_x removal. Results exhibited that NO_x could be completely removed after 45 minutes of scrubbing reaction because of the significant oxidation of NO_x by NaClO. Study the temperature effect on NO_x removal efficiency demonstrated that the NO oxidation rate enhanced with increasing the temperature which attributed to temperature effect on the dissolution, mass transfer and diffusion in scrubbing reactions. Investigation the coexisting CO_2 , O_2 and SO_2 effects on NOx removal efficiency implied that NOx was considerably removed through a wet scrubbing absorption of NO_2 , and the SO_2 showed the faster removal rate than NOx because of its great solubility and appropriate adsorption in the scrubbing solution. In addition, the efficiency of Ag(II)/Ag(I) redox mediator to oxidation of NO_x and SO_2 in electro-scrubber system was studied. The comparison between the NO_x and SO_2 removal efficiencies under scrubbing and electro-scrubbing processes revealed that the electro-scrubbing process had a significantly higher removal efficiency due to the contribution of Ag(II) as a mediator. Therefore, the electro-scrubbing process can be used as a reused and cost-effective system for the elimination of pollutants in industrial applications.

ACKNOWLEDGEMENTS

The study was supported by "Hubei Natural science foundation, China (Grant No. 2019CFC925)". and the Special Fund of Advantageous and Characteristic disciplines (Group) of Hubei Province.

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