

# Research Progress in Flue Gas Denitrification Technology Based on Oxidation Theory

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Because of its mild reaction condition, relatively high denitrification efficiency and ability of combined removal of various pollutants, the flue gas denitrification technology based on oxidation theory has become a new and feasible method for flue gas denitrification. The technology has been applied extensively and studied in depth. This paper reviews the influencing factors, reaction mechanism, reaction products and subsequent absorption reaction of the oxidation of NO in flue gas with gas phase and liquid phase oxidants at high and low temperatures. On this basis, the shortcoming of the technology and the key research directions for the future are analyzed and discussed, laying the basis for industrial applications of the denitrification technology based on oxidation theory.

## 1. Introduction

Nitrogen oxides (NO<sub>x</sub>) are major air pollutants and important causes of acid rain, photochemical smog, haze, etc (Benhorma et al., 2017; Di Iorio et al., 2016; Barajas-Solano et al., 2016; Scaglione et al., 2016; Sibilio et al., 2016). According to statistics, China's nitrogen oxide emissions reached 23.378 million tons in 2012. Therefore, the denitrification of flue gas is an urgent requirement for environmental protection.

At present, the main technologies used in flue gas denitrification include selective catalytic reduction (SCR) technology, selective non-catalytic reduction (SNCR) technology, SNCR-SCR combined denitrification technology (Gap et al., 2017). Among them, the SCR is the most popular choice for flue gas denitrification (Puccini et al., 2016; Jecha et al., 2017). Nevertheless, all these denitrification technologies only work within specific temperature ranges. What is worse, the SCR technology faces potential catalyst poisoning and ammonia escape due to the usage of a large number of catalysts. It is also troubled by high construction and operation costs. Against this backdrop, it is necessary to develop a new flue gas denitrification technology.

NO accounts for over 90% of the NO<sub>x</sub> in flue gas. The rest 10% is mainly NO<sub>2</sub>. There are essential differences between the NO and NO<sub>2</sub>: NO<sub>2</sub> is easily soluble in water; the resulting products HNO<sub>3</sub> and HNO<sub>2</sub> can react with the common alkaline substances (e.g. Ca(OH)<sub>2</sub>); in contrast, NO is barely soluble in water and unable to react with common alkaline absorbents. The differences call for new denitrification technology for NO, a new field in flue gas denitrification.

Currently, the oxidants used in oxidation treatment of NO mainly fall into the categories of gas phase oxidants (O<sub>3</sub>, halogen gases, etc.) and liquid phase oxidants (NaClO<sub>2</sub>, KMnO<sub>4</sub>, H<sub>2</sub>O<sub>2</sub>, etc.) (Schildberg, 2016; Lima et al., 2017). The oxidation of NO can be performed with gas phase oxidants at a high temperature or liquid phase oxidants at a low temperature. However, the flue gas denitrification technology based on oxidation theory is still in the phase of lab research. The technology is not fully mature, the reaction mechanism is not thoroughly understood, and engineering applications are far from sufficient. Despite the shortcoming, the flue gas denitrification technology based on oxidation theory has attracted the attention from various researchers thanks to its mild reaction condition, relatively high denitrification efficiency and ability of combined removal of various pollutants (SO<sub>2</sub>, NO<sub>x</sub>, Hg<sup>0</sup>). It is poised to become a popular flue gas denitrification technology in the near future.

## 2. Gas phase oxidants

Featuring strong oxidizing properties, certain gas phase substances can be utilized to oxidize the NO in flue gas into high-valent NO<sub>x</sub>, and ultimately absorb and remove the NO. Such gas phase oxidants include O<sub>3</sub>, halogen gas, etc. Out of all these gas phase oxidants, O<sub>3</sub> has been proved in countless studies to have the largest application potential and least likelihood of secondary pollution (Cannistraro et al., 2016; Spinelle et al., 2016).

### 2.1 Denitrification based on O<sub>3</sub> oxidation

#### 2.1.1 High temperature denitrification with gas phase O<sub>3</sub>

O<sub>3</sub> may oxidize or decompose the NO<sub>x</sub> at a high temperature, reducing or even eliminating the ability of the latter to oxidize NO. Good practice indicates that only 28% of O<sub>3</sub> is decomposed in 10s at 150°C, the typical exhaust gas temperature of boilers. At this temperature, the survival time of O<sub>3</sub> is much longer than the duration of kinetic reaction between O<sub>3</sub> and NO. Thus, the temperature has little effect on the reaction process. Besides, the decomposition of O<sub>3</sub> is independent of its initial concentration. In the range of 100~200°C, O<sub>3</sub> can efficiently oxidize NO and the oxidation of NO is insensitive to temperature changes. However, as the temperature continues to increase, the decomposition rate of O<sub>3</sub> is accelerated and the NO oxidation efficiency is decreased. By the time the temperature reaches 400°C, the O<sub>3</sub> has lost all of its oxidation capacity.

At a high temperature, the oxidative denitrification with gas phase O<sub>3</sub> covers a total of 19 substances, namely O<sub>3</sub>, O<sub>2</sub>, H, O, OH, H<sub>2</sub>O, H<sub>2</sub>O<sub>2</sub>, HO<sub>2</sub>, N<sub>2</sub>, H<sub>2</sub>, N, NO, NO<sub>2</sub>, NO<sub>3</sub>, N<sub>2</sub>O, HNO<sub>2</sub>, HNO, HNO<sub>3</sub>, and N<sub>2</sub>O<sub>5</sub>, and may involve 65 kinds of elementary reactions. According to of theoretical analysis and experimental studies, the NO oxidation is a staged process: First, the NO is oxidized into NO<sub>2</sub>, plus a few amounts of NO<sub>3</sub> and N<sub>2</sub>O<sub>5</sub> if there is excess O<sub>3</sub> ( $n(\text{O}_3) : n(\text{NO}) \geq 1$ ).

After the oxidation of NO with gas phase O<sub>3</sub> at a high temperature, the high valence NO<sub>x</sub> products are either used to prepare HNO<sub>3</sub>, or absorbed and removed by the wet flue gas washing apparatus (Wang et al., 2007). If the products are used to prepare HNO<sub>3</sub>, the generation of HNO<sub>3</sub> in gas-liquid reaction is significantly affected by the concentrations of O<sub>3</sub> and NO<sub>3</sub> when the gas flue temperature falls between 100 and 160°C. The molar ratio of O<sub>3</sub>/NO should be above 1.5. High molar ratio of O<sub>3</sub>/NO and low simulated gas flue temperature stimulate the production of NO<sub>3</sub><sup>-</sup> and provide high volume fraction of O<sub>3</sub> for the gas-liquid reaction. In this way, the formation of HNO<sub>3</sub> is promoted and that of HNO<sub>2</sub> is suppressed. With water as the tail absorbent, the denitrification efficiency is enhanced with the increase of (O<sub>3</sub>/NO). When (O<sub>3</sub>/NO)=0.9, the efficiency reaches 86.27%. With CaCO<sub>3</sub> as the tail absorbent, the absorbent concentration and the gas-liquid ratio M must be maintained in a certain range. The critical point M falls between 600~700 when [CaCO<sub>3</sub>]=0.05 mol/l, 1200~1300 when [CaCO<sub>3</sub>]=0.1 mol/l, and 1,900~2,000 when [CaCO<sub>3</sub>]=0.15 mol/l .

Although the flue gas denitrification technology based on oxidation theory is still in the phase of lab research in China, it has been systematically studied and practiced in foreign countries. Developed by BOC Sciences (US), the LoTOx technology oxidizes NO<sub>x</sub> with O<sub>2</sub>/O<sub>3</sub> gas mixture, and then carries out two-stage washing using CaCO<sub>3</sub>/NaOH. The denitrification efficiency surpasses 90% (Boc, 2000). Young et al., introduced O<sub>3</sub> into the flue gas to oxidize NO, and used Na<sub>2</sub>S and NaOH solution for liquid phase absorption, achieving an NO<sub>x</sub> removal rate of up to 95% (Sun and Lee, 2006).

#### 2.1.2 Low temperature oxidative denitrification with liquid phase O<sub>3</sub>

The first step to liquid phase oxidation of NO is the dissolution of the gas in solution, which is consistent with Henry's law. The conversion of NO into an easily absorbable form is the key to denitrification because of the extremely low solubility of NO in water (the Henry's law constant is  $1.94 \times 10^{-8} \text{ mol} \cdot \text{L}^{-1} \cdot \text{Pa}^{-1}$  at 25°C).

The liquid denitrification procedure is roughly divided into the following stages: the absorption of NO, the liquid phase oxidation of NO into high-valent NO<sub>x</sub>, and the gas phase oxidation of NO into easily soluble high-valent NO<sub>x</sub>. In the liquid phase, O<sub>3</sub> reacts with NO at a fast rate. The reaction generates easily water-soluble NO<sub>2</sub>, NO<sub>3</sub>, etc. and destroys the solubility equilibrium of NO, thereby promoting the dissolution, absorption and oxidation of NO.

Under low temperature conditions, O<sub>3</sub> either undergoes decomposition or oxidizes NO. At 150°C, O<sub>3</sub> can react with NO swiftly to produce NO<sub>2</sub>, and the decomposition rate of O<sub>3</sub> at this temperature is less than 4%. Hence, the reaction is a fast, irreversible reaction. When the temperature falls below 100°C, only 0.5% of O<sub>3</sub> will be decomposed within 10s. Although the decomposition of O<sub>3</sub> is accelerated with the increase of temperature, the decomposition rate is much slower than the reaction rate with NO under low temperature, liquid phase conditions.

Ma Shuangchen et al., carried out a systematic study on this issue. They performed an experiment on oxidative denitrification with liquid phase O<sub>3</sub> in a self-made bubbling reactor. The results show that the pH

value and temperature have a little impact on the removal rate of NO; in the range of 20–65°C, the NO removal rate does not change significantly; when the molar ratio  $[O_3]/[NO] = 0.8$ , the NO efficiency can reach 90%; SO<sub>2</sub> does not have an obvious effect on the removal of NO for it is hard to be oxidized and removed by O<sub>3</sub> in the homogeneous reaction .

### 3. Liquid phase oxidative denitrification

#### 3.1 Oxidative denitrification with H<sub>2</sub>O<sub>2</sub>

Under different reaction conditions, H<sub>2</sub>O<sub>2</sub> can be decomposed into four types of products: ·OH+·OH, HO<sub>2</sub>·+H·, H<sub>2</sub>O+O<sub>2</sub> or H<sub>2</sub>O+HO<sub>2</sub>·. Table 1 lists the standard electrode potentials of some oxidants. As shown in the table, ·OH boasts a strong oxidation capacity (its standard electrode potential is 2.8 V). To make use of the oxidation capacity of ·OH, it is necessary to create the reaction conditions to decompose H<sub>2</sub>O<sub>2</sub> into OH·. Nonetheless, the utilization of H<sub>2</sub>O<sub>2</sub> is constrained by the lack of the ability to produce sufficient ·OH at low temperatures. Many researchers have explored how to strengthen the oxidation capacity of H<sub>2</sub>O<sub>2</sub> and decompose it into a large number of ·OH. One of the focal points is the advanced oxidation technology.

Table 1: Standard electrode potentials of some oxidants

Oxidant	Electrode reaction	Standard electrode potential/V
F <sub>2</sub>	$F_2+2e \rightarrow 2F^-$	2.87
·OH	$\cdot OH+H^++e \rightarrow H_2O$	2.80
O <sub>3</sub>	$O_3+2H^++2e \rightarrow H_2O+O_2$	2.07
H <sub>2</sub> O <sub>2</sub>	$H_2O_2+2H^++2e \rightarrow 2H_2O$	1.77
MnO <sub>4</sub> <sup>-</sup>	$MnO_4^-+8H^++5e \rightarrow Mn^{2+}+4H_2O$	1.52
ClO <sub>2</sub>	$ClO_2+e \rightarrow Cl^-+O_2$	1.50
Cl <sub>2</sub>	$Cl_2+2e \rightarrow 2Cl^-$	1.36
O <sub>2</sub>	$0.5O_2+2H^++2e \rightarrow H_2O$	1.23
Oxidant	Electrode reaction	Standard electrode potential/V
Oxidant	Electrode reaction	Standard electrode potential/V

Advanced oxidation processes (AOPs), also known as deep oxidation processes, are a set of chemical treatment procedures proposed by Glaze et al. in 1987. They stand for any chemical oxidation technologies realized through reactions with lots of hydroxyl radicals (William et al., 1987; Zhuang and Yan, 2016). The AOPs usually use the free radical (·OH) to oxidize pollutants, turning them into less toxic and easy-to-remove substances.

Aiming to promote the decomposition of H<sub>2</sub>O<sub>2</sub> into ·OH, the AOPs research at home and abroad has put emphasis on the following aspects:

(1) The advanced oxidation technology of high temperature H<sub>2</sub>O<sub>2</sub>

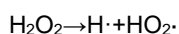
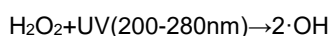
The main reactions between H<sub>2</sub>O<sub>2</sub> and NO in the flue gas are listed as below (Cooper et al., 2004):

When the reaction temperature is above 400°C, the reaction proceeds in the direction of the formation of NO<sub>2</sub> and HNO<sub>3</sub>. Specifically, the NO is oxidized by the H<sub>2</sub>O<sub>2</sub> directly injected into the high-temperature flue or spray tower, and the resulting high-valent NO<sub>x</sub> are absorbed and removed (Rossi and Unfried, 1997).

When the flue temperature surpasses 400°C, the H<sub>2</sub>O<sub>2</sub> injected into the flue will release strong oxidizing groups like ·OH and HO<sub>2</sub>·, and oxidize the NO in the flue gas into high-valent NO<sub>x</sub>. In this process, the oxidation effect is improved by adjusting and optimizing the position and type of the nozzle, the molar ratio of H<sub>2</sub>O<sub>2</sub>/NO, the temperature, and the SO<sub>2</sub> concentration. According to Collins' research in 2001, the H<sub>2</sub>O<sub>2</sub> solution injected into the hot flue gas can oxidize NO into easily soluble NO<sub>2</sub>, HNO<sub>2</sub> and HNO<sub>3</sub>, and increase the NO oxidation ratio to over 90% (Collins et al., 2001; Su et al., 2016).

(2) The advanced oxidation technology of UV/H<sub>2</sub>O<sub>2</sub>

The photolysis of H<sub>2</sub>O<sub>2</sub> under UV irradiation generates ·OH through the following steps:



In this respect, Cooper et al. conducted an in-depth and detailed study. They placed a UV lamp in the flue to activate the release of ·OH by H<sub>2</sub>O<sub>2</sub> and achieve the oxidation of NO with the released ·OH. The conversion rate fell between to 10% -70%. In addition, they had meaningful discussion over the mechanism of NO oxidation with H<sub>2</sub>O<sub>2</sub> under UV irradiation (Cooper et al., 2002).

However, the research by Cooper et al. was conducted in high-temperature flue gas. At such high temperatures, the flue gas is not yet desulfurized. In the oxidation process, the presence of SO<sub>2</sub> not only consumes a lot of H<sub>2</sub>O<sub>2</sub>, but also affects the oxidation of NO. In this background, the oxidation of desulfurized flue gas with H<sub>2</sub>O<sub>2</sub> under UV irradiation has become a research hotspot.

Ma Shuangchen et al., spent much effort on this topic. They studied the effects of H<sub>2</sub>O<sub>2</sub> concentration, NO<sub>x</sub> initial concentration, pH value, O<sub>2</sub> concentration, temperature, metal ion catalyst and other technological parameters on NO<sub>x</sub> removal rate in a self-made bubbling reactor under low temperature conditions (below 45°C), and optimizes these parameters. It is discovered that the NO<sub>x</sub> removal rate exceeds 95% when the pH value is around 3.3, the O<sub>2</sub> concentration is greater than 6%, the temperature is controlled below 45°C and the metal catalytic ions are added.

(3) The advanced oxidation technology of Fenton method

The Fenton reaction is essentially the ·OH-producing chain reaction between Fe<sup>2+</sup> and H<sub>2</sub>O<sub>2</sub>. During the oxidative denitrification by the Fenton method, the produced ·OH fully oxidizes the NO in flue gas. The oxidation effect is influenced by the H<sub>2</sub>O<sub>2</sub> concentration, the Fe<sup>2+</sup> dosage, the initial pH value, UV irradiation and temperature. Under proper reaction conditions, the denitrification efficiency may go beyond 80%.

In spite of some related studies, researchers are still exploring the reaction mechanism of oxidative denitrification by Fenton method. The main reactions in the Fe<sup>3+</sup>/H<sub>2</sub>O<sub>2</sub> system are as follows (Laat et al., 2005):

There are a series of mutual reactions between Fe<sup>2+</sup> and Fe<sup>3+</sup>. The equilibrium of the two affects the generation of free radicals. The following reactions may take place between Fe<sup>2+</sup> and Fe<sup>3+</sup> (Laat et al., 1999; Pignatello et al., 1999):

The presence of UV light will accelerate the speed of free radicals, which in turn promote the oxidation and removal of NO. At this point, the following reactions may occur in addition to the above reactions (Tokumura et al., 2008):

### 3.2 Oxidative denitrification with NaClO<sub>2</sub>

NaClO<sub>2</sub> is a disinfectant and decolorizer widely used in water supply, as well as disinfection and oxidation treatment of wastewater. Prepared by in-situ electrolysis of brine, NaClO<sub>2</sub> has a clear edge over other oxidants in that it is easily produced, strongly oxidative and safe and reliable to operate (e.g. no leak of chlorine or explosion).

Studies have shown that the removal of NO<sub>x</sub> by NaClO<sub>2</sub> oxidation is a gas film-controlled absorption-oxidation reaction (Brogren et al., 1998; Hsu et al., 1998; Chien and Chu, 2000). NO<sub>x</sub> is mainly absorbed through the hydrolysis of N<sub>2</sub>O<sub>3</sub> and N<sub>2</sub>O<sub>4</sub>; NO can be oxidized into NO<sub>3</sub><sup>-</sup> by NaClO<sub>2</sub>, and ClO<sub>2</sub><sup>-</sup> is reduced to Cl<sup>-</sup> and ClO<sup>-</sup>. The acid HNO<sub>3</sub> solution produced in the reaction dissolves NaClO<sub>2</sub> into ClO<sub>2</sub>, which further oxidizes NO.

### 3.3 Oxidative denitrification with KMnO<sub>4</sub>

Under strong alkaline conditions, NO is oxidized to NO<sub>2</sub><sup>-</sup> by MnO<sub>4</sub><sup>-</sup>; under weak alkaline or neutral conditions, it is oxidized to NO<sub>3</sub><sup>-</sup>. Chu et al., (1998 and 2001) suggested that the side effects of SO<sub>2</sub> in the flue gas on the oxidative absorption reaction should not be ignored albeit the MnO<sub>4</sub><sup>-</sup>'s ability to oxidize and remove NO; moreover, the further promotion of the method was limited by the complex preparation process and high price of the oxidant KMnO<sub>4</sub>.

## 4. Conclusions and prospects

The past decade has witnessed the rapid development of the flue gas denitrification technology. With more and more advantages, the technology now boasts broad application prospects. For the purpose of removing the NO<sub>x</sub> from the flue gas, the technology rests on the conversion of the NO in flue as into high-valent NO<sub>x</sub> that are easily absorbed and handled. The existing studies concentrate on the oxidation effect of different oxidants, the influencing factors of oxidation, and the possible oxidation mechanism. However, further research indicates that the following issues are yet to be solved for the flue gas denitrification technology based on oxidation theory:

- (1) Despite the relatively high denitrification efficiency, the flue gas denitrification technology based on oxidation theory generally usually removes about 80% of NO<sub>x</sub>. The efficiency must be further improved in future studies.
- (2) The oxidants like O<sub>3</sub> and H<sub>2</sub>O<sub>2</sub> are favored by researchers thanks to the low likelihood of secondary pollution. However, such oxidants are unstable, and the reactions are difficult to control.
- (3) The popular oxidants are usually very expensive, raising the need for the R&D of efficient and cheap oxidants for denitrification.

(4) The research into the oxidative denitrification reaction is still in the initial stage. More in-depth and detailed studies are expected to disclose the underlying mechanism of oxidative denitrification.

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