The Review of Flue Gas Desulfurization with a Readily Available Metal Ions Liquid Catalytic Oxidation Catalyst-Pulp

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Abstract

Liquid catalytic oxidation technology by transition metal ions is one of the hot topics in the field of flue gas desulfurization. As pulp contains abundant transition metal ions, desulfurization technology by pulp has been investigated. The review introduced pyrolusite pulp desulfurization research, including the main influence factors, desulfurization mechanism analysis, process equipment as well as combined effect of pyrolusite pulp and microbes; the state of industrial application of phosphate pulp desulfurization; the investigation of coal pulp flue gas desulfurization. All the above pulp flue gas desulfurization achieved excellent results, with high SO$_2$ removal rate and obtaining by-products. Pulp is a kind of cheap and readily available metal ions liquid catalytic oxidation desulfurization catalysts.

Keywords: Pulp; Flue gas desulfurization; SO$_2$; Liquid catalytic oxidation

Introduction

As one of the main atmospheric pollutants, SO$_2$ is the immediate cause of acid rain, which can result in dead trees, food production decrease, water acidification, building corrosion, deterioration of ecological environment, and serious harm to human health [1]. SO$_2$ is mainly derived from flue gas emissions of the thermal power industry, nonmetal mineral products industry, chemical industry and metallurgy industry [2]. Scholars did a lot of research on flue gas desulfurization, and developed more than one hundred kinds of desulfurization methods, divided into dry and wet kinds according to the forming process [3]. In the industrialization methods, limestone-gypsum was used mostly, which was cheap and produced by-product gypsum, while the gypsum was difficult to be used, economic was not obvious, and it caused the secondary pollution [4]. Developing a new desulphurization technology which can not only remove SO$_2$ from flue gas, but also recycle sulfur resources to get high value by-product will be an effective way to solve SO$_2$ pollution. In recent years, liquid phase catalytic oxidation flue gas desulfurization technology are paid more and more attention [5,6], such as Mn(II) system, Fe(II) and Fe(III) system, various ions coordinated system, heteropoly acid system, which don't consume catalyst in theory, and can regenerate by changing valence state of transition metal ions, without solid waste and secondary pollution. In addition, it can make valuable by-products, fertilizer, ferrous sulfate, manganese sulfate, polymeric ferric sulfate, and so on. By investigation, the addition of Mn, Cu, Co to Fe(II)catalyst, not only can play promoting and synergy performance, but also make Fe(II) catalytic activity stable, and the absorbing liquid used to remove SO$_2$ from smelter flue gas has achieved good effect [7]. From the view of sulfur resource utilization, environment protection and sustainable development, recycling method should be chosen firstly in flue gas desulfurization.

Pulp contains a variety of transition metal ions, which is in favor of SO$_2$ absorption. In the process of pulp absorbing SO$_2$, transition metal ions will continue into the solution, becoming cheap metal ions liquid catalytic oxidation desulfurization catalysts [8-10]. Currently, pyrolusite pulp, phosphate pulp and coal pulpare researched for flue gas desulfurization. In this paper, the current study on pulp flue gas desulfurization was introduced.

The Research Status of Pulp Flue Gas Desulfurization

Pyrolusite pulp

MnO$_2$ is the main ingredient of pyrolusite pulp, which is a good antioxidant for treatment SO$_2$ in flue gas [11]. In acidic environment, Mn$^{2+}$ is the most active reaction medium for SO$_2$oxidation [12]. Pyrolusite pulp also contains Fe$_3$O$_4$, Al$_2$O$_3$, CaO, and other impurities. These impurities have the catalytic oxidation of SO$_2$, and can free up...
in acidic liquid, leading to the catalytic oxidation rate of SO\(_2\) improving 3-10 times than that singly using Fe\(^{3+}\)-Fe\(^{2+}\) or Mn\(^{2+}\) as catalyst. Fan chose pyrolusite, MnO\(_2\).Fe\(_2\)O\(_3\) to modify walnut shell-derived column activated carbon by blending method respectively, and achieved the maximum sulfur capacity of activated carbon loaded by pyrolusite was 227.8mg/g [13]. Using the reaction characteristics of pyrolusite pulp and SO\(_2\), to do flue gas desulfurization and obtain by-products MnSO\(_4\), or dilute sulphuric acid, not only can eliminate secondary pollution in the desulfurization process, realize the comprehensive utilization of lean ore, but also can achieve the goal of recovery of sulfur resources, decrease the desulfurization cost [14,15].

The main influence factors

By investigating, Zhu found that stirring intensity had a dual role on desulfurization and manganese leaching efficiency [16,17]. Under low stirring speed (<500r/min), the desulfurization reaction of MnO\(_2\) and SO\(_2\), in the solution was major, with MnSO\(_4\) as main by-product. Under high stirring speed (>1000r/min) and the catalysis of Mn\(^{2+}\), Fe\(^{3+}\) etc., due to the dissolved oxygen concentration in the solution increasing, water absorbing SO\(_2\), and MnO\(_2\).SO\(_2\) oxidation reaction coexisted, with the mixture of MnSO\(_4\) and dilute sulphuric acid as by-products, which was a new finding that SO\(_2\), in flue gas could be oxidized to rare H\(_2\)SO\(_4\). If MnSO\(_4\) needs to be recycled, stirring speed should be controlled below 500r/min. In order to obtain dilute sulphuric acid, stirring speed should be increased above 1000r/min. Increasing pyrolusite pulp temperature, the reaction rate and mass transfer rate improved, while SO\(_2\) solubility decreased. The experimental results showed that from room temperature to 80 °C, the influence of two aspects offset. Improving the pulp temperature appropriately, was benefit to decrease the generation of S\(_2\).O\(_4\)\(^{2-}\), which produced in the side reaction and influenced the quality of MnSO\(_4\) [18]. Hu and Jin indicated that 40 °C was the ideal temperature by experiments [19,20]. Considering energy consumption, fresh pulp doesn’t need to be heated as the high flue gas temperature.

Maintaining a low pH value (PH=1) in desulfurization process, can enhance MnO\(_2\) oxidation ability, and promote its reaction with SO\(_2\), to increase Mn leaching rate and SO\(_4\)\(^{2-}\) generation during the same reaction time. Less the rest of MnO\(_2\), in pulp was, faster the desulfurization rate decreased [14]. Sun thought that the low pH was against SO\(_2\), dissolving, and affected the reaction of MnO\(_2\) and SO\(_2\). In order to speed up the desulfurization rate, it was necessary to add buffer to the reaction system. Hu reported that under the condition of pH 2-3.5, temperature 40 °C, the emission of SO\(_2\), was up to standard, more than 80% Mn could be recycled, and SO\(_2\), absorption rate reached over 90% [19].

For continuous operation equipment, liquid gas ratio (L/V) is an important parameter. With the increase of flue gas flow, the amount of gas into the liquid phase in a unit time rises, but SO\(_2\), in the liquid phase stays for a shorter time, leading that SO\(_2\) contacts with pulp inadequately. Besides, the initial amounts of SO\(_2\), react with pulp quickly, making SO\(_2\), excessive in the subsequent reaction, which reduces the desulfurization efficiency. The larger L/V is, the higher SO\(_2\), absorption rate is, and the larger energy consumption and the cost of conveying liquid pumps, pipes and other equipment fee are. When the L/V reached 40, the absorption rate increased not obviously. Comprehensively considering the above factors, it was indicated that 40 was optimal liquid gas ratio [20]. Li et al. [20] also investigated the influence of liquid-solid ratio on the desulfurization rate and Mn leaching rate [15]. She indicated that the higher liquid-solid ratio was, the shorter the duration of high desulfurization rate was, the faster desulfurization rate decreased, at the same time Mn leaching rate improved and SO42- generation decreased.

Sun et al. [21] investigated the influence of molar ratio of O\(_2\) and SO\(_2\)/(O\(_2\)/SO\(_2\)) on Mn leaching rate. The results showed that O\(_2\)/SO\(_2\), influenced Mn leaching rate and desulfurization efficiency by changing PH, as O\(_2\), and SO\(_2\), could react in the aqueous solution to generated sulfuric acid. The Mn leaching rate and MnSO\(_4\) generation showed a increasing firstly and then decreasing trend with the increasing of O\(_2\)/SO\(_2\). When O\(_2\)/SO\(_2\), was below 15, other Mn series by-product generated. O\(_2\)/SO\(_2\), was above 20, by-product was only MnSO\(_4\), for the flue gas with O\(_2\)/SO\(_2\), above 30 or below 15, PH value can be adjusted by adding rhodochrosite or sulfuric acid to improve Mn leaching rate and the percentage of MnSO\(_4\).

Desulfurization mechanism analysis

Pyrolusite pulp flue gas desulfurization process is a complex system related to gas, liquid and solid three phase mass transfer, chemical reaction in liquid phase, the liquid-solid surface chemical reaction. It contains the redox reaction and the catalytic oxidation process. Zhu determined the main reaction of the desulfurization reaction. It contains the redox reaction and the catalytic oxidation reaction in liquid phase, the liquid-solid surface chemical reaction, the dissolution of MnO\(_2\), and SO\(_2\), oxidation of MnO\(_2\). The main product in absorbing process is MnSO\(_4\), and its K balance constant was 6.84×10\(^{-5}\). The reported mechanism mainly includes the following aspects [23-25]:

\[(1) \quad \text{The dissolution of SO}_2. \quad \text{As flue gas contacts with pulp, SO}_2 \text{immediately dissolves into liquid phase. There is the following balance:} \]

\[
\text{SO}_2 + n \text{H}_2\text{O} = \text{SO}_4^{2-} + n \text{H}_2\text{O} \\
\text{SO}_2 + \text{H}_2\text{O} = \text{H}^+ + \text{SO}_3^{2-} + (n-1) \text{H}_2\text{O} \quad K_w = 1.3 \times 10^{-7} \\
\text{HSO}_3^{-} = \text{H}^+ + \text{SO}_3^{2-} \quad K_w = 6.3 \times 10^{-8} \\
\text{In acidic solution, } S(IV) \text{ mainly shows HSO}_3^{-} \text{ type.} \]

\[(2) \quad \text{MnO}_2 \text{ oxidation of SO}_2. \]

\[
\text{MnO}_2 + \text{SO}_2\text{H}_2\text{O} = \text{MnSO}_4 + \text{H}_2\text{O} \quad K_w = 6.84 \times 10^{-5} \\
\text{MnO}_2 + 2\text{SO}_2\text{H}_2\text{O} = \text{MnSO}_4 + 2\text{H}_2\text{O} \\
\text{SO}_2 \text{ and H}_2\text{O spread to the surface or inside of MnO}_2. \text{Due to H}_2\text{SO}_4 \text{ strong reducibility and MnO}_2 \text{ strong oxidizing; the above reaction can run smoothly. Because of Mn}^{2+} \text{ catalytic oxidation of H}_2\text{SO}_4 \text{ in the existence of O}_2, \text{there will be a sulfuric acid generation. Mn}_2\text{SO}_4 \text{ can be easy decomposed in acid medium or high temperature. It also can occur the following reaction:} \]

\[
\text{Mn}_2\text{SO}_4 \quad \text{MnO}_2 = 2\text{MnSO}_4 \\
\]
Liu thought that in the process of catalytic oxidation of \( \text{SO}_2 \) various metal oxides (MxOy) in pyrolusite pulp were all involved in the reaction [24]. Possible mechanism is as follows:

\[
\begin{align*}
\text{MO}_x + \text{O}_2 & \rightarrow \text{MO}_y \cdot \text{O}_2 \\
\text{MO}_x \cdot \text{O}_2 + \text{HSO}_y & \rightarrow \text{MO}_y \cdot \text{O}_2 \cdot \text{SO}_x \cdot \text{H} \\
\text{MO}_x \cdot \text{O}_2 \cdot \text{SO}_y & \rightarrow \text{MO}_y \cdot \text{O}_2 \cdot \text{SO}_x \cdot \text{H} \\
\text{MO}_x \cdot \text{O}_2 \cdot \text{SO}_y & \rightarrow \text{MO}_y \cdot \text{O}_2 + \text{SOS}_x \\
\text{HSO}_y + \text{S} (\text{IV}) & \rightarrow 2\text{S} (\text{VI}) \end{align*}
\]

(3) Mn, Fe and other transition metal liquid phase catalytic oxidation. \( \text{SO}_2 \) and \( \text{O}_2 \) dissolving in absorbing liquid can occur catalytic oxidation reaction due to the presence of metal ion. W. Pasiuk thought that as chain reaction, metal ions catalytic oxidation of \( \text{SO}_2 \) must have \( \text{H}_2\text{SO}_3 \) to form complex to cause reactions, with Mn(II) and Fe(II) catalyzing [26,27].

Balanced: \( \text{Mn} (\text{II}) + \text{HSO}_3^- \equiv \text{MnHSO}_4^- \)

\( 2\text{Mn} (\text{II}) = \text{Mn}^{2+} \)

**Process equipment**

![Figure 1: The process flow diagram of flue gas desulfurization absorbing liquid to product electrolytic manganese, high purity manganese carbonate and ammonium sulfate.](image)

Chen believed that it was necessary to ensure the material enough residence time. Due to liquid membrane control in the process, a larger fluid and gas purification equipment was chosen as suitable one, such as bubbling tower sieve plate tower, etc. [28]. Zhu investigated the jet bubbling reactor (JBR), and achieved above 90% desulfurization rate [29]. Ren further researched the influence of JBR process parameters on the desulfurization efficiency and Mn leaching rate, and indicated the optimal structural parameters: the diameter of mixer blade 70mm; the height of blade from the reactor bottom 35mm; the porosity of the periphery jet pipe from the reactor bottom 68mm [30]. Wang used veneer sieve plate tower to absorb \( \text{SO}_2 \). The desulfurization rate initially was as high as 80%. When other conditions kept unchanging, the liquid gas ratio improved from 2.5L/m³ to 4.5L/m³, the desulfurization efficiency increased by 13% [31]. Ning dealt with pyrolusite using fire burning to do catalytic oxidation desulfurization [32]. Although the method increased fire burning process, the production rate of \( \text{MnSO}_4 \) as well as the utilization of Mn in pyrolusite improved greatly, and the \( \text{SO}_2 \) removal efficiency could reach 100%. He also did the expanding test in smelter with a foam absorber tower. When the absorbing liquid crystallized once, 95% purity Mn\( \text{SO}_4 \cdot \text{H}_2\text{O} \) was obtained. Sun also opened a new way of flue gas desulfurization with pyrolusite pulp and absorbing liquid reclamation (Figure 1) [33]. He proved that obtaining electrolytic Mn, high purity Mn\( \text{CO}_3 \) and \( (\text{NH}_3)\text{SO}_4 \) in the process of flue gas desulfurization with pyrolusite pulp was feasible and reasonable.

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Combined effect of pyrolusite pulp and microbes

Pyrolusite pulp desulfurization research is more focused on the direct use of pyrolusite pulp to absorb $\text{SO}_2$. As the pyrolusite pulp has surface adsorption, nanometer effect and molecular sieve effect, it can be used as a carrier of microbes. Microbes on the pyrolusite surface participate in the process of sulfur oxidation-reduction, improve the desulfurization efficiency, and are advantage to the sorbent regeneration [34,35].

Dai studied the process of catalytic oxidation of $\text{SO}_2$ by thiobacillus ferroxidans, manganese oxidizing bacteria and pyrolusite pulp. He thought manganese oxidizing bacteria adhered on pyrolusite pulp, and oxidized Mn (II) to Mn(III), Mn(IV), strengthening the effect of pyrolusite pulp catalytic oxidation of $\text{SO}_2$. Manganese oxidizing bacteria promoting desulfurization had an adjustment period, and the followed reinforcement increased with the concentration of bacteria improving. The synergies of thiobacillus ferroxidans and manganese oxidizing bacteria existed and associated with the proportion of microbial composition [36]. Zhang found that the coexistence of pyrolusite pulp and other components had an effect on the desulfurization. Adding bacteria to pyrolusite pulp could restore Fe and Mn ions catalytic activity of $\text{SO}_2$, realizing the catalyst regeneration and keeping high desulfurization efficiency [37]. Wei selected thiobacillus ferroxidans and manganese oxidizing bacteria respectively to investigate the oxidation performance and the recovery of the Mn and Fe ions active performance in the system [38]. When Fe (II) and thiobacillus ferroxidans coexisted, the conversion rate of $\text{SO}_2$ reached 0.0153g/ (L•min), better than the chemical oxidation. He indicated that bacteria in pyrolusite pulp had reinforcement for desulfurization, and could realize Fe and Mn catalyst regeneration.

In addition, Sun investigated NO$_x$ removal from flue gas with pyrolusite slurry. It was found that the removal efficiency was depended strongly on the molar ration of NO$_x$ and NO[39]. He also studied the absorption of mixture of $\text{SO}_2$ and NO$_x$ with pyrolusite slurry, indicating that NO was oxidized into NO$_2$ first by injecting ozone into flue gas stream, and then NO$_2$ was absorbed from flue gas simultaneously with $\text{SO}_2$ by pyrolusite slurry to form MnSO$_4$ and Mn(NO$_3$)$_2$[40,41].

**Phosphate pulp**

Phosphate contains abundant transition metal. In the process of phosphate pulp absorbing $\text{SO}_2$, transition metal ions will continue into the solution, and become cheap liquid catalytic oxidation. Under the condition, $\text{H}_2\text{SO}_4$ is prepared and can decompose phosphate to product $\text{H}_3\text{PO}_4$ and $\text{CaSO}_4$. Fe$_2$O$_3$ was thought to be the main active ingredient in phosphate pulp for desulfurization. When MgO content was over 1% in phosphate, it had adversely effect on the wet production of phosphoric acid and subsequent further process. $\text{SO}_2$ dissolving in water generated sulfuric acid, which could react with MgCO$_3$ in phosphate to produce Mg (HSO$_4$) with high solubility [3]. The residue-concentrate phosphorite, would be obtained after separating and filtering the solution.

**Figure 1:** Diagram of experimental apparatus for aqueous catalyzed oxidation desulfurization by phosphorus ore pulp (1-air pocket; 2-thermostat water bath; 3-absorber; 4- tee valve; 5- drying tube; 6- gas flow counter; 7- air pump; 8- atmosphere sampler; 9- globe valve; 10- absorbing tube with multi-orifice).
and complete phosphate pulp desulfurization technology system. For ten years development, the technology has realized industrial application.

Coal pulp

Coal mine contains FeS₂, which can be oxidized under the normal condition. SO₂ and O₂ in the flue gas can react with FeS₂ to form Fe³⁺, Fe⁴⁺, etc. Some scholars selected coal pulp as the absorbent, to investigate SO₉ catalytic oxidation. According to certain proportion, Sun groups prepared coal pulp with coal mine containing FeS₂ 0.32%-1.5% [44]. In the experiments, SO₂ and O₂ concentration in simulation flue gas was controlled 3% and 20% respectively. The temperature above 40°C, the desulfurization rate could reach more than 99%. They thought that due to the different morphology and surface structure, coal pyrite oxidation efficiency was superior to mineral pyrite. Coal pulp used in liquid catalytic oxidation of SO₂ on the one hand could make the sulfur in pyrite leach, achieving the purpose of lowering sulfur content and improving the quality of the coal; on the other hand could achieved Fe²⁺/Fe³⁺ system by SO₂ and O₂ reacting with coal pyrite without extra catalyst. Flue gas desulfurization with coal pulp as the absorbent, could not only remove SO₂ and dust, but also reduce impurities (sulfur and iron) content in the coal, avoid the traditional method’s problems of high material consumptions and waste residue, which was a kind of environmentally friendly technology, and had a good application prospect.

Discussion and Conclusion

This review has shortly analyzed the state of the art on the pyrolusite pulp, phosphate pulp and coal pulp flue gas desulfurization. The study on pyrolusite pulp flue gas desulfurization has been kept for many years. This paper introduced the main influence factors, desulfurization mechanism analysis and process equipment as well as combined effect of pyrolusite pulp and microbes. The desulfurization process included the dissolution of SO₂; MnO₂ oxidation of SO₂; Mn, Fe and other transition metal liquid phase catalytic oxidation. Microbes in pyrolusite pulp had reinforcement for desulfurization, and could realize Fe and Mn catalyst regeneration. Using the reaction characteristics of pyrolusite pulp and SO₂ can not only do flue gas desulfurization, but also obtain by-products MnSO₄ or dilute sulphuric acid.

Phosphate pulp desulfurization technology has realized industrial application, with 100% SO₂ removal rate for 335min. Fe₃O₄ is the main active ingredient in phosphate pulp for desulfurization. In the process of phosphate pulp absorbing SO₂, phosphorus chemicals and other by-products can be obtained. Coal mine contains Fe₂S₃, which can react with SO₂ and O₂ in the flue gas to form Fe²⁺, Fe³⁺, etc. Coal pulp used in liquid catalytic oxidation of SO₂, not only could make the sulfur in pyrite leach, achieving the purpose of lowering sulfur content and improving the quality of the coal, but also achieved Fe²⁺/Fe³⁺ readily catalytically. There are also various processes in the area of pulp flue gas desulfurization, which need to be further studied. As cheap metal ions liquid catalytic oxidation desulfurization catalyst, pulp has a good application prospect.

Prospective

To summarize the present research situation, the main research directions of this field remained to be further carried out in the future are as follows:

1. In order for better industrial application of pulp flue gas desulfurization, the process mechanisms need a deep understanding. In addition, biotechnology can be combined to cultivate biological species more suitable for the desulfurization.

2. Desulfurization equipment and process need to be further studied. In the published literature, different desulfurization device was used. Each of them has advantages and disadvantages. In addition, the influence factors of reaction, such as the flue gas properties, desulfurization equipment, products outlet, should be considered comprehensively.

3. As a cheap adsorbent, pulp contains a variety of metal oxide and other active ingredients, which can be attempted to use in other fields, such as dealing with greenhouse gas CO₂. As a whole, the technology of pulp flue gas desulfurization is feasible, and can make full use of lean ore, as well as recover sulfur resources. Microbes can keep the activity of the catalyst and achieve continuous catalytic. The selection of the parameters in the process should be considered comprehensively.

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