Review of desulfurization process for biogas purification

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Abstract. Hydrogen sulfide (H₂S) is a toxic and odorous compound present in biogas produced by the anaerobic digestion of biosolids and other organic materials. Elimination of H₂S is necessary as it is extremely hazardous to human health, poisonous to process catalysts and corrosive to equipment. The desulfurization technology is an important part for efficient utilization of biogas. In this paper, the traditional wet and dry desulfurization technology for biogas was reviewed, and the new research progress of biological desulfurization technologies are also introduced.

1. Introduction

The increasing energy crisis is becoming a difficult problem for the sustainable development of mankind, and the demand for renewable energy is becoming more and more urgent. Biogas as a new green energy gradually attracted people's attention. As the target product of the anaerobic fermentation process, biogas will be more and more practical in the practical application, in order to alleviate the energy crisis, protect the natural environment. It is a mixture of gas, generally containing methane (55-80%), carbon dioxide (20-45%), nitrogen (0-10%) and trace amounts of H₂S and other impurities[1].

Hydrogen sulfide (H₂S) is a toxic and harmful gas, it not only threatens the human physical health, but also causes the metal pipes and equipment corrosion in the hot and humid conditions. When burning, H₂S will be oxidized to SO₂, which is one of major atmospheric pollutants[2]. Moreover, H₂S is responsible for the peculiar smell in some processes such as waste water treatment plants, animal processing plants, and food-processing plants.

In order to avoid the aforementioned problems associated with the use of biogas energy, the European Standards stipulates that H₂S in biogas shall not exceed 20 mg/m³. However, the concentration of H₂S in biogas is usually 200 ~ 18000 mg/m³[3], far higher than the provisions of environmental standards. Therefore, in order to achieve biogas environmental protection, high value, efficient use, the H₂S removal of biogas as an indispensable process. The traditional desulfurization technologies for biogas was included the wet desulfurization, dry desulfurization and bio desulfurization.

2. Wet desulfurization

Wet desulfurization is the removal of hydrogen sulfide from a special solvent by countercurrent contact with gas[4]. The solvent is regenerated and then absorbed again. According to the absorption mechanism,
they are divided into chemical absorption method, physical absorption method, physical and chemical absorption method, and wet oxidation method.

2.1 Chemical absorption method
Chemical absorption method is the use of H2S and chemical solvents occurred between the acid and alkali reversible reaction to remove H2S, more suitable for lower operating pressure or raw material gas content of the higher occasions. Commonly, the chemical absorption method mainly includes amine method, carbonate method, ammonia method[5].

The most commonly used amine method is the use of alkyl amines as solvent, and the main solvents used in anhydrous monoethanolamine (MEA), diglycolamine (DGA), diethanolamine (DEA), di-2-propanolamine (DIPA), methyldiethanolamine (MDEA), dimethylethanolamine (DMEA) and so on[6]. The MDEA selective removal of H2S process from natural gas and refinery gas has been used widely. The acid salt method is the first method for removing acid gases such as CO2 and H2S in the gas. It can completely remove COS (carbonyl sulfur), but it is not suitable for gas without CO2 or CO2 very little content. Ammonia is a highly efficient and low energy consumption method[7]. Ammonia is a gas-liquid phase reaction with fast reaction rate and high utilization rate of absorbent. The desulfurization efficiency can be kept from 95% to 99%. The solubility of ammonia in water is more than 20%, but it is more corrosive to the equipment and pollutes the environment.

2.2 Physical absorption method
Physical absorption is the use of different components in a particular solvent solubility to remove H2S and then through the pressure drop and other measures to precipitate H2S, and then the solvent regeneration and recycling. The methods for desulfurization of methane are naphthoquinone absorption method and ammonia method[8].

2.3 Wet oxidation method
Wet oxidation desulfurization is to absorb and oxidize H2S in the gas by the neutral or weak basic solution containing oxidant, so that it can be reduced to elemental sulfur, and the catalyst can be regenerated by using air[9]. Due to the different absorption and catalyst, there are a variety of desulfurization methods, such as arsenic based process, vanadium based process, iron based process and so on. Among them, the principle of iron based process is that hydrogen sulfide is supported by oxygen carrier in alkaline solution, and ferric oxide is catalyzed to oxidize sulfur. The catalyst which is reduced by hydrogen sulfide can be regenerated by air, and Fe2+ is oxidized to Fe3+.

3. Dry desulfurization
Dry desulfurization is the removal of hydrogen sulfide by powder or particle desulfurization agent. The reaction is carried out in a completely dry state, so there will be no corrosion, scaling and other issues. Dry desulfurization is often used to treat gases with lower sulfur content[10]. The commonly used methods are membrane separation[11], molecular sieve[12], pressure swing adsorption (PSA)[13], fixed bed adsorption method[14] and Claus oxidation process[15].

3.1 Membrane separation
Membrane separation is a new separation technique which combines membrane based gas separation with traditional physical adsorption, chemical absorption, cryogenic rectification and cryogenic treatment. Compared with the traditional absorption technology, membrane separation attracts much attention due to its large gas-liquid contact area, high mass transfer rate, no fog entrainment and mild operation conditions. Jansen[16] using membrane absorption device can remove H2S and CO2 when the pressure difference between gas and liquid is 50–500 kPa. Zhao[17] chose polyimide hollow fiber to remove H2S from natural gas. The results show that the desulfurization rate of single grade membrane module is up to 97% when the H2S content is 296 mg /m3.
3.2 Molecular sieve
The molecular sieves have a large surface area with a high degree of localized charge, which allows the molecular sieve to strongly adsorb polar or polarizable compounds such as H$_2$S and other sulfur compounds. There are many studies$^{[18-20]}$ on the adsorption of H$_2$S on molecular sieves. It is generally believed that the hydrogen and hydrogen groups in the sulfur and zeolitic molecular sieves in H$_2$S are hydrogen-bonded. Under certain conditions, the adsorption process is reversible, forming different structures and strength of the complex. Tanada$^{[21]}$ studied the adsorption behavior of two kinds of molecular sieves on hydrogen sulphide. Wang$^{[22]}$ studied the removal of hydrogen sulfide using 4A molecular sieve zeolite and the sulfur sorption capacities of zeolite synthesized under optimum conditions are up to nearly 10 and 15mg/g-sorbent, respectively, and the H$_2$S removal rate is nearly 100%. The adsorption kinetics nonlinear fitting results show that the adsorption system follows Bingham model.

3.3 Fixed bed adsorption method
In many of the fixed bed adsorption method, iron oxide desulfurization method is classic and effective desulfurization method which still widely used in natural gas in the city due to the simple process, easy operation and low energy consumption.

The principle of iron oxide desulfurization is as follows:
Desulfurization reaction:

\[ \text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O} + 3\text{H}_2\text{S} = \text{Fe}_2\text{S}_3 \cdot \text{H}_2\text{O} + 3\text{H}_2\text{O} \]  \hspace{1cm} (1)

\[ \text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O} + 3\text{H}_2\text{S} = 2\text{FeS} + \text{S} + 4\text{H}_2\text{O} \]  \hspace{1cm} (2)

Regeneration reaction:

\[ \text{Fe}_2\text{S}_3 \cdot \text{H}_2\text{O} + \frac{3}{2} \text{O}_2 = \text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O} + 3\text{S} \]  \hspace{1cm} (3)

\[ 2\text{FeS} \cdot \text{H}_2\text{O} + \frac{3}{2} \text{O}_2 = \text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O} + 2\text{S} \]  \hspace{1cm} (4)

The main component of the desulfurizing agent is activated ferric oxide. Xing$^{[23]}$ has pointed out that only the model of α, γ− Fe$_2$O$_3$ is the effective component of desulfurization agents, it usually called the activity of iron oxide. Single iron based desulfurizer has not been widely used because of its low desulfurization efficiency. Now it is gradually replaced by desulfurizer which is compounded with other metal compounds. Cara$^{[24]}$ studied the MCM−41 support for ultrasmall γ−Fe$_2$O$_3$ nanoparticles for H$_2$S removal. The final micrometric (Fe−MCM41−M) and nanometric (Fe−MCM41−N) composites were tested as sorbents for hydrogen sulphide removal at 300 °C and the results were compared with a reference sorbent (commercial unsupported ZnO) and an analogous silica-based sorbent (Fe−SBA15). Dhange$^{[25]}$ studied the regenerable Fe-Mn-ZnO/SiO$_2$ sorbents for room temperature removal of H$_2$S from fuel reformates and the result of sulfur uptake capacity at 25 °C significantly exceeds that of both commercial unsupported ZnO sorbents and un-promoted supported ZnO/SiO$_2$ sorbents. Pinar$^{[26]}$ studied the kinetics of H$_2$S sorption on manganese oxide and Mn-Fe-Cu mixed oxide in a fixed bed reactor and the Mn-Fe-Cu mixed oxide sorbent had a high sulfur retention capacity (0.07 g S/g sorbent). Zeng$^{[27]}$ studied the desulfurization behavior of Fe−Mn-Based regenerable sorbents at high-temperature. The result show that the addition of manganese (Fe/Mn mole ratios less than 8:2) considerably improved the desulfurization efficiency and effective sulfur capacity of the Fe-based sorbents.

3.4 Claus oxidation process
The Claus process is one of the most popular processes used for hydrogen sulfide removal with sulfur recovery on an industrial scale$^{[28]}$. In the first step of the Claus process, H$_2$S is partially oxidized to SO$_2$ with air. The H$_2$S/SO$_2$ mixture is then reacted over a bauxite catalyst to yield elemental sulfur (S$^0$) and water$^{[29]}$. If the mixture of air and hydrogen sulfide is suitable, all hydrogen sulfide can be changed into...
sulfur and water. In the desulfurization process, the direct Claus method, split flow Claus method and direct oxidation Claus method are adopted respectively according to the gas flow rate\(^{30, 31}\). The total recovery of sulfur was 94%–96% by Claus method. In 1988, the Netherlands Comprimo and other companies to develop a super-Claus process, and in Germany Wintershall natural gas purification plant Claus sulfur recovery device on the industrialization of successful trials. The process of operation without dehydration and in selective oxidation, it can be mixed with excess oxygen with no obvious influence on selectivity. The process is simple and the operation is easy. The efficiency of H\(_2\)S oxidation is 85%–95%, and no other side effects occur.

4. **Biological desulfurization technology**

Compared with physical and chemical methods, biological desulfurization process has the advantages of mild desulfurization conditions, high removal rate, no chemical catalyst, low energy consumption, no two pollution, and can generate sulfur recovery resource\(^{32, 33}\). The process of biogas desulfurization can be divided into three stages: (1) the dissolution process of H\(_2\)S gas, according to the theory of two-film theory from the gas phase by gas-liquid double membrane transfer to the liquid phase; (2) the dissolved H\(_2\)S enters the desulfurization bacteria through the cell membrane; (3) the intracellular H\(_2\)S is converted and utilized to remove H\(_2\)S from methane\(^{34}\). Sulfur bacteria can be divided into two groups: colored sulfur bacteria and colorless sulfur bacteria. The photosynthetic pigments in colored sulfur bacteria can be used for photosynthesis, and the main types are photosynthetic bacteria desulfurization bacteria. Colorless sulfur bacteria cannot photosynthesis because of no photosynthetic pigment, mainly for autotrophic bacteria desulfurization bacteria.

4.1 **Photoautotrophy**

In the presence of light, CO\(_2\) and inorganic nutrients, light autotrophic desulfurization bacteria can use CO\(_2\) to synthesize new cellular substances while H\(_2\)S was oxidized into elemental sulfur or sulfate. The reaction process is as follows\(^{33}\):

\[
2nH_2S + nCO_2 \rightarrow 2nS^0 + n(CH_2O) + nH_2O
\]  
\[
nH_2S + 2nCO_2 + 2nH_2O \rightarrow nSO_4^{2-} + 2nH^+ + n(CH_2O)
\]

In order to oxidize H\(_2\)S to elemental sulfur instead of sulfate, the sulfur pollution is eliminated and the sulfur resource is recovered, in addition to controlling the concentration of H\(_2\)S, the light intensity is also important. Larsen\(^{35}\) proposed that when light intensity and sulfide flow rate were adjusted to a point on the curve (balanced loading), all of the sulfide introduced to the reactor was oxidized to elemental sulfur without the formation of sulfate. But the low treatment load and longer reaction time of the autotrophic desulfurization bacteria have limited the practical application.

4.2 **Chemoautotrophy**

The autotrophic desulfurization bacteria were not limited by light source, and CO\(_2\) was used as carbon source. The energy released during oxidation of H\(_2\)S can be used by cells to immobilize CO\(_2\) for growth. So the chemoautotrophy desulfurization bacteria are the most widely used desulfurization bacteria and the major electron acceptor is O\(_2\) or NO\(_3^-\). The study show that the concentration of sulfide and sulfur and nitrogen ratio (S\(^2-\)/NO\(_3^-\)) is key factor of whether the product is sulfur\(^{36}\). However, the sulfur oxygen ratio (H\(_2\)S/O\(_2\)) is an important parameter to achieve the product sulfur in aerobic environment\(^{37}\).

At present, the research on simultaneous removal of H\(_2\)S from wastewater and NO\(_3^-\)/NO\(_2^-\) in wastewater has just started\(^{38-42}\). Using anaerobic autotrophic denitrification process, the desulfurization of methane and the removal of nitrogen from wastewater are coupled together to removal of sulfur and nitrogen. The other is the introduction of air or oxygen into the biogas desulfurization reactor and achieve aerobic desulfurization of biogas by desulfurization bacteria\(^{37, 43-48}\).

At present, biological desulfurization process has not formed a certain scale of industrial applications. There are two main reasons: the culture of microorganisms is restricted by the composition of biological...
populations and environmental factors.; the research of microbial desulfurization technology and equipment is lagging behind. Biochemical control is difficult, which restricts the development of biological desulfurization technology.

5. Conclusion
At present, the traditional desulfurization technologies including dry desulfurization and wet desulfurization are more frequently used, but there are some problems such as high desulfurization costs and easy to produce two pollution problems. With the increasingly stringent environmental laws and regulations, the development of efficient, low input, recycling and no two pollution technology has become the mainstream of desulfurization technology development. As a new desulfurization technology, biological desulfurization has attracted more and more attention because of its advantages of low energy consumption and no two pollution. From the desulfurization equipment and technology, the bio desulfurization still remained in the stage of laboratory research. In order to obtain large-scale applications, it is necessary to make efforts in the fields of desulfurization bacteria, bioreactor and desulfurization process.

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