Removal of Hydrogen Sulfide (H₂S) from Biogas Using Zero-Valent Iron

Muhammad Rashed Al Mamun and Shuichi Torii

Abstract—In this study, 90% purity zero-valent iron selected as hydrogen sulfide adsorbent was introduced to a biogas-tobiomethane generation. The average H₂S concentration of 211, 138 and 139 ppm was introduced into the chemical H₂S elimination system. The both tests result showed that the H₂Sconcentration could reach below 50 ppm which acceptable for running in internal combustion engines. In this process the removal of H₂S depends on the use of the removal substances and pH. The effect of pH on the H₂S removal with Fe^o is attributed to the formation of FeS through the precipitation of Fe(II) and sulfide. The maximum absorption efficiency was obtained 95% at pH 6 for Fe^o, which are higher than conventional adsorbents. The H₂S removal using zero-valent iron reduced high operation cost and risk factor to the process. Therefore, it is still highly recommended not only for preventing metal corrosion but also prevents the environmental pollution.

Index Terms—Anaerobic digestion, biogas, purification, zero-valent iron (Fe^o), hydrogen sulfide (H₂S).

I. INTRODUCTION

Anaerobic digestion of solid biomass is capable of methane-rich biogas and producing reducing the environmental impact caused by the biowastes. Biogas is the combustible gas produced through a biological process, known as anaerobic digestion which is the process operated at low-high temperature ranging from 30-65°C [1] and without air. The nature of the raw materials and the operational conditions used during anaerobic digestion, determine the chemical composition of the biogas [2]. Biogas consists of 55-80% CH₄, 20-45% CO₂, 5-10% H₂ with trace amount of H₂S and other impurities [3]. In these compositions, we can see that the combustible components of biogas are CH₄ and H₂. Other gases are useless, toxic or harmful and have no energy contribution in biogas. Also, among these two gases only CH4 is present in a significant amount. With its physical and chemical properties close to those of natural gas, albeit with a lower methane content more than 50%, biogas can be used to boil water or for cooking instead of LPG, kerosene, charcoal, fire wood [4] used as fuel for process heating, used for lighting purpose, produce electricity via internal combustion engine or used in replacement of diesel or gasoline to drive equipment. On the other hand, the Kyoto Protocol has explicitly defined methane as one of the six key greenhouse gases where the global warming potential of methane is 25 times higher than

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The authors are with the Department of Advanced Mechanical System Engineering, Graduate School of Science and Technology, Kumamoto University, 2-39-1 Kurokami, Kumamoto 860-8555 Japan (e-mail: rashedshahi@gmail.com). that of carbon dioxide [5]. Thus, the use of methane for power generation can not only provide energy but also reduce the methane emission. Sources for biogas production cover a wide range of feedstocks including animal wastes, household wastes, crop residues, sewage sludge, wastewater and landfill [6]. Therefore, not only wastes can be disposed of in appropriate ways to help solve the environmental pollution problems such as foul smell and flies, the process of biogas production will also provide a cheap and renewable energy source while conserving fossil resources such as natural gas, oil or coal. Nowadays, the small biogas production system which refers to the biogas digester with the size ranging from 12 to 100 m³ has become popular renewable energy technologies to reduce energy consumption and emissions in developing countries [7].

With the typical composition of biogas described earlier, the biogas can be readily used with engine in term of the heating content. However, the H₂S in biogas, which is found to range between as low as about 50–10,000 ppm depending on the feed material composition to the digester [8], can cause corrosion to the engine and metal parts via emission of SO₂ from combustion, especially when the engine is not operated continuously, as well as toxic H₂S/SO₂ concentrations in the workplace. Users have shown little interest in applying biogas for power production because hydrogen sulfide has detrimental effects on the cast iron and steel used equipment. Moreover, the failure to eliminate the corrosive and toxic H₂S in raw biogas has been the drawback of many biogas applications. Therefore, H₂S must be removed prior to further utilization.

Common H₂S removal technologies for H₂S removal from biogas fall into one of the following: (1) absorption into a liquid either water or caustic solution; (2) adsorption on a solid such as iron oxide based materials, activated carbon or impregnated activated carbon and (3) biological conversion by which sulfur compounds are converted into elemental sulfur by sulfide oxidizing microorganisms with addition of air/oxygen. Each technology has its own advantages and disadvantages. Additions of air (2-6%) to the digester headspace result in H₂S in the biogas oxidized into sulfur. This method shows promise as partial H₂S removal, but have limited operational histories. Moreover, care should be taken to avoid overdosing of air, as biogas in air is explosive in the range of 6-12%, depending on the CH₄ content [9]. Liquid based and membrane processes require significantly higher capital, energy and media costs, although regeneration is possible. Commercial biological processes for H₂S removal are also available and claimed to effectively reduce operating, chemical, and energy costs, but require higher capital costs than dry based processes [10]. Absorption by water or basic solution will cause wastewater problem, if non regenerable or not treated well [6]. Adsorption on solids such as activated carbon, iron hydroxide or oxide added high operation cost to the system. Among these technologies, adsorption with high adsorption capacity is recognized to be an energy efficient technology for hydrogen sulfide removal. Because of nanotechnology providing high surface areas of nanomaterials, developing a nanoscale adsorbent with high adsorption capacity becomes promising for hydrogen sulfide removal [11], [12].

The zero-valent iron (Fe^o) represents as a nanomaterial that is very important nanotechnology of the environmental remediation that has been developed since 1996 [13]. The Fe^o with a particle size of 1–100 nm has been implemented in the field remediation of contaminated groundwater by direct injection technology [14]-[16]. Extensive studies have indicated the ability of Fe^o to remove a wide variety of environmental contaminants including PCBs, chlorinated aliphatic and aromatic hydrocarbons, chlorinated pesticides, heavy metals, and inorganic ions (e.g., nitrate and perchlorate) [12], [13], [17]–[19]. It has been reported that Fe^o has an adsorption capacity towards arsenic ranging from 9 to 174 times greater than conventional Fe^o because of its relatively high specific surface areas [20]. The high adsorption capacity of Fe^o has been documented for the control of malodorous sulfide containing compounds generated in biosolids [11], [12].

The H_2S removal technologies employed in large or industrial scale biogas plants include chemical absorption, aeration and bio-filter [21]; however, for small size biogas plants, there are almost no H_2S removal units in practice. This is mainly due to the lack of awareness of the H_2S toxicity as well as the lack of information of the H_2S removal technologies. In this work, we present here the use of zero-valent iron (Fe^o) for effective removal of hydrogen sulfide from the laboratory scale anaerobically digested solid wastes. The objectives of this study include (1) achieving the hydrogen sulfide concentration below 50 ppm by Fe^o, (2) exploring the efficiency of Fe^o for sulfide removal at a lab scale, and (3) examining the potential impact of Fe^o on biogas upgradation in term of CH₄concentration.

II. MATERIALS AND METHOD

A. Collection of Fermentation Slurries

The vegetable and fruit waste were collected from kokai vegetable market, kumamoto, Japan. Cafeteria waste was collected from the cafeteria of Kumamoto University. The collected materials was stored at 4°C and used for the experiment.

B. Batch Experiment

A laboratory scale plant was assembled to produce and purification of biogas. It consists a digester, collection chamber, gas purification unit and gas storage system. A schematic for the whole setup is illustrated in Fig. 1. The 200 liter polypropylene tank (used as a reactor) with an air tight rubber gasket was connected to the purification unit. Cafeteria, vegetable and fruit wastes were mixed in equal proportion after blending to obtain a homogenous mixture and were diluted with tap water at the ratio of 1:1. In order to optimize gas production, feed stock was maintained 8% of TS concentration by mass diluting with water. The prepared raw materials were used as input charge for the production of biogas in the digester tank. About 80% of the digester volume was filled with the diluted organic waste. For better digestion pH was maintained between 6.8 to 7.6 by applying (CaO) in the digester. Mesophilic batch digestion was conducted at (33 ± 2) °C temperature less than 35 days hydraulic retention time (HRT). Inside the digester biogas was generated from anaerobic decomposition of the input sample by the action of various microorganisms and bacteria.



Fig. 1. Schematic diagram of H₂S removal from biogas.

C. Chemicals and Materials

For the laboratory study, all chemicals were reagent grade or above and used without further purification. Purity (as metallic iron) of Zero-valent iron (Fe[°]) is minimum 90%, concentration of hydrochloric acid (HCl) (35-37%) and ammonium hydroxide (NH₄OH) (28-30%) were employed in the present investigation. However, commercial grade chemicals, purchased from KANTO CHEMICAL CO., INC., Japan, were applied to the test in the lab.

D. H₂S Removal Unit Set Up

For purification, we intended to enrich the methane content in the biogas by removing undesired H_2S . The spontaneous pressure and constant flow rate (2.5 L min⁻¹) of biogas produced inside the digester was utilized to flow the gas through zero-valent iron filled U tubes. The setup consists of two liter glass flask connected through flow control valves. The 1st flask was filled by zero-valent iron solution, whereas the 2nd and 3rd one was filled by biogas which was flowed inside before starting the purification process. The solution contains Feº of varied quantity in repeated experiments (from 1g to 10g each) and pH ranges (6-7.8) per liter of water for the removal of H_2S was examined. The solution pH was adjusted at the beginning of the reaction using 1M HCl or NH₄OH and monitored for each sample throughout the experiment. Under batch type operational conditions, the biogas is introduced at the bottom of the absorber flask as small bubbles, passing through the Fe^o solution for proper mixing in each observation. The adsorbent unit which contained two flasks is interconnected by 13 mm diameter and 1.5 meter long Ushaped hose pipe. For the removal of H₂S the hose pipe filled with Fe°. The experiments were operated to keep H₂S- concentration of treated biogas below 50 ppm, which is well below the maximum concentration of H_2S acceptable for running in internal combustion engines [9]. Finally, the upgraded gas is stored in a gas bag for sampling. Gas bag was chose for its advantage to be completely evacuated refilling for different observations. Biogas was sampled before and after it flowed through these chemical and the removal efficiency was determined as the percentage of H_2S removed from the biogas of each samples.

E. Analytical Methods

Gas composition was analyzed by using a gas chromatography (GC-8AIT/C-R8A SHIMADZU Corporation, JAPAN). The instrument was fitted with a Porapak N 80/100, 274.32 cm, 1/8 mesh 250×250×145 mm column, a molecular sieve (Molesieve 5 A 60/80, 182.88 cm, 1/8), column oven maximum temperature 399 [°C], temperature stability ± 0.1 [°C] a stainless-steel column and a thermal conductivity detector. Detector type was TCD made by Tungsten rhenium filament. Maximum temperature and sensitivity of the detector was 400 [$^{\circ}$ C] and 7000 [mVml mg⁻¹] respectively. Argon (Ar) was used as the carrier gas at a flow rate of 30 ml/min. The column temperature was 60°C and the injector temperature was 80°C, with current 60 [mA]. The lowering of H₂S-concentration and CH₄ enrichment were determined by analyzing the data obtained from gas chromatographic measurement. Gas flow rates were regulated using a gas flow meter. The pressure was higher than atmospheric level measured by gas pressure gauge. The pH of the liquid solvent was measured using a pH meter. All measurements were repeated at least three times.

III. RESULTS AND DISCUSSION

A. H₂S Adsorption Tests

All the tests were conducted with the controlled flow rate of biogas directly from the digesters at 2.5 L min⁻¹. The H₂S adsorption capacities by the zero-valent iron (Fe^o) used in the study were compared with various amounts. Fig. 2 represents the resulting curves for the H₂S-concentration in the inlet biogas comparing the exit biogas with amount of zero-valent iron. With the average H₂S-concentration in raw biogas during the operation was 138 ppm, which gradually decreased below 50 ppm after treating with increasing the amount of the zero-valent iron inside the U tube, which is well below the maximum concentration of H₂S acceptable for running in internal combustion engines [9]. The H₂S content is the minimum 16%, when the amount of the zerovalent iron was 10 g. Also the variation of H₂S removal efficiency over the study period is presented in Fig. 2. The solid zero-valent iron performed above 85% removal efficiency at 10 g. At 1 g, the zero-valent irons removed 32% of the H₂S, and then keep the increasing trends with the amount of zero-valent iron which was not significantly different from others. Increasing amount to 1 g to 10 g had significantly positive effects on H₂S removal efficiency from 32% to 88%. Fig. 3 shows that the CH₄-concentration enrichment trends from inlet to exit biogas. Very small variances were observed on upgradation with respect to variable mass. The concentration of CH_4 increases after treating with increased of zero-valent iron and the highest amount of methane 77% was possible to avail by using 10 g of zero-valent iron. It is clear that when Fe^o is increasing CH_4 -concentration also increases due to decreased of H₂S. In this study described that Fe^o has a potentiality for removing of H₂S from biogas.



Fig. 2. H₂S-concentration profile in biogas and % removal efficiency with Fe $^{\circ}$.



Fig. 3. Comparison of CH₄-concentration profile in biogas (i) before and (ii) after purification.

B. H_2S Absorption Tests without pH Study

The selected zero-valent iron was further tested along with water at variable mass of H₂S removing phase Fe^o. The purpose of these tests was to determine the effectiveness of the zero-valent iron solution with varied concentration to remove H₂S from raw biogas. As shown in Fig. 4, the average H₂S-concentration was 139 ppm was introduced into the chemical solvent, after absorption this concentration gradually decreased based on amount by up to 10 ppm at 10 g. The H₂S-concentration is lowered to the permitted limit of standard gas grid. With respect to absorption capacity of the solvents the maximum amount of H₂S absorbed by these solutions in 128 ppm H₂S per 10 g zero-valent iron. The removal efficiencies of zero-valent iron significantly increase at concentrations lower than 50 ppm H₂S. The estimated amounts 1 g and 10 g of solution was showed in batch mode operation experiments, above 34 % and 92% of the H_2S can be removed respectively. This means that increases an amount of zero-valent iron with an appropriate ratio of contacting phases, it is possible to achieve above 90% removal of H₂S. Fig. 5 shows that the CH₄concentration enrichment trend from raw gas to purified gas is depicted. The result also shows that the relative amount of CH₄ increased with proportional to the increasing concentration of zero-valent iron solution. The concentration of CH₄ at treated biogas is 62% and 78% at 1 g and 10 g. However, the highest percentage of methane 78% was possible to avail by using 10 g of zero-valent iron due to H₂S is physically absorbed in water and then, by the use of a Fe^o solution.



Fig. 4. H₂S-concentration profile in biogas and % removal efficiency with $Fe^{\circ}.$



Fig. 5. Comparison of CH_4 -concentration profile in biogas (i) before and (ii) after purification.

C. H₂S Absorption Tests with pH Study

Finally, the effectiveness of zero-valent iron for hydrogen sulfide removal under various pH conditions is illustrated in Fig. 6. The average concentration of H₂S, 211 ppm was passed in the Fe^0 solution. The result shows that the H₂Sconcentration was gradually increased based on pH. With respect to absorption capacity of the solvents the maximum amount of H₂S absorbed by these solutions in 201 ppm H₂S at pH 6. Test performed with Fe⁰ and pH indicated levels of H₂S, 10 ppm in the outlet gas with high levels of CH₄. Approximately 95% of H₂S-concentration was removed at pH 6. However, the H₂S removal efficiency decreased as pH increased. The blue line represents the measured average inlet inlet CH₄-concentration of the biogas, while the green line is the CH₄-concentration of the upgraded biogas, measured with respect to variable pH are graphed in Fig. 7. The average 64% methane content biogas was passed in this sytem. The zero-valent iron study with pH decreased methane concentration from 80% to 62% at pH 6 to pH 7.8. The oxidation of Fe^{0} leading to generation of Fe(II) is favorable under acidic conditions:

$$\operatorname{Fe}^{0}{}_{(s)} + 2\operatorname{H}_{2}\operatorname{O} \rightarrow \operatorname{Fe}^{2+} + \operatorname{H}_{2}{}_{(g)} + 2\operatorname{OH}^{-}$$
 (2)

Sulfide readily reacted with Fe(II) to form iron sulfide

(FeS) [22], [23]:

$$Fe^{2+} + H_2S \rightarrow FeS + 2H^+$$
(3)

$$\operatorname{Fe}^{2+} + \operatorname{HS}^{-} \to \operatorname{FeS} + \operatorname{H}^{+}$$
 (4)

Accordingly, the pH effect on the H_2S removal with Fe⁰ may be attributed to the formation of FeS through the precipitation of Fe(II) and sulfide. The Fe(II) concentrations in the reaction system containing Fe⁰ and sulfide at various pH values the Fe(II) concentration increased as pH decreased. This is in a good agreement with the theory of FeS formation.



Fig. 6. Effect of P^H on H₂S-concentration profile in biogas and % removal efficiency with Fe^o.



Fig. 7. Comparison of CH₄-concentration profile in biogas (i) before and (ii) after purification.

IV. CONCLUSIONS

This study paid high attention to the biogas utilization in small size farms as well as industrial applications. Underutilization of the produced biogas results in the unused biogas released to the atmosphere. Moreover, there are almost no H₂S removal units in practice, mainly due to the lack of awareness of the H₂S toxicity as well as the lack of information of the H₂S removal technologies. In this study, we have applied Fe⁰ for dissolved H₂S removal from biogas which performed a laboratory test in anaerobically digested solid wastes. The test results revealed that Fe⁰ is an effective reagent for H₂S removal in acidic conditions; however, with the average inlet H₂S concentration of about 211,138 and 138.9 ppm to the adsorption and absorption unit respectively, the both tests showed that the H_2S concentration could reach below 50 ppm which acceptable for running in internal combustion engines. The H₂S removal using zero-valent iron reduced high operation cost and risk factor to the system. It is still highly recommended not only for preventing engine corrosion but also for the environment benefit in which air pollution by H_2S/SO_2 emission and impact on human health could be potentially reduced.

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