

Desulfurization for Biogas Generated by Lab Anaerobic Digestion unit

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Abstract: The presence of hydrogen sulfide can represent an important limitation for the biogas utilization. The paper is an examination of the two biogas desulfurization methods; one is the adsorption on iron oxide (Fe_2O_3) fixed bed filter and the other is the addition of iron chlorides ($FeCl_3$) directly to the digester feedstock that is capable of removing H_2S from a gas stream without the uptake of CO_2 . The disadvantage of filter media (fixed bed) placed in the path of the biogas is that most solid iron oxide can be regenerated through exposure to air with heating to form elemental sulfur and over time the media will become clogged with elemental sulfur and must be replaced. Laboratory experiments have been carried out using anaerobic digestion system model FH6 (from Germany) to investigate the effect of adding $FeCl_3$ on the anaerobic digestion process. The results show that In-situ sulfide abatement by dosing low concentration of ($FeCl_3$) directly to the digester slurry or in a pre-storage tank, H_2S levels can be reduced from (2000-200) ppm. The obtained results showed that $FeCl_3$ negatively impacted the anaerobic digestion process by reducing the volume of produced biogas. Fe-dosed sludge produced 20% less biogas. While $FeCl_3$ has no impact on the anaerobic digestion process when dosing to the pre-storage tank.

Keywords: Biogas, Hydrogen Sulfide Removal and Iron Oxide Filter

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I. Introduction

Biogas is a digester gas arising from the activity of anaerobic bacteria which decompose organic matter. Its composition depends on the type of raw material subjected to the digestion process and on the method of conducting this process and is as follows. Methane CH_4 (50–75%), carbon dioxide CO_2 (25–45%), hydrogen sulfide H_2S (0-1%), hydrogen H_2 (0-1%), carbon monoxide CO (0–2%), nitrogen N_2 (0–2%), ammonia NH_3 (0-1%), oxygen O_2 (0–2%), and water H_2O (2–7%) (De Graaf and Fendler, 2010).

The Combustion of biogas containing H_2S produces sulfur dioxide (SO_2). When SO_2 combines with water vapor it produces sulfuric acid that corrodes the exhaust pipes of burners, gas lamps and engines (Muche and Zimmerman 1985). Thus, removal of H_2S is highly recommended.

Sulfur is an essential nutrient for methanogenesis during the anaerobic digestion process, but excessive sulfur levels too high may limit biogas production (Chynoweth 1987). Sulfur can enter the digester in several pathways, such as farm animals consume sulfur in their food source, mostly in the form of sulfur containing amino acids such as Cystine and Methionine, or from their drinking water source, which may contain sulfates (Zicari 2003). Additionally, farm animals excrete sulfur that is not digested for nutrition in the manure, which is then fed to the digester.

Previously studies about hydrogen sulfide adsorption at low temperatures have been searched. According to Rodriguez *et al* (1998)2, who have studied some metal oxides, is the reactivity order inverse proportional towards the band gap. The studied oxides have a reactivity order as follow:



α - Fe_2O_3 , which has a high theoretical adsorption maximum (0.6g H_2S /g Fe_2O_3), has been studied by (Davydov *et al.* 1998). Various techniques have been developed over the years for the removal of H_2S . Dry-based iron-oxide product come in prepackaged cylindrical units that are recommended for small and medium anaerobic digestion plant. Several commercial dry iron oxide materials are available including SulfaTreat®, Sulfure-Rite®, Media-G2®, and SulfaMaster®. They consist of Fe_2O_3 and Fe_3O_4 compounds coated onto a support material and operate in a low-pressure vessel with a down-flow of gas (Kohl and Neilsen 1997). The material has shown the ability to reduce H_2S concentrations from 30,000 ppm to below detectable levels (SulfaMaster, 2010).

Iron salts were used at wastewater treatment plants for several reasons: for removing chemical phosphorus, preventing from struvite formation and reducing the content of hydrogen sulfide (H_2S) in biogas

(Mamais *et al.* 1994) and one of the existing methods of avoiding the formation of struvite is adding FeCl_3 salts that could affect the anaerobic digestion process.

Also, the effect of adding iron salts on anaerobic digestion was studied by many researchers (Smith and Carliell-Marquet, 2008; 2009) the majority of which reported a negative effect of dosing iron salts on a daily production of biogas comparing to un-dosed sludge (Svetlana, 2011).

McFarland and Jewell studied the effects of digester pH and the addition of iron phosphate directly to the digester. Their research suggests that increasing the digester pH from (6.7 to 8.2) through the addition of phosphate buffers reduced the H_2S concentration in the biogas from (2,900 -100) ppm. This pH adjustment increases the soluble sulfide concentrations in the digester from (18 - 61) mg/l. If soluble sulfide levels reach 120 mg/l or more, CH_4 production is inhibited. As with the addition of iron chloride to the digester, this method for reducing H_2S in the biogas must be used with another removal technology in order to bring H_2S levels down to (4) ppm or less, making the gas suitable for natural gas pipeline injection (McFarland and Jewell, 1989).

The present study is undertaken to determine any possible effect of the iron oxide solid filter placed in the path of the biogas and direct iron chloride dosing on the H_2S gas concentration in the digester itself. The sulfide either reacts with metal ions to form an insoluble metal sulfide or is oxidized to elementary sulfur. This method is effective as a partial removal process for removing H_2S from the biogas stream but must be used in conjunction with another technology for further H_2S removal if the biogas is to be injected into the natural gas pipeline.

II. Materials And Methods

Materials and Instruments:

The following materials/instruments were used for the purpose of this research: AR grade sodium hydroxide, Iron Chloride, Iron Oxide, sodium hydrogen carbonate, ammonium chloride (NH_4Cl) or sodium hydrogen phosphate (NaH_2PO_4) and acetic acid were used as procured without further purification.

The Mini Digester (Model FH6) is used for laboratory tests (Fig. 1, left). It is purchased from the Beher Company, Germany. It comprises of six gas cells. Each cell consists of a reaction vessel (500 ml fermenter) and a well-closed gas pipe. The gas pipe - eudiometer is of 350 ml size and contains the confining liquid. It is connected to the leveling vessel with a solution. The biogas produced in fermenters supplants the confining liquid in the gas pipe into the outside leveling vessel of 750 ml volume. The gas produced is read on the gas pipe. The fermenters are connected with the glass gas pipe and submerged into the water with constant temperature $35\text{ }^\circ\text{C}$. Figure (1) shows (Mini Digester) for biogas production for laboratory purposes and other instruments like; Weighing balance, a portable gas detector for measuring gas composition (CH_4 , CO_2 , H_2S and O_2), pH meter, oven, grinding mill, and biogas burner fabricated locally for checking gas flammability. The exact composition of produced biogas is determined by the gas detector (Fig. 2).

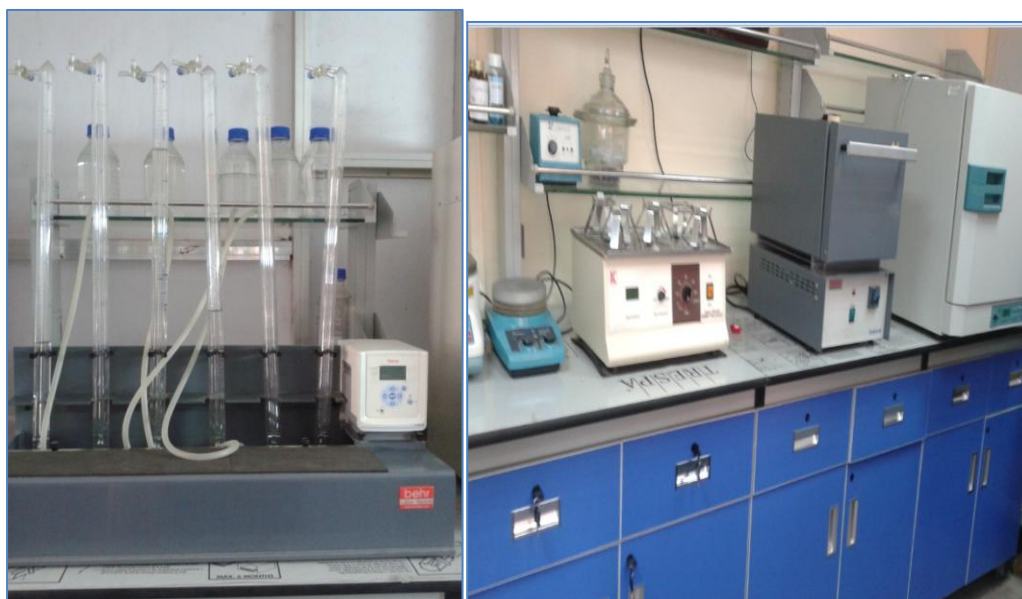


Figure 1. Some Lab instruments used for the purpose of this research



Figure 2. Commercial fixed bed column after passing raw biogas (Left); H₂S gas analyzer connect to collected biogas bag (Right).

Analytical methods:

The used Farmyard manure typically consists of manure mixed with the beddings (straw, wood shavings...). The straw absorbs the manure in dry matter contents ranging from 10 to 30% (Monnet 2003). Cattle dung is the easiest feedstock to use for a biogas plant because it already contains the right bacteria and it has been ground up by the animal’s teeth and broken down chemically by acids and enzymes in the animal’s gut.

There are several measurements that can be made to define the properties of the feedstock or the slurry :

1. Total solids and volatile solids analysis (Sluiter *et al.* 2008): The measurement of total solids involves placing a weighed sample in an oven and drying it for several hours at 105°C. The sample should be kept in a high-temperature glazed ceramic container and care should be taken that the dried sample does not reabsorb moisture from the atmosphere before it is weighed. The proportion of total solids in the sample is given by the weight of the dried sample divided by the weight of the wet original. The volatile solids content is measured by heating the dried sample at 500 or 600°C for several hours in a furnace and weighing the residue. The heating of animal dung to such high temperatures causes it to burn, so the furnace should be sited where the obnoxious smoke given off does not give offense. The volatile solids proportion is the difference between the weight of the dried sample before and after combustion, divided by its weight before.
2. pH measurement with an accurate instrument pH meter
3. Carbon to nitrogen ratio (C: N) is an important parameter as anaerobic bacteria need nitrogen compounds to grow and multiply. Too much nitrogen, however, can inhibit methanogenic activity (Dioha et al 2013). The fresh cow manure was analyzed with respect to total solids and volatile solid content, total nitrogen content, pH, and others. The results are tabulated in Table 1.

Table 1. Some cow manure properties as biogas –producing material

Total solids, T.S.%	Volatile solid (% of T.S.)	Carbon (C) %	Nitrogen (N) %	C/N	pH	gas yield ^a [NI (kg VS ⁻¹)]	H2O%
16-20	77	35-40	3-4	20-30	6.95	208.2	72-85

a NI = Norm litre (273 K, 1.013 bar). Notes: Data for fresh dung. All dung will lose water and nitrogen on keeping, especially if air humidity is low.

Test Procedure:

Series of tests were carried out in a water bath at 38 °C. Reaction vessels, were filled with a great quantity of substrate (Table 2), only the current thinning ratio varied. The test comprised two check samples of the cow slurry inoculum producing the minimum biogas quantity, followed by three series of tests with three replications from which the biogas yield from Iron (II) Oxide was evident. Additional equipment: heating pump, digital thermometer, and barometer, well-covered fermentor in a water bath are of key significance for the successful process of biogas production.

Table 2. Recommended ratio for execution of test

Masses of inoculum and trial inoculum in mixture; in gram			
Test No.	Inoculum sample	Inoculum	Mixture
0	0	400	400
1	15	385	400
2	20	380	400

After 20 days, usually, a very low gas formation is observed. On each reading of the gas volume in the eudiometer tube, the temperature and the air pressure are determined so that the gas volume can be re-calculated into normal conditions. The level of the confining liquid is to be adjusted, too, depending on the gas formation, after each individual reading or after several readings with open eudiometer cock, supposing that the air must not enter into the tapping cock. In many cases, the established volume of the gas formed is enough large.

III. Results And Discussion

At the beginning of the test, the residual dry matter and the organic matter of the inoculum and digester feedstock have been determined. The typical values for some several properties of the feedstock are given in (Table 1). These results depend very much on the size of the animal, what it is eating, the weather and etc. Hot dry weather will cause water to evaporate from the dung before it is collected, giving an apparent increase in TS, while the humid weather will have the opposite effect.

The total solid content of cow dung varies between (15–20) percent while the recommended value for slurry is between (8–12) percent. This means that dung must be diluted with water before it is used in a biogas vessel because a low solids concentration mean that the digester volume is used inefficiently and it can also lead to separation of the slurry, the heavier solids sinking to the bottom to form a sludge layer and the lighter solids floating to form a scum layer on top of the liquid (supernatant). The scum layer can dry out to form a solid mat, preventing the gas release from the liquid and blocking rubber tubes. This should not happen if the total solid (TS) of the slurry is kept above about six per cent. (Boe K., 2006; Verma,2002)

A slurry with a high solids concentration (greater than 12%) does not easily flow through the inlet tube. The volatile solid content (VS) of dung is usually around (80%) of the total solids (Table 1). The remaining ash (fixed solids) is composed of soil particles, inert portions of vegetable matter (some grasses, e.g. rice, concentrate silica in their stalks) and some solid carbon left from the decomposition of foodstuffs. Therefore,(VS) is not an ideal measure of the digestibility of a feedstock.

In general, the C: N ratio of dung from cattle fed with poor feeds, such as straw and dry grass, tends to be too high (up to 35 per cent). If the C: N is high, then gas production can be enhanced by adding nitrogen in the form of cattle urine or urea, or by fitting a latrine to the plant. If the C: N ratio is low, the addition of carbon, such as chopped grass can reduce the possibility of toxicity from too much nitrogen affecting the bacteria (Chandra K. 2005).

Prior to the beginning of test, the pH value of the inoculum was adjusted to (7-8 pH), with simultaneous adding of sodium hydrogen carbonate; the inoculum was tempered to about 38 °C. In order to be more accurate, adjusting the C: N: P ratio of the mass to about 100: 6:1 is required. This is effected by adding ammonium chloride (NH₄Cl) or sodium hydrogen phosphate (NaH₂PO₄). Additional tests depend on specific problems and on the manner of initial processing of the sample.

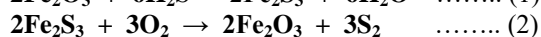
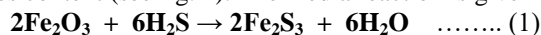
According to Table (1), the test comprised two check samples of the cow slurry producing [208.2 NI, (kg VS⁻¹)] biogas quantity at (273 K and 1.013 bars), followed by three series of tests with three replications from which the biogas yield from Iron (II) Oxide fixed bed filter was evident. The range corresponds well with our experiments that gave biogas yields of [208 – 268 NI (kg VS)⁻¹]. This result is fitted with Balsari work that most of the biodegradable carbon in cattle feed is already digested in the rumen and in the gut. Thus, cattle manure has a lower potential to produce biogas than poultry manure therefore CH₄ concentration in the biogas is lower (Balsari, *et. al.*, 1983).

Requirements for H₂S removal for biogas vary depending on the biogas utilization technology. H₂S levels below 1000 ppm are necessary for use in boilers to produce heat. Levels less than 250 ppm are necessary to avoid excessive corrosion and expensive deterioration of lubrication oil (Weiland 2010). The H₂S limit for electricity production by internal combustion engines is 100 ppm (Zicari, 2003).

The result formed after the combustion in which H₂S is involved will yield sulfur oxide that will corrode the metal component and cause the lubricant oil to become acidic. Therefore, to avoid the damage caused by H₂S, it must be eliminated or at least be reduced in the system(Deublein, 2008).

In the literatures; Biogas purification methods can be classified into two generic categories: those involving physicochemical phenomena (reactive or non-reactive absorption, reactive or non-reactive adsorption) and those involving biological processes (H₂S biodegradation by microorganisms to give less harmful forms) (Abatzoglou and Boivin, 2009).

In our work, the first physicochemical method to control the H₂S gas content in biogas used Iron oxide fix bed filter. This H₂S removal filter media is placed in the path of the biogas that reacts with the corrosive gasses content (see fig. 2). The media reaction is given in reactions(1 and 2):



The theoretical stoichiometry of reaction (2) reflects in following equation (1):

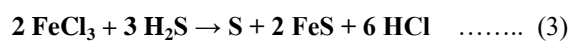
$$\frac{mw\ 6H_2S}{mw\ 2Fe_2O_3} = 0.6403 \frac{kgH_2S}{kgFe_2O_3}$$

Reductions of H₂S concentrations in the biogas down to 200 – 100 ppm have been achieved. The primary disadvantage of this absorptive media is that the media needs to be replaced (Fig.2) or recharged after a certain period of time.



Figure 3. Iron Oxide filter media before and after passing raw biogas.

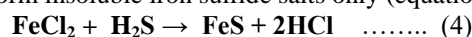
The second method practiced removing H₂S gas called (In-situ sulfide removal). The method based on the addition of dissolved ferric chlorides (FeCl₃) in water directly into the digester slurry or into the feed substrate in a pre-storage tank. It reacted with the produced hydrogen sulfide and form insoluble iron sulfide salts and sulfur, (Reaction 3).



The Dosage ratio used in this method was (4:1.0) ppm FeCl₂ to ppm H₂S (in solution) which reflects theoretical stoichiometry in following equation (2):

$$\frac{mw\ 3H_2S}{mw\ 2FeCl_3} = 0.3151 \frac{kgH_2S}{kgFeCl_3}$$

The third method practiced removing H₂S gas based on the addition of dissolved ferrous chlorides (FeCl₂) in water directly into the digester slurry or into the feed substrate in a pre-storage tank. It reacted with the produced hydrogen sulfide and form insoluble iron sulfide salts only (equation 4).



The theoretical stoichiometry of reaction (4) reflects in following equation(3):

$$\frac{mw\ H_2S}{mw\ FeCl_2} = 0.2689 \frac{kgH_2S}{kgFeCl_2}$$

As seen from Equation 1; (1.0 kg) of Fe₂O₃ stoichiometrically removes (0.64 kg) of H₂S and from Equation 2; (1.0 kg) of FeCl₃ removes (0.3151 kg) of H₂S, thus decreasing the removal of H₂S by (0.3249) kg per kg of ferric materials.

Equation 3, shows that (1.0 kg) of FeCl₂ removes (0.2689 kg) of H₂S thus, decreasing the removal of H₂S by (0.3711) kg per kg of ferrous chloride materials.

The fourth possibility to control the contents of biogas being released is buffering pH in the digester. Different pH levels may destroy enzymes or alter the chemical equilibriums of bioreactions within the digestion process (Pesta, 2006). Increasing the reactor pH from 6.7 to 8.9 will decrease the sulfide production from 2900 ppm to 100 ppm (McFarland and Jewell, 1989). However, increasing the pH increases the concentration of free ammonia which is inhibitory to methanogenesis. Low pH encourages the release of hydrogen sulfide and high pH discourages it, as seen in the table (3). So, we conclude from the table that it is possible to control the odor by raising the percentage of (pH) to reduce the concentration of hydrogen sulfide

Table 3. Percentage of hydrogen sulfide removal with pH adjustments.

pH	4	6	7.2	7.8	8.2	8.6
% H₂S	90	80	45	16	8.0	3.5

Before discussion, the H₂S mechanism; We must confirm that the microbiology of anaerobic transformation of organic wastes is a process which involves many different bacterial species, such as hydrolytic, acid forming, acetogenic, and methanogenic bacteria which produce CO₂ and CH₄ as the main products of the digestion process. Many different groups of bacteria within the anaerobic digester often compete for the same substrate and electron acceptor (Fig. 3). Methane is produced by methane-forming bacteria and a variety of acids and alcohols are produced by sulfate reducing bacteria. Hydrogen is used with sulfate (SO₄⁻²) by sulfate-reducing bacteria and hydrogen sulfide (H₂S) is produced (Shah et al 2014).

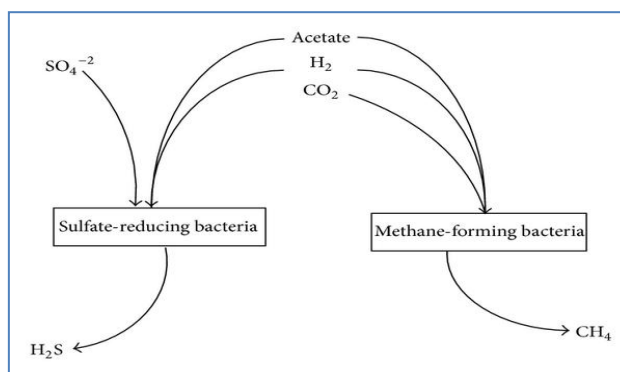
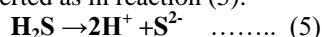


Figure 3. Compete between methane-forming bacteria and sulfate-reducing bacteria for producing methane (H₂S) or hydrogen sulfide (H₂S), (Shah et al 2014).

The H₂S mechanism as a result of the sulfates and other oxidized compounds of sulfur is easily reduced to sulfide under the conditions prevalent in anaerobic digesters. Sulfides require special attention in case of all anaerobic processes as they can lead to a lot of corrosion and other problems. Sometimes high sulfates may come from the original water supplies of the city. The sulfates so removed are converted to sulfides and also sulfides are partly converted to H₂S (Krishna, 2013). Refer to Fig. 3; The reduction of SO₄²⁻ is accomplished by strict anaerobic organisms. These organisms are only able to use a limited number of electron donors, e.g. particularly hydrogen. The occurrence of sulfate reduction is important for the following two aspects; The reduction of sulfate will result in the formation of H₂S, which is toxic for methanogens. In the neutral pH range, approximately 50% of the dissolved sulfide is present in the undissociated (volatile) form (H₂S). The formation of sulfide may cause considerable malodor nuisance to the environment.

Ref. to Table (3); Depending on the pH and dissociation constant at given temperature, the unionized H₂S can be converted as in reaction (5):



The free or unionized H₂S fraction at pH 7.0 to 7.4 may be about 40% or more can then reduce gas production and the corresponding economy of the system also diminished. The dissolved (S²⁻) still remaining in the liquid phase goes out in the effluent as liquid fertilizer. Therefore, we aerated the effluent to convert residual sulfides to sulfates as stated in reaction (6):

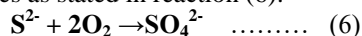


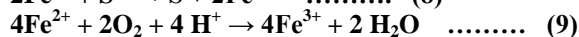
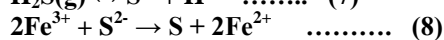
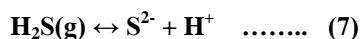
Table (3) summarized the results in this paper of Packed column, absorbent solution and pH which were used to remove H₂S contaminants from a biogas stream.

Table 3. The applied Biogas purification methods in our research

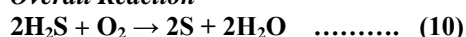
Type of H ₂ S removal methods	Biogas production*	Selected experimental doses of Fe salt [Fe (III), g/L]
Iron oxide fix bed filter	No impact on biogas Increase CH ₄ content from 65% to 85% H ₂ S reduced (2000-200ppm)	
Adding Fe salt to feed the sludge mixture,	decreased by about 20–25% 50%	0.84 (min) 1.68 (max)
pH	6.90 control .58 Fe-dosed sludge (min) .34 Fe-dosed sludge (max)	
Adding Fe salt to feed the sludge mixture, pre-treatment tank	decreased by about 20–25%, 50%	

* compare to biogas production from un-dosed sludge

The results show that In-situ sulfide removal by dosing low concentration of (FeCl₃) directly to the digester slurry or in a pre-storage tank, H₂S levels can be reduced from (2000-200) ppm. The obtained results showed that FeCl₃ negatively impacted the anaerobic digestion process by reducing the volume of produced biogas. Fe-dosed sludge produced 20% less biogas. While FeCl₃ has no impact on the anaerobic digestion process when dosing to the pre-storage tank. FeCl₃ has been shown to provide the largest pH reduction of the liquid fraction. Mechanism of H₂S oxidation on (Fe³⁺) catalyst and the regeneration can be shown in following reactions (Nagl, 1997). The overall reaction of H₂S degradation and elemental sulfur (S) formation can be written as the reaction (10):



Overall Reaction



IV. Conclusions

Methods which can be used to control sulfides concentrations in order to reduce hydrogen sulfide concentration are:

- a. Add an adsorption substance as fixed bed filter placed in the path of the biogas.
- b. Dilute the feed to below the sulfide threshold value; and/or
- c. Add chemicals to form a non-toxic complex or insoluble precipitate;

The first two methods may be straightforward in some cases but not practical in others. The third method has been demonstrated using iron salts addition. Since iron is the most soluble of the heavy metal sulfides, its presence causes the precipitation of other metals.

Results indicated that biogas generation was not affected by the addition of Fe as a trace metal. However, it affected others such as CH₄, CO₂, and H₂S content in biogas, the degradation rate of total and volatile solid, and alkalinity of fermentor liquid. With the increasing of Fe concentration in the fermentor, produced CH₄ in biogas and degradation rate of total and volatile solid decreased, but CO₂ increased. This could be due to the concentration of Fe was too high so that it became toxic for the micro organism and lowered their performance in degrading organic compounds.

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