Reaktor

p-ISSN 0852 – 0798
e-ISSN 2407 – 5973

Acredited: SK No.: 60/E/KPT/2016
Website : http://ejournal.undip.ac.id/index.php/reaktor/

Reaktor, Vol. 18 No. 3, September Year 2018, pp. 160-165

Hydrogen Sulfide Removal from Biogas Using Digester Effluent Absorbent in a Continuous Vertical Column

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(Received: January 01, 2018 ; Accepted: September 01, 2018)

Abstract

Gas-Liquid mass transfer is one of the most commonly used phenomena in the chemical process, such as absorption. This research evaluates the value of liquid phase mass transfer coefficient of hydrogen sulfide removal from biogas in a continuous contactor column for digester effluent-biogas system. This study was carried out by continuously contacting biogas at certain flow rate with digester effluent under counter-current mode in a packed bed column. Samples were taken after steady state condition was achieved. This research used raschig rings, large balls, and small balls as packing materials with surface areas of 0.9269, 0.6279, and 0.2992 m^2/m^3*, respectively at volumetric flow rates of biogas from 0.1109 to 0.8846* m^3/h*. The results show that the relationship between the studied variables and the mass transfer coefficient can be written as dimensionless number according to the following empirical equation* $\frac{k_L a\cdot d_t^2}{D_L}=5.653\cdot[A_s\cdot d_t]^{0.6171}\left[\frac{V_g}{d_t\cdot D_L}\right]^{0.7514}$*. This model is valid in the range of* $(A_s\cdot dt)$ *and* $(V_g/dt\cdot D_L)$ *respectively from 0.03 to 0.09 and from 237,267.08 to 3,307,522.67. The average error is 17.85%.*

Keywords: *biogas; digester effluent; hydrogen sulfide; mass transfer coefficient; packed bed column*

How to Cite This Article: Pranolo, S.H., Paryanto, Margono, Rizaldy, B., and Yansah, H., (2018), Hydrogen Sulfide Removal from Biogas Using Digester Effluent Absorbent in a Continuous Vertical Column, Reaktor, 18(3), 160-165, http://dx.doi.org/10.14710/ reaktor.18.3.160-165

INTRODUCTION

There has been a steady increase in energy consumption along with the increase in world population. Although the development of alternative and renewable energy sources has been continuously promoted, the flexibility of fossil fuels still makes these fuels as the primary choices of the community. In fact, demands of fossil fuels reached 88% of the total energy consumption. One of the current trends is the utilization of organic waste (biomass) as an alternative source of fuel. Biogas is a type of fuel obtained from organic waste treatments (Dolejs *et al.*, 2014).

Palm Oil Mill Effluent (POME) or palm oil mill's liquid waste is one of the agro-industrial wastes that may cause water an environmental pollution. POME has a high concentration of biological oxygen demand (BOD) and chemical oxygen demand (COD) and dark brown in colour (Table 1). So far, it is only limited in the reduction of BOD and COD concentration to qualify the maximum level according to Regulation of the Minister of Environment Number 3 of 2010 concerning Wastewater Quality Standards for Industrial Estate (BOD = 50 mg/L and COD = 100 mg/L) before being discharged into river body. In regard to its high organic substances contents, POME is highly potential to be used as raw material for biogas production through anaerobic digestion process.

Table 1. POME characteristics (Madaki and Seng, 2013)

No.	Parameter	Composition (mg/L)
1.	Biological oxygen demand (BOD)	10,250 – 43,750
2.	Chemical oxygen demand (COD)	15,000 – 100,000
3.	Total solids (TS)	11,500 – 79,000
4.	Suspended solids (SS)	5,000 – 54,000
5.	Total volatile solids (VS)	9,000 – 72,000
6.	Ammoniacal-nitrogen (NH_3-N)	4 – 80
7.	Total nitrogen (N)	180 – 1,400

The anaerobic digestion process is a fermentation process of organic matters by the activity of anaerobic bacteria, which converts the suspended forms into water soluble materials and biogas. The anaerobic digestion comprises of a number of stages, namely hydrolysis, acidogenesis, acetogenesis, and methanogenesis and performed on mesophilic conditions (30-37ºC) and thermophilic (50-60ºC). The commonly used digesters are continuous stirred tank reactor (CSTR), batch reactor, semi-continuous reactor, and batch sequencing reactor (Ji *et al*., 2013). It was reported in the literature that biogas from POME has 55-70% methane (CH_4), 30-45% carbon dioxide (CO_2), and small amounts of nitrogen (N_2) and hydrogen sulfide (H_2S).

Hydrogen sulfide (H_2S) is one of the undesirable impurities in biogas because it can turns into sulfur dioxide (SO_2) and sulfuric acid (H_2SO_4) during the combustion process. These compounds are corrosive and can potentially damage the equipment of a power plant (Kennedy *et al*., 2015). In addition, the H_2S content in biogas reduces the calorific value of biogas that decreases the combustion efficiency. So, the biogas requires purification before being used as fuel.

There are several methods to remove sulfuric compounds from biogas. Lin *et al*. (2013) developed a biological method of H_2S removal from biogas, which is oxidation with $FeSO_4$ solution followed by microbial oxidation. The result is very promising since more than 90% of the H_2S can be removed. However, this method is only suitable for purification of biogas with high level of H_2S (> 1%). Ramírez *et al.* (2013) also studied the biological purification of biogas. He concerned in the anoxic single-stage processes for H_2S removal from biogas. Depending on the H_2S concentration, an aerobic biological process requires a stoichiometric oxygen level, which is approximately 5%. It is crucial to control the oxygen concentration in the digestion system to avoid explosion. The results showed that pall rings can be used as a packing material in an anoxic biotrickling filter to remove H_2S from biogas without a serious reduction in methane concentration, and it could reached removal efficiency of 99% with low pressure drop.

One of the other most dominant methods used for biogas purification is water scrubbing. This method is relatively simple and low cost. Biogas is continuously passed to the bottom of the bubble column through a diffuser and produce bubbles. Since the solubility of H_2S in water is higher than methane, water will absorb H_2S and H_2S is separated from biogas. Unfortunately, the simultaneous absorption of H_2S and CO_2 into the water can cause fouling and clogging on the piping system, and the absorption of H_2S is only 50%. Thus, it is better to get rid of H_2S separately (Persson *et al*., 2006).

Liu *et al*. (2015) developed a method using TiO_2/zeolite composite as an absorbent medium followed by photocatalysis. The result is quite promising with the absorption of H_2S is 72-87%. However, this method is complicated for commercialization on an industrial scale. On the other hand, Micoli *et al.* (2014) investigated the use of modified zeolites loaded by Cu or Zn and activated carbons on biogas purification process. These materials showed very interesting performances in a previous work for HCl removal from the gas phase. The advantage is the use of the same materials for the elimination of both HCl and sulfur compounds in a single-step adsorption process for biogas purification, which reduce the production cost. The best result of this research is absorption of biogas 2.46 mmol/g absorbent at breakthrough time 1.222 minutes. Onthong *et al.* (2017), also proposed the use of metal as adsorbent named literite, a composite structured from Fe_2O_3. The efficiency of H_2S removal from biogas was high enough that is close to 91.67%.

Kennedy *et al.* (2015) shows that the effluent of the digester can be used as a good absorbent for H_2S removal from biogas. NH_3 separation and aeration as pretreatment is performed to increase the effluent pH. Biogas is contacted with this effluent in a bubble column reactor. It was reported that the H_2S absorption percentage is 84%.

This research uses vertical column (absorber) as the equipment to facilitate mass transfer process of H_2S from biogas to the effluent. Vertical column was selected on the basis that the contacted substances are liquid and gas (Ortiz *et al*., 2014). This research uses different effluent, that is the effluent of biogas production from POME which is different with effluent digester from livestock waste. The absorption system used a fixed bed absorber column made of stainless steel with a counter-current contact process. The purpose of this research is to develop a mathematical model of H_2S absorption process to obtain the mass transfer coefficient value of H_2S from biogasto digester effluent. This coefficient can further be used in the design of gas-liquid fixed bed contactor either in pilot plant scale and industrial scale.

Absorption is a mass transfer from gas to liquid. The gas input stream contains absorbable components, while the absorbent liquid is insoluble to the gas phase. Figure 1 shows an absorption scheme of mass transfer from the main body of gas to the gas film layer, mass transfer through the interface layer to the second phase, and mass transfer from the liquid film layer to the main body of the liquid.

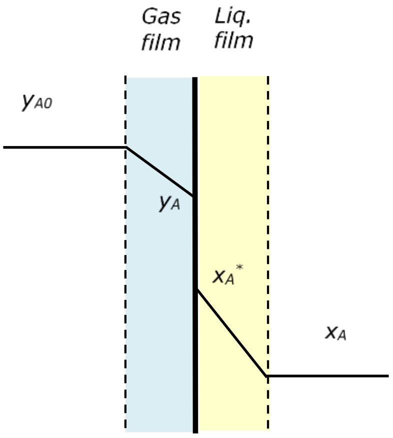

Figure 1. H_2S mass transfer scheme from gas phase to liquid phase

The evaluation of mass transfer coefficient employs the mass balance analysis in the volume element of A. Δz, as shown in Figure 2.

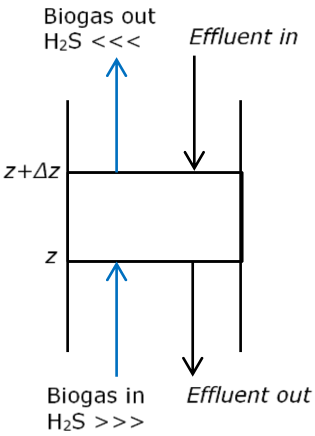

Figure 2. Volume element review

Mass balance for the volume element of *Δz* thickness:

$$\textit{input} - \textit{output} + \textit{transfer} = \textit{accumulation} \tag{1}$$

$$V_g \cdot y_A\Big|_z - V_g \cdot y_A\Big|_{z+\Delta z} + k_L a \cdot \frac{1}{\beta \cdot L_s} \cdot A_t \cdot \Delta z \cdot \left(x_A - x_A{}^*\right) = 0 \tag{2}$$

Divided by*Δz* and taken the limit with *Δz*→0:

$$\lim_{\Delta z \to 0} \frac{V_g \cdot \left(y_A|_{z+\Delta z} - y_A|_z\right)}{\Delta z} = k_L a \cdot \frac{1}{\beta \cdot L_s} \cdot A_t \cdot \left(x_A - x_A{}^*\right) \tag{3}$$

$$V_g \cdot \frac{dy_A}{dz} = k_L a \cdot \frac{1}{\beta \cdot L_s} \cdot A_t \cdot \left(x_A - x_A{}^*\right) \tag{4}$$

$$V_g \cdot \int dy_A = k_L a \cdot \frac{1}{\beta \cdot L_s} \cdot A_t \cdot \int \left(x_A - x_A{}^*\right) \cdot dz \tag{5}$$

$$V_g \cdot \left(y_A - y_{Ao}\right) = k_L a \cdot \frac{1}{\beta \cdot L_s} \cdot A_t \cdot \left(x_A - x_A{}^*\right) \cdot \Delta z \tag{6}$$

So,

$$k_L a = \frac{V_g \cdot \beta \cdot L_s \cdot \left(y_A - y_{Ao}\right)}{A_t \cdot \left(x_A - x_A{}^*\right) \cdot \Delta z} \tag{7}$$

At the inter-phase, the mass transfer resistance only exists at the main body of each phase. In the interface area, there is no resistance so that the concentration y_A and x_A^* are in equilibrium condition. In the interface, y_A and x_A^* are connected by a distribution coefficient called the Henry constant for the gas-liquid system:

$$y_A = H \cdot x_A^* \tag{8}$$

with *H*=0.389 obtained by approach using Henry constant value for waste water and air system at 298.15 K (Rumsey and Aneja, 2014).

This research involves partner, PLTBg Rantau Sakti, a biogas based power generating company located in Rokan Hulu, Riau. Biogas production uses palm oil mill effluent (POME) from crude palm oil plant around there. Biogas production process still produce liquid waste (effluent digester). There are 33,197 m^3 of effluent produced per day (data from June-July 2015). So far, the effluent digester is treated only by aeration before being discharged into the environment. The purifying process of biogas in PLTBg Rantau Sakti currently uses water scrubbing method that is the absorption of impurities in biogas using water. The organic content in the effluent digester is still high (BOD and COD is still above the minimum limit), so it is potentially re-utilized. Preliminary trials showed that pH of the effluent decrease after contacted with biogas, indicates absorption of hydrogen sulfide. This research focuses on hydrogen sulfide absorption method using the effluent digester, so that the mass transfer coefficient (k_La) of hydrogen sulfide from biogas into digester effluent can be obtained.

MATERIALS AND METHODS

The main ingredients of this research are digester effluent from POME digester and biogas. Digester effluent taken from PLTBg unit in Rantau Sakti, North Tambusai Subdistrict, Rokan Hulu District, Riau. Biogas is taken from the biogas production unit in Ringinwok, Jagoan Village, Sambi Subdistrict, Boyolali District, Central Java. The H_2S content in biogas feed is 0.038%. This research uses auxiliary materials that is calcium chloride ($CaCl_2$) as moisture content absorbent in biogas (water scrubber).

The main equipment are water scrubber from calcium chloride, packed bed column (diameter: 10 cm, height: 50 cm), effluent tank (volume: 1 L), and pretreated effluent tank (volume: 1 L). The instruments are another manometer for measuring biogas input pressure, biogas meter with volumetric flow rate measurement range of biogas from 0.04 to 6.00 m^3/hour, and hydrogen sulfide detector with measuring range of H_2S from 0 ppm to 500 ppm. The set of tools can be seen in Figure 3.

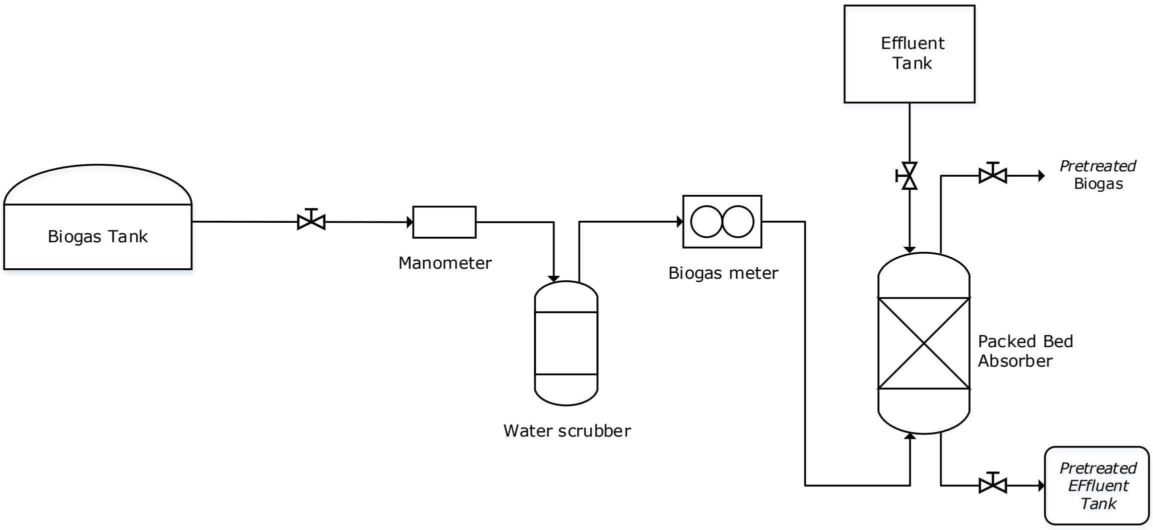

Figure 3. Process flow diagram of H_2S removal from biogas

The hydrogen sulfide removal process began with the filling of the effluent tank as much as 1 liter. Then the effluent digester valve was fully open and the biogas valve was then open based on variations of the biogas flow rate. Contact time and the volume of biogas flowing was recorded during the process for the calculation of the biogas volumetric flow rate. Pretreated effluent and biogas from column output samples were taken for H_2S content analysis. H_2S content in biogas analysis was done by using hydrogen sulfide detector and H_2S in pretreated effluent analysis was done by spectrophotometry method. The data obtained are used for calculation of k_La value based on mass balance analysis using Equation (7).

This research using contact surface area (represented by the type of packing) and biogas volumetric flow rate (arranged with valve and read with biogas meter) variations. The packing used were raschig ring with contact area 0.9269 m^2/m^3, ball with contact area 0.2992 m^2/m^3, and ball with contact area 0.6279 m^2/m^3. The biogas volumetric flow rate applied for the raschig ring packing was from 0.1822 to 0.6579 m^3/h; small ball packing from 0.1109 m^3/h to 0.2016 m^3/h; and large ball packing from 0.1896 m^3/h to 1.9170 m^3/h, with four variations of biogas flow rate for each packing material.

RESULTS AND DISCUSSIONS

The mass transfer coefficient is the diffusion constant associated with the mass transfer rate (mass flux), mass transfer area, and concentration gradient as the driving force. Mass transfer rate is proportional to the driving force and mass transfer coefficient (k_La). The bigger $k_La$value the mass transfer rate will be greater. Thus, the value of k_La indirectly shows the rate of absorption (Treybal, 1981).

The data of H_2S mass transfer coefficients from biogas to effluent digester for various contact surface area and volumetric flow rate of biogas according to mass balance calculation in Eq. (7) are presented in Table 2. The table shows that the greater the contact surface area and biogas volumetric flow rate, the greater the mass transfer coefficient. This is due to increasing of biogas flow rate, then the transferred substance will be greater, consequently the mass transfer coefficient is greater. The results show that the calculation k_La from mass balance equation has been appropriate with the theory of mass transfer. The next step is modeling the mass transfer coefficient in a generalized equation, in order to be used for scaling up this equipment.

Table 2. The k_La value for various A_s and V_g

A_s (m^2/m^3)	V_g(m^3/s)	k_La (s^{-1})
0.9269	0.000183	0.0045
	0.000115	0.0041
	0.000062	0.0033
	0.000051	0.0028
0.2992	0.000056	0.0015
	0.000047	0.0013
	0.000038	0.0013
	0.000031	0.0019
0.6279	0.000533	0.0113
	0.000288	0.0082
	0.000118	0.0050
	0.000053	0.0032

Empirical Equations of Mass Transfer Coefficients

The relationship between experimental variables and k_La is modeled in a form of dimensionless numbers equation. The dimensionless equation does not depend on the geometric scale, so it can simply be used for scale up. Analysis of dimension was completed by Buckingham method. The variables influencing the k_La value in the absorption process are: specific contact surface area (A_s, m^2/m^3), solution density (ρ_L, kg/m^3), viscosity (μ_L, kg/m.s), vessel diameter (d_t, m), volumetric flow rate of biogas (V_g, m^3/s), volumetric flow rate of solution (V_L, m^3/s), and diffusivity of solution (D_L, m^2/s). The diffusion process occurs in the mass transfer of a substance in which the mass of substance should move from higher concentration to lower concentration. Mass transfer can occur in both gas and liquid phase. Diffusion process ends when the equilibrium state between two phases has been reached. Diffusion rate of a compound depends on the diffusivity coefficient. Since the physical properties of digester effluent are similar to water, the diffusivity coefficient of hydrogen sulfide in digester effluent is approximated by the diffusivity coefficient of H_2S in water. According to Perry and Green (2008), the diffusivity value (D_L) of hydrogen sulfide in water is 0,161 m^2/s. The relationship between those variables is expressed by the equation:

$$k_La = K \cdot {A_s}^{C_1} \cdot {\rho_L}^{C_2} \cdot {\mu_L}^{C_3} \cdot {d_t}^{C_4} \cdot {V_g}^{C_5} \cdot {V_L}^{C_6} \cdot {D_L}^{C_7} \qquad (9)$$

Result of Buckingham method:

$$\frac{k_La \cdot {d_t}^2}{D_L} = K \cdot [A_s \cdot d_t]^{C_1} \cdot \left[\frac{\mu_L}{\rho_L \cdot D_L}\right]^{C_3} \cdot \left[\frac{V_g}{d_t \cdot D_L}\right]^{C_5} \cdot \left[\frac{V_L}{d_t \cdot D_L}\right]^{C_6} \qquad (10)$$

since $\left[\frac{\mu_L}{\rho_L \cdot D_L}\right]$ and $\left[\frac{V_L}{d_t \cdot D_L}\right]$ are constant, so:

$$K \cdot \left[\frac{\mu_L}{\rho_L \cdot D_L}\right] \cdot \left[\frac{\mu_L}{\rho_L \cdot D_L}\right] = K' \qquad (11)$$

$$\frac{k_La \cdot {d_t}^2}{D_L} = K' \cdot [A_s \cdot d_t]^{C_1} \cdot \left[\frac{V_g}{d_t \cdot D_L}\right]^{C_5} \qquad (12)$$

The values of the constants K', C_1, andC_2 are evaluated by multivariable linear regression method to obtain the empirical equation of k_La in Equation (12). The value of k_La is evaluated using Equation (12) so Table 3 was obtained, with average error of 17.85% towards the experimental data of k_La.

$$N_{Sh} = \frac{k_La \cdot {d_t}^2}{D_L} = 5.653 \cdot [A_s \cdot d_t]^{0.6171} \left[\frac{V_g}{d_t \cdot D_L}\right]^{0.7514} \qquad (13)$$

Tabel 3. Calculation result of k_La in the form of Sherwood Number

$A_s \cdot d_t$	$\frac{V_g}{d_t \cdot D_L}$	$\frac{k_La \cdot d_t^2}{D_L}$
0.09	1.135.105.72	0.000183
0.09	715.110.89	0.000115
0.09	387.512.12	0.000062
0.09	314.365.01	0.000051
0.03	347.826.09	0.000056
0.03	290.683.23	0.000047
0.03	237.267.08	0.000038
0.03	191.285.38	0.000031
0.06	3.307.522.67	0.000533
0.06	1.791.874.63	0.000288
0.06	731.902.35	0.000118
0.06	327.063.89	0.000053

The Relationship between Experimental Variables and Mass Transfer Coefficient (k_La)

The experimental results as listed in Table 3 show that the greater the contact surface area (expressed in $A_s.dt$ dimensionless group), the mass transfer coefficient value (expressed in Sherwood Number $k_La.d_t^2/D_L$) is also greater. The effect of contact surface area (A_s) can be expressed by the equation:

$$k_La \sim {A_s}^{0.6171} \qquad (14)$$

according to the theory of mass transfer that the mass transfer coefficient is proportional to the contact surface area.

Shah *et al*. (1982) investigated the effect of volumetric flow rates of gas on the liquid phase mass transfer coefficients on a bubble reactor using water. The result is expressed in the equation:

$$k_La = 0.467{V_g}^{0.82} \qquad (15)$$

This research used variation of biogas volumetric flow rate in range from 0.1109 m^3/h to 0.8846 m^3/hr. The experimental results show the same trend with the results of Shah *et al*. (1982) that the greater the flow rate of biogas then the mass transfer coefficient is greater. The relationship between the biogas flow rate with the mass transfer coefficient in this research is expressed by the equation:

$$k_La \sim {V_g}^{0.7514} \qquad (16)$$

Equation (13) shows the value of order in the dimensionless group $V_g/d_t.D_L$(C_5 = 0.7514) is greater than the order in the dimensionless group A_s/d_t^2 (C_1 = 0.6171). Thus, it can be concluded that the biogas volumetric flow rate (V_g) is more influence to k_La than the contact surface area (A_s).

The value of K' consists of the constant of the dimensionless equation (K) and the dimensionless groups which are the physical properties of the material and the parameters which the values were kept constant during the experiment. The formation of empirical equation by Buckingham method gives a value of K' of 5.653 as the calculation result of equation:

$$K' = K \cdot \left[\frac{\mu_L}{\rho_L \cdot D_L}\right] \cdot \left[\frac{V_L}{d_t \cdot D_L}\right] \qquad (17)$$

CONCLUSIONS

The experimental results showed that the greater the contact surface area (A_s) results in the greater the mass transfer coefficient (k_La). The trend is also valid for volumetric flow rate of biogas (V_g). The relationship between the contact surface area and volumetric flow rate of biogas with the mass transfer coefficient in the digester effluent–biogas solution system gives the following empirical model:

$$N_{Sh} = \frac{k_La \cdot {d_t}^2}{D_L} = 5.653 \cdot [A_s \cdot d_t]^{0.6171} \left[\frac{V_g}{d_t \cdot D_L}\right]^{0.7514}$$

This model is valid for the range of (*As.dt*) and (*Vg/dt.DL*) respectively from 0.03 to 0.09 and from 237,267.08 to 3,307,522.67. Average error is 17.85%.

ACKNOWLEDGEMENTS

This research received funding from the Fundamental Research Grant of PNBP UNS (Batch-1) corresponding to Letter of Contract No. 623/UN27.21/PP/2017. The digester effluent is taken from PLTBg Unit at Rantau Sakti Village, North Tambusai Subdistrict, Rokan Hulu Regency, Riau.

This research is partially conducted in biogas production unit owned by Mr. Sulistyo in Ringinwok, Jagoan Village, Sambi Subdistrict, Boyolali Regency, Central Java.

NOTATION

β : Sprecific Contact Surface Area (m^2/m^3)
A_s : Specific surface area of packing, m^2/m^3
A_t : sectional area of the vessel, m^2
D_L : Diffusivity of Hydrogen Sulfide in water, m^2/s
D_L= 0,161 (Perry and Green, 2008)
d_t : Vessel diameter, *m*
H : Henry's Constant
k_La : Liquid phase mass transfer coefficient, s^{-1}
L_s : Height of packing, *m*
N_{Sh} : Sherwood Number
t : Contact duration (residence time), *s*
V_g : Volumetric flow rate of biogas, m^3/s
V_L : Volumetric flow rate of effluent, m^3/s
x_A : Mol fraction in liquid phase
x_A^* : Mol fraction in liquid phase atinterface
y_A : Mol fraction in gas phase
y_{A0} : Mol fraction in feed gas
z : Packed bed height, *m*
μ : Viscosity, *kg/m.s*
ρ : Density, kg/m^3

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