EFFECTS OF TEMPERATURE-pH ON LIQUID PHASE MASS TRANSFER AND DIFFUSION COEFFICIENTS AT LEACHATE TREATMENT IN ANAEROBIC BIOREACTOR

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ARTICLE INFO	ABSTRACT
	Leachate contains complex dissolved organic and inorganic substrates that are biodegradable and non-biodegradable. Principally, anaerobic treatment utilizes anaerobic bacteria to degrade the dissolved organic matters. Anaerobic treatment is very sensitive towards the change of temperature and pH. This research used an anaerobic bioreactor with volume of 160L, with a ratio of leachate:biogas was 70:30. Seeding, acclimatization and leachate treatment were executed at temperature 35°C; pH ambient, temperature 45°C; pH ambient, temperature 35°C; pH 7.2 and temperature 45°C; pH 8.0. Based on this research, that there is dependency on mass of solutes organic substrate (COD) in the leachate, at all operating conditions of leachate treatment in anaerobic bioreactor. Hence, the organic substrate concentration (COD) will affect the VFA, the liquid phase mass transfer and diffusion of solute organic. Consequently, the higher the temperature-pH, the higher the liquid-phase mass transfer, but lower diffusion coefficients is.

INTRODUCTION

Leachate

Leachate is liquid waste arising as a result of external water, which enters into a trash heap, then it dissolves and rinses the dissolved and suspended organic and inorganic matter in the garbage, including complex organic matter as a result of the process of physical, biological, and chemical decomposition (Kumar *et al.*, 2013). Therefore, the leachate is a complex mixture consisting of dissolved organic matters and inorganic contaminants. Leachate contains: VFA, LCFA, fulvic and humic substance, ammonia-nitrogen, phosphate, sulphate, heavy metals, organic xenobiotics (XOCs); aromatic hydrocarbons, phenols and chlorinated aliphatics, inorganic salts and microorganisms (Christensen *et al.,* 2001; Renou *et al.*, 2008; Zainol *et al.,* 2012; Kawai *et al.*, 2012; Hassan and Xie, 2014); as well as biorefractory contaminants (Tatsi *et al.*, 2003). Consequently, the leachate contains complex dissolved organic and inorganic substrate, which are biodegradable and non-biodegradable (Christensen *et al.*, 2001).

The characteristics make leachate to be very dangerous matters for environment and it has more potential contamination than other industrial wastes (Zainol *et al.*, 2012; Hassan and Xie, 2014). Variation of leachate composition of municipal landfill depends on several factors, among others: the composition and age of the landfill, design and operation of the landfill, as well as conditions of landfill, climate, hydrogeological conditions, humidity, temperature, and stabilization level (Renou *et al.*, 2008).

Anaerobic bioreactors

Anaerobic treatment of organic matter constitutes a complex and specific biochemical reactions. Biodegradation of organic matter is dissolved through reaction stages; hydrolysis, acidogenesis, acetogenesis and methanogenesis occurring simultaneously, either serial or parallel (Zinatizadeh *et al.*, 2006; van Lier *et al.*, 2008; Deublein and Steinhauser, 2008; Appels *et al.*, 2008; Abdelgadir *et al.*, 2014, Kahar *et al.*, 2017). The bacteria that have role in the four mechanism work specifically and interdependence (Hossain *et al.*, 2009).

Hydrolysis is a liquefaction of organic matter using extra-cellular enzymes produced by hydrolytic bacteria (Zinatizadeh *et al*., 2006; Deublein and Steinhauser, 2008; Appels *et al*., 2008). Cellulolytic bacteria plays a role in the hydrolysis stage, it works in a pH range of 6-7. Other studies report that the hydrolysis rate constant depends on pH but not related to total and the concentration of VFA (Veeken *et al*., 2000). The best pH ranges to achieve maximum biogas resulting in anaerobic bioreactor is 6.5-7.5 (Liu *et al*., 2008; Khalid *et al*., 2011) and 6.8-7.2 (Ward *et al*., 2008).

Acidogenesis is the stage where organic matter as a result of hydrolysis mechanism is converted into amino acids, simple sugars and volatile fatty acids (VFA), including formic acid, acetic, propionic, butyric, lactic, succinic, ethanol, and CO_2, H_2, NH_3, H_2S by acid-forming bacteria (Batstone *et al*., 2002; van Lier *et al*., 2008; Ziemiński and Frac, 2012). For experimental batch system, the pH range at acidogenesis thermophilic mechanism is 6-7 (Park *et al*., 2008). Volatile fatty acids (VFA) is an intermediate product, which is very important in anaerobic treatment (Appels *et al*., 2008). Acetogenesis is a mechanism to form acetates, carbon dioxide and hydrogen (Ziemiński and Frac, 2012).

It is reported that methanogenesis in anaerobic bioreactor is efficient at pH 6.5-8.2 (Lee *et al*., 2009), while hydrolysis and acidogenesisis at pH 5.5 and 6.5 (Kim *et al*., 2003). The last mechanism in the anaerobic biodegradation is methanogenesis. Most of methanogens bacteria are mesophilic with a temperature range of 28-42 °C, while at a thermophilic temperature is in range of 55-72 °C (Ziemiński and Frac, 2012). Methanogens bacteria are optimum in pH range of 7.2-8.0 (Suryawanshi *et al.*, 2013).

Mass Transfer

Mass transfer may occur in liquid phase, gas phase, or in both phases simultaneously. And it may occur with a multi-phase or single-phase system (Thibodeaux, 1996; Geankoplis, 2003). In the case of complex organic substrates, which is generally expressed as COD, a substrate that is degraded hard (Zaiat *et al*., 2000; Christensen *et al.*, 2001; Hassan and Xie, 2014)). Even though mass transfer is not the main factor in, it is crucial in anaerobic bioreactor (Leib *et al*., 2001; Doble, 2006; Benz, 2011).

Liquid phase mass transfer coefficient is a function of the properties of liquid physic and liquid superficial velocity (Zaiat *et al*., 2000; Abdelgadir *et al*., 2014). If the liquid phase mass transfer is as a constraint of the overall process rate, then k_L can be estimated by equation 1 (Zaiat *et al*., 2000; Cho and Young, 2001).

$$N_L = k_L(S_b - S_i) \tag{1}$$

Where, N_L is the liquid phase mass transfer fluxes, (mg/cm^2.h); k_L is the liquid phase mass transfer coefficient; S_b is substrate concentration in bulk liquid, (mg COD/L); and S_i is the substrate concentration in the solid-liquid interface, (mg COD/L).

Diffusion Coefficients

Mass transfer depends on solute diffusion and variable controlling the characteristics of fluid flow, which are: mass transfer depends on solute diffusion and variables controlling the characters of fluid flow, which are: flow rate, viscosity, density, and a linear dimension. Diffusion based on empirical modifications was reported by Wilke-Chang (Geankoplis, 2003), where the solute diffusion can be calculated by the equation 2.

$$D_L = 7,4 x 10^{-8} \frac{(\psi_B.M_B)^{1/2} T}{\mu_L.V_A{}^{0,6}} \tag{2}$$

Where; D_L is solute and solvent liquid phase diffusion, cm^2/s; T is temperature, K; μ_L is solvent viscosity, cP; V_A solute molar volume in normal boiling point, cm3/gmol; Ψ_B is solvent association parameter (water = 2.6) and M_B is molecular weight of solvent (18 g/gmol).

Several studies have reported bioreactor failure or poor performance due to a decrease in pH caused by the accumulation of VFA in the anaerobic treatment system (Visser *et al*., 1993; Fabián and Gourdon, 1999; Poh and Chong, 2009; Tabataba'i *et al*., 2011). High VFA concentration will inhibit hydrolysis, and acidogenesis and methanogenesis. Therefore, the process of mass transfer and adequate seeding microorganisms will be important in anaerobic bioreactor (Vavilin *et al*., 2002).

This research aims to analyze the effect of temperature-pH on the liquid phase mass transfer coefficient; k_L, and solute diffusion coefficient, D_L in leachate treatment in anaerobic bioreactor.

MATERIALS AND METHODS

The leachate used came from Sambutan Landfill, Samarinda, East Kalimantan, Indonesia. Bioreactor anaerobic tools used were equipped with a heater, leachate recirculation pump, leachate recirculation flowmeter, manometer, inlet of leachate feeding, biogas thermometer, pressure gauge, leachate thermometer, sampling ports, inlet valve of leachate recirculation, outlet valve of leachate recirculation and leachate effluent valve. This is pilot-scale experiment research with a semi-batch system.

This research used an anaerobic bioreactor with the volume of 160L. After characterizing and analyzing leachate quality, then leakage and calibration test of anaerobic bioreactor system was performed. Seeding and acclimatization stage were respectively executed for 10 days, while the leachate treatment was performed for 21 days. Seeding, acclimatization and leachate treatment were performed in a bioreactor anaerobic at temperature 35°C; pH ambient, temperature 45°C; pH ambient, temperature 35°C; pH 7.2 and temperature 45°C; pH 8.0. Microorganisms used came from cow rumen fluid as inoculums and leachate with a ratio of 1: 3 and then it was filtered to take the extract. Analytical and parameters test of COD and VFA were performed every two days. Leachate treatment process was stopped if the reduction percentage of COD (COD$_{removal}$) had reached 60-80 %.

RESULTS AND DISCUSSION

Results

Anaerobic treatment was at a temperature of 35°C, then ambient pH was treated; constant leachate temperature was at 35°C, biogas temperature was at 34-35°C, pH tended to fall in the range of 7.9-7.3. In addition, BOD had decreased from 3,850.32-693.50 mg/L, COD had decreased from 6,520-1,327.45 mg/L. Meanwhile, the VFA concentration was up and down, on day 21st, it was 166.5 mg/L, then it rose to the highest concentration at 25th day, which was 1,698.97 mg/L, and then it was up down until the 41st day to 331.85 mg/L.

Anaerobic treatment was at a temperature of 45°C, then ambient pH was treated; constant leachate temperature was at 45°C, biogas temperature was at 42-44°C, pH tended to fall in the range of 7.3-7.9. In addition, BOD had decreased from 4,104.18-902.88 mg/L, COD had decreased from 7,445.11-1,563.45 mg/L. Meanwhile, the VFA concentration was up and down, on 21st day, it was 166.5 mg/L, and then it rose to the highest concentration at 27th day, which was 1,232.1 mg/L, and then it was up and down until the 41st day to 80.78 mg/L.

Anaerobic treatment was at a temperature of 35°C, then ambient pH of 7.2 was treated; constant leachate temperature was at 35°C, biogas temperature was at 34-34.9°C, pH was constant at 7.2. In addition, BOD had decreased from 3,925.50-689.50 mg/L, COD had decreased from 6,155.9-1,124.5 mg/L. Meanwhile, the VFA concentration was up and down, on the 21st day, it was 234.8 mg/L, then it rose to the highest concentration at 27th day, which was 1,678.5 mg/L, and then it was up and down until the 41st day to 338.5 mg/L.

Meanwhile, Anaerobic treatment was at a temperature of 45°C, then pH of 8,0 was treated; constant leachate temperature was at 45°C, biogas temperature was at 42-44.5°C, pH was constant at 8.0. In addition, BOD had decreased from 3904.20-702.68 mg/L, COD had decreased from 6,531.1-1,306.35 mg/L. Meanwhile, the VFA concentration was up and down, on the 21st day, it was 266.5 mg/L, then it rose to the highest concentration at 29th day, which was 1,566.5 mg/L, and then it was up and down until the 41st day to 168.7 mg/L.

Mass Transfer Rate of r_{kL} and Solute Diffusion Rate of r_{DL}

The average of mass transfer rate per day, r_{kL} and the solute diffusion rate per day; r_{DL} on temperature variation, are presented in Figure 1 and Figure 2. At a temperature of 35°C; ambient pH, it was found that the lowest average of mass transfer rate per day was 0.04069 mg/L, while the highest was on the 31st day, which was 0.20608 mg/L, per day. The solute diffusion rate initially fell and then rose, with a range between $2.16678.10^{-5}$-$8.18626.10^{-5}$ cm^2/s. At a temperature of 45°C; ambient pH, it was found that the lowest average mass transfer rate per day was 0.00584 mg/L, while the highest was on the 23rd, which was 0.24842 mg/L, per day. The solute diffusion rate initially fell and then rose, with a range between $2.80848.10^{-5}$-$1.5488.10^{-4}$ cm^2/s.

At the temperature of 35°C; pH 7.2, it was found the lowest average of mass transfer rate per day was 0.01552 mg/L, while the highest was on the 31st day, which was 0.16000 mg/L, per day. The solute diffusion rate initially fell and then rose, with a range between $2.30263.10^{-5}$-$6.62193.10^{-5}$ cm^2/s. Meanwhile, at a temperature of 45°C; pH 8,0, it was found that the lowest average of mass transfer rate per day was 0.03415 mg/L, and the highest was on 23rd day, which was 0.19148 mg/L, per day. The solute diffusion rate initially fell and then rose, with a range between $2.50138.10^{-5}$-$9.96698.10^{-5}$ cm^2/s.

Discussion

By the presence of concentration gradient, there would be movement of mass transfer from the high concentration areas to low concentration areas. This movement was utilized to build intensive contact between solute and microorganisms, therefore, it enabled more frequently mass transfer to occur (Geankoplis, 2003; Welty *et al*., 2007). The occurrence of Mass transfer in the liquid phase was due to the difference of solute substrate concentration (organic and inorganic) inside. Movement among the solute, solvent and microorganisms occurred continuously. Then, as a result of its collisions, the solute, solvent and microorganism moved in irregular and randomly.

During the process of anaerobic treatment, substrates were biodegraded simultaneously by one phase to another phase, where complex organic substrates were biodegraded into intermediate product (VFA) and then converted into biogas (CH_4 and CO_2) (van Lier *et al.*, 2008). The more the dissolved substrate mass biodegraded, the lower the substrate mass dissolved, therefore, the dissolved substrate concentration dissolved in leachate would be also decreased.

Mass Transfer Rate, r_{kL}

The average mass transfer per day was the amount of solute biodegradable substrate every day. COD reduction was accompanied by the increase in the mass transfer rate, average r_{kL} per day. There was increase of r_{kL} average per day because at that time, the activities of microorganisms and the amount of dissolved substrate was still high. Intensive contact between substrate and microorganisms occurred in areas of the highest average of mass transfer per day, which occurred between 29th-30th day. However, on the 31st day, the average of mass transfer per day began to fall. As solute substrate reduced, the average of r_{kL} per day also reduced. As shown in Figure 1.
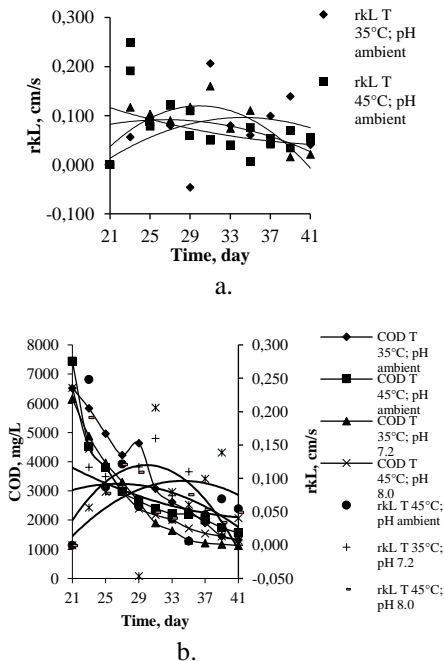

a.

b.

Figure 1. a. r_{DL} on Temperature-pH variation, b. r_{kL} vs COD on Temperature-pH variation

Based on this research, it is found that if COD decreases, mass transfer will be higher. It is important to note before and after the highest average of r_{kL} per day at all operating conditions of leachate treatment in anaerobic bioreactor. Before the highest average of r_{kL} per day, organic substrate solute is still high. While, after the highest average

of rkL per day, the concentration of solute organic substrate decreases.

Figure 1 also shows that the condition before the peak and highest r_{kL} were in the 21st to 27th day and 29th to 30th day, and basically, it can be considered as stage of exponential and stationary of microorganism growth. The highest microorganism activity was in both conditions. Therefore, the average r_{kL} per day indirectly implies that the rate of solute substrate mass biodegradation occurs in the leachate. Actually, this condition must be preserved because it is the average optimum condition at all operating conditions of leachate treatment that is performed. Because the thrust of the liquid phase mass transfer, then the highest concentration gradient is at these both stages.

After this condition, which is after the 31st day, the decrease of average COD at all processing operations is > 64.5%. So the mass transfer occurring after the 31st day is the "insufficient" mass transfer. Because substrate concentrations have been greatly reduced, the thrust of mass transfer is small.

Diffusion Rate, r_{DL}

In the early stages of anaerobic treatment, the average solute diffusion rate; r_{DL} is seen to decrease. As the time goes by, r_{DL} continues to increase. As seen in Figure 2. Similarly with r_{kL}, It is important to note before and after the highest average of r_{DL} per day at all operating conditions of leachate treatment in anaerobic bioreactor. Before the lowest average of r_{DL} per day, organic substrate concentration is still high, while, after the lowest average of r_{DL} per day, the concentration of solute organic substrate has decreased. the lowest r_{DL} was on 27th-29th day, wherein the decrease of average COD substrate concentration on all processing operations is > 56.8%.
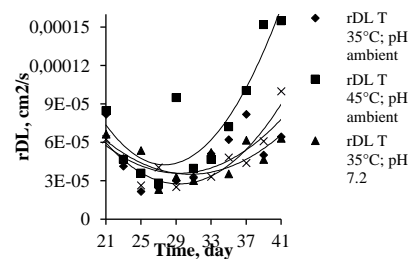

Figure 2. . r_{DL} on Temperature-pH variation

Solutes diffusion rate, r_{DL} will decrease if the pressure increase. However, in the "limit" of particular biogas pressure, r_{DL} increases, hence it can be considered that there is optimal limit for pressure. As seen in Figure 3.
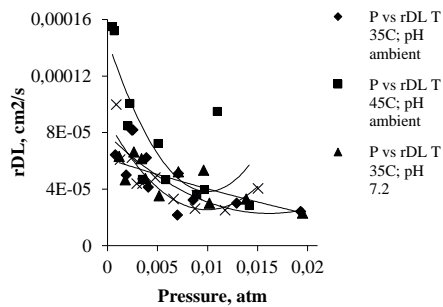
Figure 3. r_{DL} vs P on Temperature-pH variation

Liquid phase mass transfer is affected by the substrate concentration (Cubas *et al.*, 2007), the total solute organic conversion rate (Zaiat *et al.*, 2000; Cho and Young, 2001; Ramos *et al.*, 2003; Chou and Huang, 2005). Based on this research, it is clearly seen that there is dependency on solutes substrate mass (COD) in the leachate, at all operating conditions of leachate treatment in anaerobic bioreactor. Hence, the substrate concentration (COD) will affect the VFA, the liquid phase mass transfer; r_{kL} and solutes diffusion; r_{DL} which are shown in Figure 4 and Figure 5.
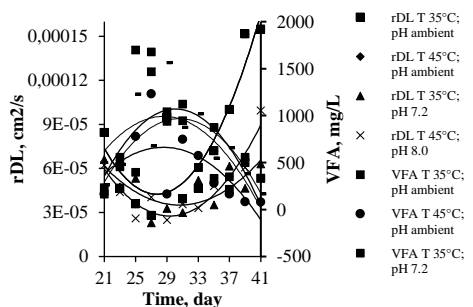

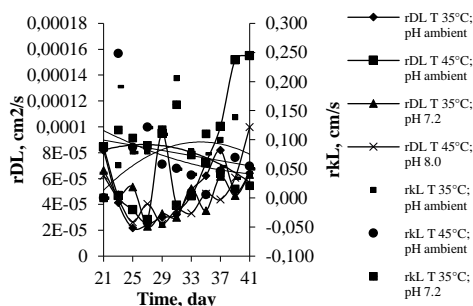**Figure 4.** r_{DL} vs VFA on Temperature-pH variation

Figure 5. r_{DL} vs r_{kL} on Temperatuer-pH variation

This dependence is absolute. It is because the thrust of mass transfer, in addition, it is also because the concentration of each this parameter depends on the other parameter concentration. Therefore, if one parameter increases or decreases, another parameter will also increase or decrease.

Table 2. Studies on Mass Transfer

Liquid Phase Mass Transfer Coefficient; k_L and Diffusion Coefficient; D_L

Liquid phase mass transfer coefficient; k_L, and the solute and solvent diffusion coefficient; D_L on leachate treatment in anaerobic bioreactors, as presented in Table 1. In the treatment of T variation, k_L is obtained in T 35°C; T 45°C; T 35°C; pH 7.2; and T 45°C; pH 8.0 in respectively is 0.085 cm/s, 0.053 cm/s, 0.087 cm/s and 0.067 cm/s. While the solute diffusion coefficient in the treatment of T variation, D_L is on T 35°C, 45°C T, T 35°C, pH 7.2 and T 45°C; pH 8.0 in respectively is 1.10^{-06} cm²/s, 5.10^{-06} cm²/s, 2.10^{-07} cm²/s and 2.10^{-06} cm²/s.

Tabel 1. Liquid phase mass transfer coefficient, k_L, and diffusion coefficient; D_L

Treatment	k_L, cm/s	D_L, cm²/s
35 °C; pH ambient	0.085	1.10^{-06}
45 °C; pH ambient	0.053	5.10^{-06}
35 °C, pH 7.2	0.087	2.10^{-07}
45 °C, pH 8.0	0.067	2.10^{-06}

On the treatment of temperature variation, even though it is seen that if temperature T is higher, then k_L will increase while D_L will decrease, then the condition is up and down. k_L is on T 35°C, pH 7.2 > T 35°C > T 45°C, pH 8.0 > T 45°C. Meanwhile, D_L is on T 45°C > T45°C, pH 8.0 > T 35°C > T 35°C, pH 7.2, as described in Figure 6.
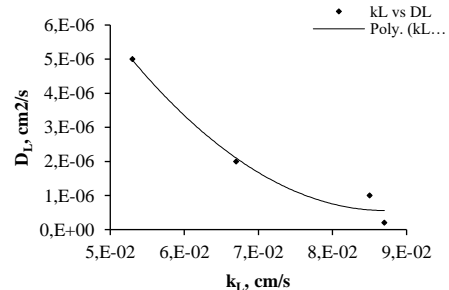

Figure 6. D_L vs k_L on Temperature-pH variation

In general, it can be said that, the more the affecting variables, the bigger the factor of "obstacle" on the process of anaerobic treatment. Liquid-phase mass transfer coefficient, k_L is a function of the solutes physical chemical properties (pH, COD and VFA), internal characteristics and operating conditions of the bioreactor (geometry bioreactor, recirculation flow rate, temperature, pressure) (Zaiat *et al.*, 2000; Geankoplis, 2003; Kraakman *et al.*, 2011). Several studies of mass transfer and the comparison of this research are shown in Table 2.

Bioreactor	Wastewater	V_R, L	HRT (day)	Temp. (ºC)	COD$_{Inf.}$ (mg/L)	Mass Transfer Coefficient, k_L (cm/s)	References
HAIB (pilot-scale)	Glucose-based synthetic substrate	237	8 h	30	2,090-41	3.40.10-2 cm/h	Zaiat *et al.*, 2000
	Domestic Sewage		8 h	30	341-71	2.23.10-1 cm/h	Zaiat *et al.*, 2000
Two-stage Anaerobic Filter	Brewery wastewater	7.45 dan 6.55	0.5-6 d	35	1,500-2,500 (OLR 0.5-20 g SCOD/L.d	1.4-2.2 d-1	Cho and Young, 2001
Fixed-Bed ASBR	Synthetic Wastewater (Polyurethane Foam)	1.2	8 h	30	500-68	1.98-1.85 h-1	Ramos *et al.*, 2003
UASB	Synthetic Wastewater (phenol)	3.78	0.97-1.03 d	35	10.53-12.61 kg COD/m3.d	19.2 mg phenol/L	Chou and Huang, 2005
SASBR	Synthetic Wastewater (Polyurethane Foam)	5		30	285-333	0.48-0.60 h-1	Cubas *et al.*, 2007
Anaerobic Bioreactor (pilot-scale)	Leachate	160	1 d	35-45	6,155.9-7,445.11	0.053-0.087 cm/s	This research

CONCLUSIONS

Based on the research results and discussion, the conclusion that can be drawn are as follows; Liquid phase mass transfer coefficient; k_L, on the T 35°C; T 45°C; T 35°C; pH 7.2; and T 45°C; pH 8.0 respectively are 0.085 cm/s, 0.053 cm/s, 0.087 cm/s and 0,067 cm/s. Diffusion coefficient D_L on T 35°C, T 45°C, T 35°C, pH 7.2 and T 45°C; pH 8.0 respectively are 1.10^{-06} cm^2/s, 5.10^{-06} cm^2/s, 2.10^{-07} cm^2/s and 2.10^{-06} cm^2/s. The higher the temperature and pH, the higher the k_L, but the lower the D_L will be. k_L is on T 35°C, pH 7.2 > T 35°C > T 45°C, pH 8.0 > T 45°C, while D_L is on T 45°C > T 45°C, pH 8.0 > T 35°C > T 35°C, pH 7.2. Based on this research, that there is dependency on mass of solutes organic substrate (COD) in the leachate, at all operating conditions of leachate treatment in anaerobic bioreactor. Hence, the organic substrate concentration (COD) will affect the VFA, the liquid phase mass transfer and diffusion of solute organic. Consequently, the higher the temperature-pH, the higher the liquid-phase mass transfer, but lower diffusion coefficients is.

ACKNOWLEDGMENT

NOMENCLATURE
Symbols
D_L solute and solvent liquid phase diffusion, cm^2/s
k_L liquid phase mass transfer coefficient; cm/s
M_B molecular weight of solvent, 18 g/gmol
N_L liquid phase mass transfer fluxes, mg/cm^2.d
r_{DL} solute diffusion rate, cm^2/s
r_{kL} mass transfer rate, mg/L.d
S_b substrate concentration on bulk liquid, mg COD/L
S_i substrate concentration on solid-liqiud interface, mg COD/L
T temperature, K
V_A solute molar volume on normal boiling point, cm^3/gmol
V_R Reactor volume, L
Ψ_B association parameter of solvent, water = 2.6
μ_L viscosity solution, cP

Abbreviations
ASBR   Anaerobic Sequencing Batch Reactor
COD   Chemical Oxygen Demand
HAIB   Horizontal-Flow Anaerobic Immobilized Biomass
HRT   Hydraulic Retention Time
SASBR   Stirred Anaerobic Sequencing Batch Reactor
UASB   Upflow Anaerobic Sludge Bed Reactors
VFA   Volatile Fatty Acids

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