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Zeolite Utilization as a Catalyst and Nutrient Adsorbent of an Organic Fertilizer Process From Palm Oil Mill Effluent as Raw Material

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ABSTRACT

Palm Oil Mill Effluent (POME) cannot be directly used as an organic fertilizer source due to its high Biological Oxygen Demand (BOD) thus it is not environmentally safely. To increase the high quality of organic fertilizer obtained, the liquid wastes are needed to be processed in order to decrease the BOD to degrade both the soluble and suspension materials of organic materials. The altenative process to be conducted to make a better quality of POME is by adding the adsorbent. The aim of the research was to study the effect of zeolite utilization and duration of hydrolysis process in order to increase the nutrients content and to decrease the BOD of POME. The research was conducted at the PT Sumbertama Nusa Pertiwi Jambi, Indonesia in August 2012 until February 2013. The sample of POME was taken from the inlet of the factory's acidulating pool. There were several doses of zeolite as treatments which were 0, 5, 10, 15% and several durations of hydrolysis process which were 1,2,3 and 4 weeks. Active zeolite was added to POME and then it was fermented with different hydrolysis duration times as mentioned above. The research showed that application of zeolite and duration of hydrolysis process significantly affected the pH, N, P, K, Al, Fe, BOD of POME and the adsorption of N, P, K, Al, Fe by zeolite. It can be concluded that 10% of zeolite incubated in two weeks duration of hydrolysis process produced higher nutrient of N, P, K with BOD, Al, Fe and pH matched with the waste quality standard. The highest efficiency of N, P and K adsorbent was show by the 15% of zeolite which was incubated for two weeks of hydrolysis process.

***Keyword*:** Hydrolysis process duration, nutrient content, palm oil mill effluent, zeolite.

INTRODUCTION

The fresh palm oil mill effluent has brownish color which consists of soluble and suspended solid such as colloid and oil residues. The solid has high Chemical Oxygen Demand (COD) and Biological Oxygen Demand (BOD) with values of 68,000 mg L^{-1} and 27,000 mg L^{-1}, respectively, pH 3.5-4.0, 95% water, 4-5% soluble and suspended solid (*e.g.* cellulose, protein and fat) and 0.5%-1% oil residue with majority as an emulsion. The total suspended solid (TSS) concentrations of 1,330 – 50,700 mgL^{-1} of POME consisted of copper 0.89 mgL^{-1}, iron 46.5 mgL^{-1}, zinc 2.3 mgL^{-1}, and ammonia 35 mgL^{-1} (Ma 2000).

The processing steps of POME in the oil palm mill consist of fat Pit pool, fertilization pool, acidulating pool, neutralizing pool, primer anaerobic pool I, primer anaerobic pool II, aerobic pool, sedimentation pool, facultative pool, and controlling pool. It is about 150-220 days of processing. Some drawbacks in the processing condition were: unpleasant odor and soil contamination around the pools, the pools need a large land area, it produces methane gases, it needs a periodic maintenance to control the sludge and decrease of nutrients (Raharjo 2006).

POME with 75 days stayed in the primer anaerobic pool produces 3,500-5,000 mgL^{-1} of BOD. According to Raharjo (2009) the liquid waste produced by 40 days in anaerobic pool following by 60 days in aerobic pool was able to decrease 200-230 mgL^{-1} of BOD. BOD which were decreased ranging from 27,000 by 2,500 mgL^{-1} followed by 40% decreased in nutrients (N, P, and K) after standard factory processing conducted in secondary anaerobic pool, if compared to the pre processing stage (Budianta 2005). The same statement that decreasing BOD after the processing would be followed by decreasing the nutrient (N, P, and K) of POME (Simanjuntak 2009).

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Zeolite has a *negative charge neutralization* ion mineral that can be neutralized by alkali metal, it is microporous that consist of cationic elements (K, Na, Ca, Mg) and H_2O as it is possible to release the water and ion exchange. The other benefits are as an adsorbent of cations that contaminate the environment (Pb, Al, Fe, Mn, Zn and Cu). The zeolite reduced the environmental contamination (Oste *et al*. 2002).

The zeolite utilization will be able to adsorb the heavy metal e.g. Pb, Hg, and Cd (Vaulina 2002). Zeolite was able to adsorb CO_2, H_2S and NH_3 and also able to inhibit the releasing of soil nitrogen. So far, the utilizations of zeolite in agriculture are: as a raw material to increase the quality of organic fertilizer and slow release fertilizer processing, soil conditioner and stock water controller. It was found that, the zeolite utilization should be combined with organic fertilizer (Jabri 2008).

Application of 0.5 kg of zeolite to the compost that was decomposed for 3 weeks were able to increase the compost acidity, the availablity of N, P, and K, as well as to decrease ratio C/N of compost (Susanti and Panjaitan 2010). Five percents of zeolite on the sand increased the population of bacteria and fungi (Djajadi *et al*. 2010). Ten percents of zeolite on the mining mud which was incubated during 6 weeks were able to decrease 40% Zn, contained of 23.18% oil and 14.16% polyaromatic hydrocarbon, and it affected the bacterial and fungal quantity (Dhayat 2011). Zeolite minerals are capable to increase the phosphate concentration, and inhibits the removal of compost phosphate, having P retention capacity, as an adsorbent and nutrition slow release (Gu *et al*. 2011). Urine with 20% zeolite decrease the N removal, due to 21.27 mgL^{-1} N (ammonium) had been adsorbed by zeolite, furthermore the N would be slowly released. Zeolite is capable to reduce a bad aroma and reduce the urine ammoniac concentration (Sumarlin *et al*. 2008).

To be a high quality organic fertilizer, the POME should be processed with the aims: to decrease its BOD, COD, and TSS concentrations, to increase the acidity and nutritions, to degrade the organic materials (soluble and suspension materials). The aims of the research were to study the effects of zeolite utilization and hydrolysis durations, to increase the N, P, K, Al and Fe concentrations of POME.

MATERIALS AND METHODS

Study Sites

The research materials a 60 mesh clinoptilolite zeolite, were taken from Gedang Sari village, Gunung Kidul, Jogyakarta and POME from acidulating pool belonging to palm oil mill (PT. Sumbertama Nusa Pertiwi Jambi). Fermentation bottled and other laboratories equipment were used for analyses.

Experimental Design

A factorial treatment in a completely randomized design was used with three replicates. Four dosages of zeolite used (w/v) were z_0=0%, z_1=5%, z_2=10%, z_3=15%, respectively. Hydrolysis process consisted of four time durations which were h1=1-week, h2=2-weeks, h_3=3-weeks and h_4=4-weeks. Palm oil mill effluent has been placed in the open-air room, then it was put in the fermentation bottles and the bottles should be tightly closed. Furhtermore, zeolite that has been activated by 150°C or frying for 15 minutes was fermented on each hydrolysis duration with different zeolite rates. The processed zeolite were put into each bottle.

Soil Analysis

POME were analyzed by pH (H_2O), N (Kjedahl digestion), P (spectophotometry), K (AAS), BOD (Winkler method), total bacteria, and Fe and Al. Adsorption of N, P, K, Al and Fe (Batch method) were also analyzed. The zeolite and POME analysis were conducted at Tekmira Laboratory, a Developing and Research Centre of Mineral and Coal Technology, Bandung. As a finishing step, the mineral (N, P, K, Al and Fe) adsorption efficiency of zeolite was calculated.

Statistical Analysis

Data obtained were analyzed with ANOVA and the mean separations among treatments were analyzed by DMRT test at the level P= 0.05.

RESULTS AND DISCUSSION

Zeolite and POME Characterization

The POME which was taken from the in-let of acidulating pool had ratio C/N (39.42), and contained organic-C (10.44%), total-N (0.27%), total-P (0.09%), K (0.10%), COD (43,137 mgL^{-1}), BOD (27,545 mgL^{-1}), TSS (18,782 mgL^{-1}), and pH (4.77). The zeolite used was a 60 mesh clinoptilolite type which was taken from Gedang Sari village, Gunung Kidul, Jogyakarta. It contained SiO_2 (78.11%), Fe_2O_3 (0.69%), Al_2O_3 (3.94%), CaO (2.10%), K_2O (0.30%), MgO (0.24%), H_2O (11.19%), and pH (7.2). It had a 35% porous volume with 154.17 $cmol_{(+)}kg^{-1}$ cation exchange capacity.

Effect of Zeolite on N, P, K, BOD Concentration, and pH of POME

The best effect of zeolite on the macro nutrients and chemical compounds of POME was found on a 10% zeolite treatment. However it had a nonsignificant effect on 5% zeolite treatment but it had a significant effect compared to without zeolite. Application of zeolite on pome increased total-N by 29.79% and total-P by 28.77% compared to without zeolite, while total-K of POME increased by 27.17%. The acidity of pome closed to neutral after 10% zeolite application (Figure 1).

Changes nutrient contents of POME which was added by several dosages of zeolite (0%, 5%, 10%, 15%) during the hydrolysis process of fermentasion is shown in Figure 1. The incubation process did not significantly decrease the nutrients contents (N, P, and K) of pome it indicates that it still matched with standard liquid waste quality and ready to be applied as organic fertilizer.

Moreover, the POME quality has also complied with the POME quality standard in the ecology ministerial decree in 2003, no:28 with the standard as follow: BOD concentration: 3,000 – 5,000 mgL^{-1}, pH: 6.5 – 7.5 and Fe: 7 – 8 mgL^{-1}.

BOD concentration decreased from 19,260 mg L^{-1} to 880 mg L^{-1}. There was an increasing pH from 5.06 up to 7.58. Iron and Al concentrations of liquid waste decreased from 27.75 mgL^{-1} to 4.51 mgL^{-1} and 4.83 mgL^{-1} to 2.21 mgL^{-1}, respectively (Figure 1).

Ersoy and Celik (2003), stated that zeolite as a catalyst materials has a stability and selective character to transform organic compound (both the soluble and suspension) from heavy molecular weight (polymer) to a simple organic compound (monomer) which was supported by the electrostatic character of zeolite and its micro porous structure. Furthermore, zeolit plays an important role as an absorbent, in neutralizing pH, and increase the ionic exchange. This condition allows the POME zeolit samples to be able to increase the N, P, and K concentrations.

The average nutrient (N, P, K, Al, and also Fe) adsorption of POME zeolite increased by increasing the zeolite concentration as shown by Figure 2. There were three samples of POME + zeolite which showed a high adsorption of N, Al and Fe (43.8,

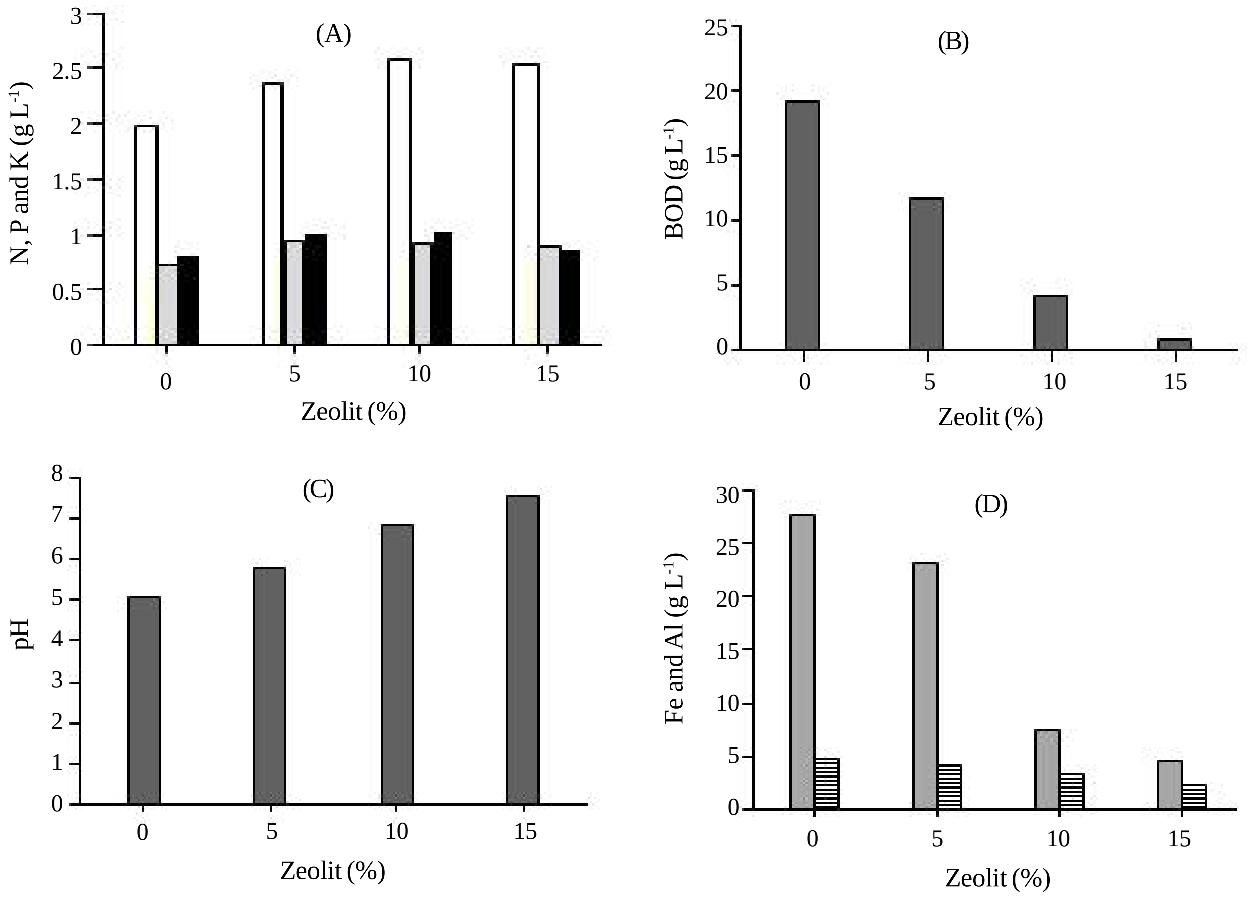

Figure 1. Effect of zeolit dosages on Total-N (□), Total-P (▨), and K (■) concentrations (A), BOD (B), degree of acidity (C), Fe (▨) and Al (▤) concentrations (D) of POME.

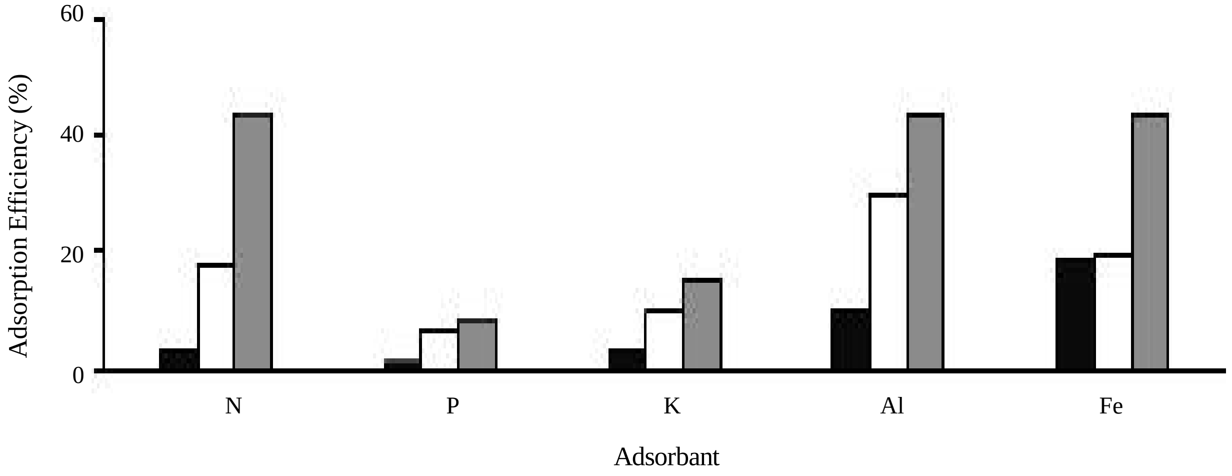

Figure 2. Adsorption efficiency of POME nutrient (N, P, K, Al, and Fe) as the effect of zeolite utilization. Zeolit 5% (■), zeolit 10% (□), and zeolit 15% (▨).

43.53, and 43.58 respectively), followed by K, and P (15.23 and 8.80 respectively).

The results showed that the zeolite was capable to adsorb N, P and, K that produced from degradation of organic compound of POME directly. The adsorption efficiency of N, P and, K by zeolite were 48.85, 8.8, and 65.68, respectively. It has been clarified by Li *et al.* (2000), that zeolite has capability to absorb anions (PO_4^{-3} and SO_4^{-2}) and cations such as (NH_4^+). Sumarlin *et al.* (2008) showed that a 20% zeolite in urine decreased the N removal, because the zeolite adsorbed 21.27 mgL^{-1} of NH_4^+-N. Moreover, Susanti and Panjaitan (2010) stated that the zeolite utilization in compost increased the total-N and P of compost.

The zeolite capability to increase the acidity of POME was supported by its role as acid catalyst and acidity neutralizer. Susanti and Panjaitan (2010) clarified that zeolite utilization in compost fermentation for 3 weeks decomposition increased the degree of acidity and the nutrients availablility of N, P, K, and decreased the ratio C/N. The possibility of increasing pH was caused by the zeolite role in exchanging between the alkaline cations (Ca, K, and Mg) and H^+ and Al^{3+}. Ano and Ubochi (2007) stated that Ca was released through hydrolysis. The hidroxides were reacted with soluble aluminum to form insoluble $Al(OH)_3$. The hydroxide ions (OH^-) in the calcium hydroxide compounds ($Ca(OH)_2$) were also reacted with hydrogen ions (H^+) to form water (H_2O).

Effect of Hydrolysis Duration on N, P, K, BOD Concentration and pH of POME

Hidrolysis duration treatment resulted in varying major effect on each nutrient. The highest concentrations of N, P, and K were shown in 2 weeks of hidrolysis duration. While the neutral acidity was obtained in the 4-weeks of hydrolysis duration. The lowest Al, Fe, BOD concentration was shown in 4 weeks of hidrolysis duration (Figure 3).

In the hydrolysis process the different duration time resulted in the changing of organic compound into simple organic compound due to extracellular enzymes. Increasing hydrolysis duration resulted in decreasing BOD and nutrients (N, P, K, Fe and Al) contents, in contrary it resulted in increasing pH (Figure 3). Luturkey *et al.* (2010) stated that there was several steps of biodegradation process of the organic compound of POME likely hydrolysis process, acidogenes process, asetogenes process, and methanogenes.

An anaerobic waste process is a degradation process of organic compound (carbohydrate, protein, and fat) by anaerobic bacterial to produce biogases *e.g.* CH_4 (50-70%), CO_2 (25-45%) and a few of N_2, H_2, and H_2S. Generally, the anaerobic process is usually applied on liquid waste with COD higher than 4,000 mgL^{-1}, and it needs a long enough duration to avoid a contaminant forming out of it (Luturkey *et al.* 2010). The decrease of N, P, and K was a result caused by the effluent reduction of waste in the form of a castaway gases. The degradation of organic material was also used by the bacterial as energy sources, and cells or biomass formation. Waluyo (2009) stated that the fermentation process of liquid waste in anaerobic condition has deformed an organic compound to be organic acid and biogases. The fermentation process produces many products and dominantly as energy, cells or biomass formation and castaway gases.

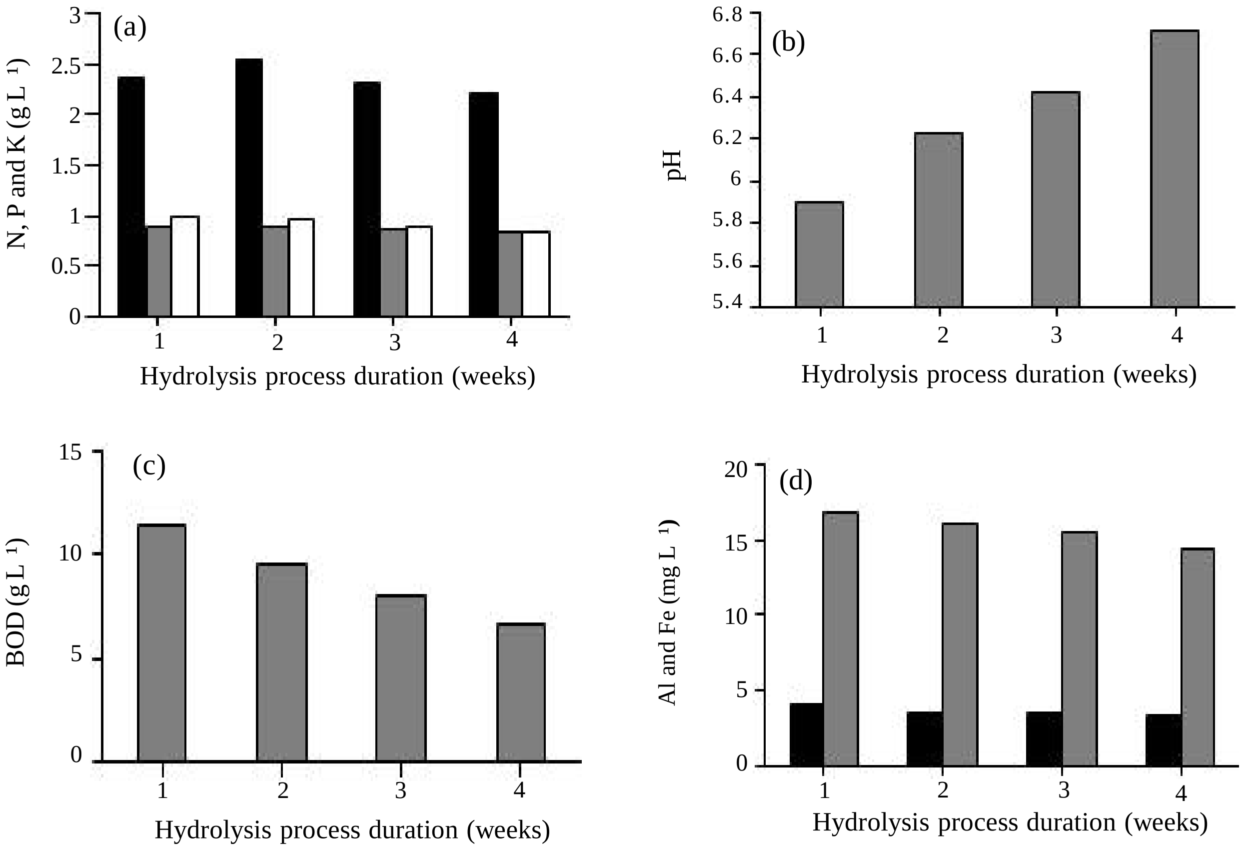

Figure 3. N (■), P (▨), and K (☐) concentration (a), degree of acidity (pH) (b), BOD concentration (c), Al (■), Fe (▨) concentration (d) of POME as a treatment effect of hydrolysis duration.

Interaction between Zeolite and Hydrolysis Duration on the POME Characteristics

The highest concentration of both N (2.79 gL^{-1}) and total-P (0.97 gL^{-1}) was shown in the 2- weeks hydrolysis duration with 5% zeolit. The highest average of K concentration (1.07%) was shown in the 2-weeks hydrolysis duration with 5% zeolit (Table 1).

Table 1 showed that increasing both zeolite utilization and hydrolysis duration resulted in the increasing of POME acidity, Al, Fe and BOD concentrations. The neutral acidity was achieved for the 10% zeolite treatment in the 2-weeks of hydrolysis duration.

The average of nutrient adsorption efficiency (Table 2) showed that increasing zeolite in POME increased the zeolite adsorption capability in nutrients (N, P, K) and alkalinity (Al, and Fe). The combination treatment of 15% zeolit and 2-weeks of hydrolysis duration resulted in the highest efficiency of the nutrient absorptions that were 45.93% N, 12.16% P, 14.26% K, 41.90% Fe, and 42.35% Al.

The analysis of Multi-correlation (Table 3) showed that total-N has highly positive significant correlations with total-P, pH, and K, and has negative correlations with Al, Fe and BOD concentrations. Furthermore, total-K has correlations with total-N, total-P, pH, and K, and it has no correlations with Al, Fe and BOD concentrations.

Increasing POME acidity increased N, P, and K, but decreased Al, and Fe (Table 3). This phenomena was possibly due to the ability of zeolite to release to exchange alkaline cations, to produce hydroxide aluminum, and to adsorp Fe and Al. Moreover adsorption efficiency of Fe and Al by 15% zeolite in 4-weeks hydrolysis duration was 47.49% and 47%, respectivelly (Table 2). Kundari *et al.* (2010) stated that the liquid waste that consist of Fe and Mn can be effectively adapted. Karamah *et al.* (2010) also reported that zeolite has a capability to absorb 93.3% Fe in waste that consist of Fe, Cu, Ni and ammonium at pH 8.

The result of the research showed that zeolite has a capability to decrease BOD concentration (Table 1). BOD is the amount of oxygen needed by bacteria to degrade the organic material of POME. It was found that the increasing of N, P, and K concentrations decreased of BOD concentration (Table 3). The decrease in BOD concentration by 35% was caused by the zeolite's multi-porous (its density 2.2 g cm^{-3}) and porous volume which consist

Table 1. The chemical characteristics of POME as affected by zeolite utilization and hydrolysis duration.

Zeolite (%)	HPD (weeks)	Total-N (g L^{1})	Total-P (g L^{-1})	K (g L^{1})	pH	Al (mg L^{-1})	Fe (mg L^{-1})	BOD (g L^{-1})
0	1	2.07 ab	0.82 c	1.00 f	4.67 a	4.81 j	28.58 l	22.48 m
	2	2.02 ab	0.79 c	0.91 g	4.69 a	4.80 j	28.16 k	20.55 l
	3	1.97 ab	0.72 b	0.72 b	5.14 b	4.79 j	27.09 j	17.77 k
	4	1.83 a	0.59 a	0.61 a	5.74 c	4.94 j	27.15 j	16.26 j
5	1	2.36 cd	0.92 ef	1.04 g	5.28 b	4.53 i	25.17 i	15.69 j
	2	2.79 e	0.97 f	1.07 h	5.79 c	4.42 i	24.18 h	12.15 i
	3	2.29 c	0.91 e	1.01 f	5.86 c	3.90 h	23.19 g	10.23 h
	4	2.08 ab	0.95 f	0.97 e	6.26 d	3.62 fg	20.13 f	8.70 g
10	1	2.55 de	0.92 ef	1.04 g	6.31 d	3.81 gh	8.46 e	6.07 f
	2	2.92 e	0.91 e	1.05 g	6.89 e	3.41 ef	7.25 d	4.70 e
	3	2.28 c	0.94 f	1.03 g	6.85 e	3.17 c	7.03 d	3.54 d
	4	2.06 ab	0.92 ef	0.99 f	7.31 f	3.24 cd	6.82 c	2.05 c
15	1	2.52 de	0.92 ef	0.88 d	7.36 f	2.93 b	5.03 b	1.78 b
	2	2.37 cd	0.91 e	0.85 c	7.54 f	1.85 a	4.97 b	0.89 a
	3	2.74 de	0.92 ef	0.84 c	7.57 f	2.02 a	4.75 b	0.53 a
	4	2.89 e	0.88 d	0.82 c	7.84 g	2.03 a	3,28 a	0.34 a

Note: values in the same column followed by the same small letter are not significantly different to DMRT (P >0.05). HPD = hydrolysis process duration.

Table 2. Nutrition adsorption efficiency (%) by zeolite on the POME as affected by zeolite utilization and hydrolysis duration.

Zeolite (%)	HPD (weeks)	N	P	K	Fe	Al
5	1	2.47 a	1.08 a	2.16 a	16.53 ab	2.73 a
	2	3.33 a	0.86 a	3.15 a	16.54 ab	8.88 a
	3	4.07 a	1.64 a	4.10 a	21.01 bc	12.94 ab
	4	3.95 a	2.09 a	5.08 ab	24.36 cd	17.08 ab
10	1	15.31 b	5.80 b	7.88 bc	14.53 a	29.37 bc
	2	17.90 bc	5.13 b	9.50 cd	20.11 bc	32.42 bc
	3	19.51 c	7.41 bc	11.45 de	16.76 ab	29.37 bc
	4	18.52 c	8.89 cd	11.77 de	26.82 d	29.37 bc
15	1	41.85 d	10.93 e	12.63 d	38.55 e	38.25 c
	2	45.93 e	12.16 f	14.26 ef	41.90 ef	42.35 c
	3	47.96 e	9.83 de	16.53 f	46.37 ef	46.44 c
	4	39.63 d	2.30 a	17.49 f	47.49 f	47.08 c

Note: values in the same column followed by the same small letter are not significantly different to DMRT (P >0.05).

Table 3. The correlations between N, P, K concentrations of POME and pH, Al, Fe, BOD concentrations as affected by zeolite utilization and hydrolysis duration.

No	Variable	R value						
		Total-N	Total-P	K	Al	Fe	BOD	pH
1	N-total	-	$+0.51^{**}$	$+0.38^{**}$	-0.41^{**}	-0.51^{**}	-0.48^{**}	$+0.41^{**}$
2	P-total	$+0.51^{**}$	-	$+0.78^{**}$	-0.49^{**}	-0.48^{**}	-0.54^{**}	$+0.43^{**}$
3	K	$+0.38^{**}$	$+0.78^{**}$	-	-0.05^{ns}	-0.09^{ns}	-0.08^{ns}	$+0.02^{ns}$

Note :** highly significant ; *significant ; ns = non significant of releasable oxygen that could increase the degradation process. Simanjuntak (2009) stated that there are highly positive significant correlations between BOD and N, P, and K concentrations of POME. Besides, Fungaro (2002) find out that zeolite has an opening frame that consist of primary base material to build the secondary base material, and it can continuously build up the next base materials and so on. The morphology and structure of zeolite crystal is dominantly made up of porous structur which then increases the zeolite surface area. This condition has allowed the zeolite to be able to exchange and to adsorp. Feurerstein *et al*. (2000), found that zeolite has the capability to adsorb gases (N_2, CH_4, SO_2, SO_3 and NH_3).

There are several forms of soluble N compounds in the liquid waste such as $N_{2,}$ NO_2^-, NO_3^- and NH_3. In oxidation process ammonium, nitrate, and nitrite form were a labile compound and act as a temporary condition. The phosphate in the liquid waste exist in the form of orthophosphate compounds ($H_2PO_4^-$$_,$ HPO_4^{-2}, and PO_4^{-3}), polyphosphate or polymer compound (hexametha-phosphate, tripoli-phosphate, pyro-phosphate) and as an organic-phosphate (P that bunched in the organic compound). Each phosphate compound above exist as a soluble and suspension phosphate form or it is be bunched in the organism cell. Total-P exist as soluble and suspension phosphate form. The decrease in total-P concentration in the liquid waste was due to Al and Fe fixation of the phosphate compound in the microorganism cell (Simanjuntak 2009).

Cations (Ca^{2+}, Mg^{2+}, and K^+) were released in the fermentation process so this increased the pH of POME. The role of calcium ions in increasing the pH was explained by Ano and Ubochi (2007). Calcium ions are released into the solution when there is a mineralization process of organic materials. The release occurs through decarboxilating process of complex organic material by microbial. The calcium is released and then hydrolysis. The hydroxide is reacted with Al and Fe ion to produce the insoluble Al-hydroxide and Fe-hydroxide.

The microbial population of POME increased after the zeolite addition and hydrolysis treatment, there after its population decreased. The fungus population of POME also increased after the zeolite addition and hydrolysis treatment, there after its population was also decreased. However total fungus was lower than total bacteria (Figure 4).

The population of bacteria and fungi in the POME increased after the zeolite addition and hydrolysis treatment, ther after these population decreased, and the fungus population continuously decreased (Figure 4). This condition was supported by the environment especially the solution acidity of POME. Waluyo (2009) explained that the optimal growth of bacteria and fungi was in the pH range 6 – 7.7. Furthermore, he stated that the growth of bacteria and fungi was affected by the biodegradation of organic compound. The interaction between zeolite utilization and hydrolysis duration has tremendously affected the nutrients content of POME. The research found that N, P, K, Al, and Fe concentrations, BOD, and pH were better than quality standard at 10% zeolite application in the 2 weeks hydrolysis duration (Table 1). The combination between zeolite and the hydrolysis duration of POME produced better (N, P, K, Al, Fe), concentration BOD and pH than the combination between zeolite without hydrolysis duration or hydrolysis duration treatment only.

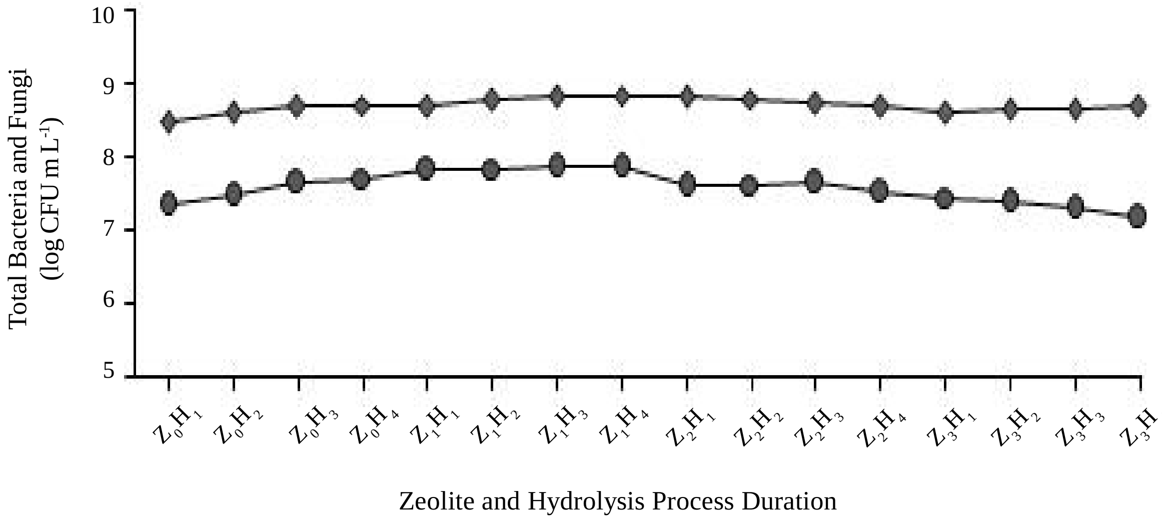

Figure 4. The population of bacteria and fungi (log CFU mL^{-1}) of POME as affected by zeolite utilization (Z) and hydrolysis duration (H).

CONCLUSIONS

The treatment of zeolite utilization and hydrolysis duration has tremendously affected the nutrients content of POME. The utilization of 10% zeolite incubated in the 2 weeks of hydrolysis duration had better concentrations of nutrient (N, P, K), BOD and pH which accomplied with waste quality standard. The zeolite utilization followed by hydrolysis duration of POME produce ' better N, P, K, Fe, BOD and pH than the zeolite utilization without hydrolysis duration or hydrolysis duration treatment only.

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