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Research article

Effect of Food to Microbe (F/M) Ratio on Anaerobic Digestion of Refinery Waste Sludge under Mesophilic Conditions: Biogas Potential and Phytotoxicity

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Abstract

This study investigated the possibilities of improving biogas yield from anaerobic digestion of refinery activated sludge (RAS) by optimizing the food to microorganism (F/M) ratio. Different F/M ratios of 0.25, 0.50, 1.00 and 2.00 were studied. The highest biogas production (147.98±40.7 ml/g_{vs}), methane production (51.41±1.78 ml/g_{vs}) and methane content (42.00±5.90%) were obtained from the F/M ratio of 1.00 followed by 0.50, 0.25 and 2.00, respectively. The phytotoxicity of biosolids that came from anaerobic digestion was also evaluated using three different types of seed (*Vigna radiata*, *Brassica rapa* and *Lycopersicon esculentum*) during this process. Increasing amount of RAS via increasing of the F/M ratio (0.25-1.00) stimulated plant development (GI>100) and reduced the phytotoxicity of RAS.

Keywords

biogas;

refinery waste sludge;

anaerobic digestion;

phytotoxicity

1. Introduction

A typical refinery wastewater treatment plant consists of physical-chemical (dissolved air flotation, DAF) treatment and biological treatment (activated sludge system). Oily sludge formed by the DAF process has been identified as a hazardous waste because it contains various complex compounds

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such as combinations of different petroleum hydrocarbons, heavy metals and solid particles [1]. Organic compounds in the refinery wastewater stream are removed via the biological mechanism such as an activated sludge process, after the DAF process. In addition to the organic compounds, harmful organic compounds such as volatile aromatics and phenols may be detected in the excess waste activated sludge. Refinery wastewater sludge (treatment of excess biological sludge) must be treated and disposed of in a carefully managed manner because it may contain contaminants such as heavy metals that, in terms of non-biodegradability and bioaccumulation, have negative environmental impacts [2]. Typically, the excess activated sludge, which is a waste from the biological process, is concentrated and disposed of in a landfill without further waste utilizing processes. Landfills can emit harmful gases into the atmosphere, and hazardous chemicals can contaminate groundwater in the region if a leak occurs. However, the composition of wastewater streams varies due to factors such as the type of oil being refined, refinery configuration, and operating parameter. According to the growing petrochemical and refinery industries, the world is facing problems with the toxic contamination of the sludge that comes from the petrochemical and refinery industries. Various methods have been proposed for sludge disposal from the refineries such as incineration, land treating off-site, landfilling onsite, neutralization, and other treatment methods [3].

A common treatment technique for sludge stabilization is anaerobic digestion, which can radically decrease the number of organic compounds present and generate biogas production at the same time. In addition, the digested materials can be used as soil conditioners and fertilizers for plant growth. Anaerobic digestion is popularly used to stabilize sludge and decrease the amount of solids for final disposal via landfill [4-6]. Through anaerobic digestion process, industry can improve sustainability with benefits accruing in energy production, economic factors, and waste minimization [7]. Anaerobic digestion has been applied within the petroleum refining industry, and there are few studies on the anaerobic digestion of refinery waste activated sludge [7-10]. The operating parameters such as temperature, pH, sludge retention time (SRT) and form of anaerobic digestion system have been optimized and investigated during previous studies. At thermophilic temperatures, a faster anaerobic digestion rate was observed, and the optimum pH ranged between 5.0 and 9.0 [10]. Less than 8 days of sludge retention time (SRT) resulted in the development of acidic conditions, while periods longer than 8 days resulted in methanogenesis [8]. In two-phase anaerobic digestion, methane production and COD removal efficiency were 1.6 and 2.1 times higher than in single-phase anaerobic digestion [9].

The F/M ratio is an important parameter that affects the treatment performance and biogas yield of an anaerobic digester. Food to microorganism (F/M) ratio indicates volatile solids loading into the system. A high F/M ratio may be inhibitory or toxic, whereas a low F/M ratio may prevent the induction of enzymes necessary for biodegradation [11]. However, the optimum F/M ratio for anaerobic digestion depends on substrate type. The optimum F/M ratios for the anaerobic digestion of fish offal waste, Chinese cabbage waste and synthetic wastewater were 0.2, 0.57-0.68 and 2.00, respectively [11-13]. Previous works have studied biogas production potential of refinery waste sludge under anaerobic digestion [7], however, less attention has been paid to the effects of F/M ratio on biogas production from refinery waste sludge in order to reduce the hazardous waste volume for final disposal via landfill.

Biosolid (sludge) from the wastewater treatment process, which includes a large quantity of organic substances, minerals, trace elements and nutrients, is usually applied and composted on agricultural soils. It can, however, contain hazardous components such as heavy metals, persistent organic contaminants that adversely affect human health, microbial communities, and soil functionality [14]. Tests for phytotoxicity are a helpful method for the control of sludge waste used in agriculture. This method is a tool for detecting substances that are harmful to plant or environment such as metals and organic compounds (polychlorinated biphenyls and polycyclic aromatic hydrocarbons). Seed germination and growth inhibition are some of the most often tested factors for the assessment of the phytotoxicity of biosolids [15]. There is very little information in the literature concerning the effects of the toxicity of anaerobic sludge from refinery waste.

The aims of this study were to: (i) evaluate the optimized F/M ratio for anaerobic digestion of activated sludge from a refinery wastewater treatment plant in order to enhance the highest biogas yield and anaerobic biodegradability, and (ii) determine the effect of F/M ratio of treated anaerobic sludge on the phytotoxicity level.

2. Materials and Methods

2.1 Characterization of feedstock and inoculum composition

Refinery waste sludge (excess activated sludge) was collected from an oil refinery company in Thailand. The inoculum was obtained from an anoxic tank of the wastewater treatment plant in Thailand. Prior to use, the sludge was mixed homogeneously and screened to remove debris and large particles (larger than 5 mm). The refinery waste sludge and inoculum were stored at a temperature of 4°C for future use. The moisture, TS, and VS were analyzed using the standard methods as presented in APHA [16]. The total COD (TCOD) and soluble COD (SCOD) were measured using the closed reflux digestion method. Briefly, a sample was refluxed in strongly acid solution with a known excess of potassium dichromate ($K_2Cr_2O_7$). After digestion, the remaining unreduced $K_2Cr_2O_7$ was titrated with ferrous ammonium sulfate to determine the amount of $K_2Cr_2O_7$ consumed, and the oxidizable organic matter was calculated in term of oxygen equivalent. For the SCOD, the samples were previously passed through a Whatman filter (0.45 µm) and the filtrate was applied for analysis.

2.2 Experiment design

A laboratory-scale anaerobic batch was conducted to evaluate the biodegradability of refinery waste sludge. The anaerobic digester used in this experiment was made of serum glass bottled with a total volume of 125 ml and a working volume of 100 ml [17]. The refinery activated sludge (RAS) was used as feedstock and four different mixing ratios of F/M (based on volatile solids, VS) set at 0.25, 0.50, 1.00 and 2.00 were used, to determine the optimum portion of feedstock and inoculum. After adding the requisite feedstock and inoculum, distilled water was added to adjust the volume to 100 ml, and pH was adjusted to 7.00±0.50. For each set triplicate, anaerobic digestions were conducted. All anaerobic digesters were purged with N_2 to remove O_2 from the system. All bottles were closed with rubber stoppers and incubated under 35±2°C. The production of biogas from the digesters was determined by the water displacement method. Head space gas was withdrawn by gas tight syringe. The composition of biogas was analyzed using a gas chromatograph instrument (SCION 456-GC, Bruker) in tandem with a flame ionization detector (FID). The temperatures of the injector, detector and oven temperatures were: 200°C, 250°C and 150°C, respectively. Helium (99.995%) carrier gas was used at a flow rate of 1.0 ml/min.

2.3 Phytotoxicity assay

The phytotoxicity was evaluated on refinery activated sludge (RAS) and biosolids that came from anaerobic digestion in different conditions after 50 days of the experimental period. The phytotoxicity test was analyzed by measuring the germination index (GI) of seeds of *Vigna radiate* (mung bean), *Lycopersicon esculentum* (tomato) and *Brassica rapa* (Chinese cabbage) according to the method of Tiquia *et al*. [18]. Briefly, water extracts were prepared by shaking the sludge sample with distilled water at 1:10 w/v for 10 min, then filtered. Ten seeds of each plant type were placed on filter paper in Petri dishes and 10 ml of filtrate was added. Controls with 10 ml of distilled water were added as a reference. After 72 h of incubation in the dark, the percentages of relative seed germination (RSG), relative root growth (RRG) and germination index (GI) were calculated as follows [19]:

$$RSG = \frac{\text{Number of germinated seeds (sample)}}{\text{Number of germinated seeds (control)}} \times 100\% \quad (1)$$

$$RRG = \frac{\text{Total radicle length of germinated seed (sample)}}{\text{Total radicle length of germinated seed (control)}} \times 100\% \quad (2)$$

$$GI = RSG \text{ x } RRG \text{ x } 100\% \quad (3)$$

2.4 Statistical analysis

All analytical parameters were obtained in triplicates, and obtained data were subjected to analysis of variance (One-way ANOVA). The results were expressed as mean ± standard deviation.

3. Results and Discussion

3.1 Feedstock and inoculum

Table 1 gives the characteristics of refinery waste sludge and inoculum prior to the anaerobic digestion process. The total solids (TS) and volatile solids (VS) values of RAS were higher than those of inoculum. Similarly, the VS/TS of RAS (82.16%) was higher than that of inoculum (57.92%). The high VS/TS ratio of feedstock indicated an adequate amount of VS in the feedstock to facilitate the rapid rate of anaerobic hydrolysis. Methane gas volumes were related to the amount of both TS and VS digested during anaerobic digestion [20]. Karim *et al.* [21] found a higher VS/TS ratio gave the higher biogas production. Compared with other sludge types, the VS/TS of RAS was higher than dewatered sewage sludge, which was 75.41% [20]. Then, RAS was more suitable for an anaerobic digestion than dewatered sewage sludge. Normally, VS/TS ratio of a substrate more than 80% was a potential feedstock for the anaerobic digestion [22]. The refinery waste sludge contained high nitrogen. The C/N ratio of RAS was 5.91, which was similar to the C/N ratio of wastewater treatment sludge (ranged between 5 and 10). However, C/N ratio of RAS was not located in the range of suggested optimum C/N ratio of substrate (20-30) for anaerobic digestion [23].

Table 1. Characteristic of refinery waste sludge and inoculum

Parameter	Refinery activated sludge (RAS)	Inoculum
Total solid (%)[a]	9.70±0.21	1.83±0.11
Volatile solid (%)[a]	7.97±0.44	1.06±0.23
VS/TS (%)	82.16	57.92
Moisture content (%)[a]	90.30±0.31	98.17±0.11
Carbon (%)[b]	32.33	-
Nitrogen (%)[b]	5.47	-
C/N	5.91	-
pH	7.71	6.86

[a] As the total weight of the sample
[b] As the total solid of the sample

3.2 Effects of the F/M ratio on treatment efficiency and biogas production

CODs and VS removal are determined to evaluate the treatment efficiency of the anaerobic digester system. The initial CODs and VS are a factor that relates to the total levels of organic matter in the feedstock [24]. The results showed that the initial TCOD concentrations ranged from 27,000-45,000 mg/l, and SCOD concentration was between 25,000-30,000 mg/l; increasing the F/M ratio increased the CODs values (Table 2). The SCOD/TCOD ratio of all conditions was high (0.66-0.92) which indicated that a huge amount of organic matter was ready for use in the dissolved form. For VS, the trend of the initial VS value also increased by increasing the F/M ratio. These results demonstrated that increasing the F/M ratio increased organic matter in the feedstock.

Table 2. The TCOD, SCOD and VS values of each experimental design

F/M ratio	RAS (g_{vs})	Inoculum (g_{vs})	Substrate loading (g_{vs}/l)	Initial		
				TCOD (mg/l)	SCOD (mg/l)	VS (mg/l)
0.25	0.15	0.61	1.50	27,000±2,000	25,000±1,500	1,500±250
0.50	0.30	0.61	3.00	30,000±1,600	26,000±1,000	3,000±102
1.00	0.61	0.61	6.10	38,666±1,300	28,000±500	6,100±112
2.00	1.22	0.61	12.20	45,000±1,300	30,000±1,200	12,200±354

One of the important parameters is pH that can affect acidogenic and methanogenic activities. The pH of refinery waste sludge should be located in the optimal pH range of anaerobic digestion (6.50-8.20) [25]. In this study, the variation in pH during the experimental period is shown in Figure 1. The initial pH values of all F/M ratio digesters were adjusted to 7.00±0.50. The pH of all digesters dropped to 6.3-6.7 within 10 days. This was probably because substantial amount of volatile fatty acids was generated due to the high organic content in the system. Volatile fatty acids or short-chain fatty acids are the primary intermediate products produced in an anaerobic digester [26]. The pH of all digesters began to increase after 10 days to reach neutral. However, the influent pH of all digesters varied between 6.30 and 7.42, values which were within the range of the optimum pH level for anaerobic digestion processes [25, 27]. The pH range for hydrolysis and acetogenesis are 6.0 and 6.0-7.0, while the pH range for methanogenesis is almost 6.5-7.5 [27].

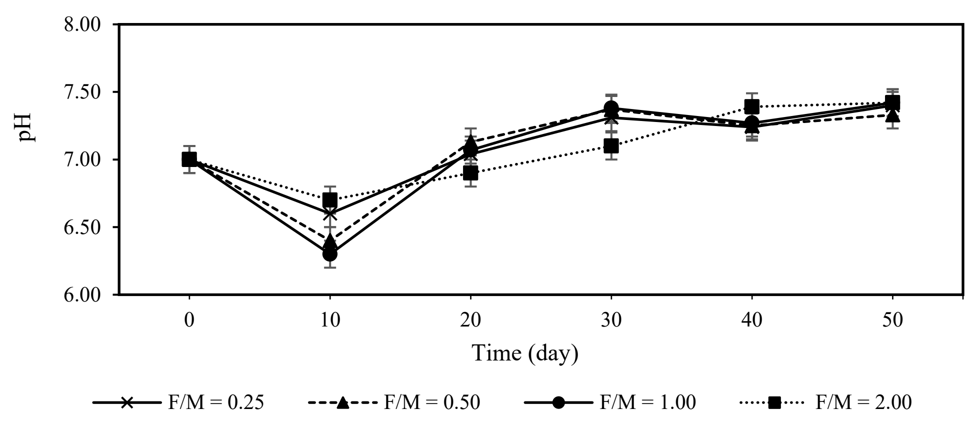


Figure 1. Variation of pH values in various batch reactors at different F/M ratio

Considering the biogas production, the biogas production volume was lower when the F/M ratio increased within 10 days (Figure 2). This result showed that during the first week of digestion, a higher F/M ratio showed some lag phase and the production of biogas was hindered due to the relatively low concentration of seed microorganisms. After two weeks, biogas production volume at high F/M ratio was higher than at lower F/M ratios except at the F/M ratio of 2.00. The biogas production from the F/M ratios of 0.50, 1.00 and 2.00 continued to rise after 28 days, while biogas production from F/M at the value of 0.25 decreased. The durations of the exponential phase at F/M of 0.50 and 1.00 were 36 days, whereas the durations of the exponential phase at F/M of 0.25 and 2.00 were 26 days. The shorter exponential phase duration occurred due to the nutritious substrate being limited [13]. For the values of F/M of 0.50, 1.00 and 2.00, the digesters took 32 days to reach 80% of their final biogas productions, which was longer than the digesters with the F/M value of 0.25. The biogas yield, methane production and methane content at F/M ratios of 0.25, 0.50, 1.00 and 2.00 were significantly different (Table 3). The maximum final biogas yield (147.98±7.40 ml/g_{vs}), methane production (51.41±1.78 ml/g_{vs}) and methane content (42.00±5.90 %) were observed in the digester with the F/M ratio of 1.00, followed by 0.50, 0.25 and 2.00, respectively. It was evident that increasing the F/M ratio could improve biogas yield. Similar trends were observed by Haak *et al*. [7], who reported that biogas production from anaerobic digestion of DAF sludge increased with increasing F/M ratio from 0.01 to 0.20. On the other hand, when the F/M ratio increased from 1.00 to 2.00, biogas yield was reduced because organic substrates surpassed the requirement needed by a microorganism leading to metabolic imbalance [13]. In addition, the reduction of biogas production at excessive F/M ratios could occur due to microbial inhibition from toxic or hazardous organic and inorganic pollutants in RAS. It is well known that heavy metals and organic pollutants are usually found in refinery waste [28]. The existence of oxygen, heavy metals or too high levels of VFA could cause low biogas production and be the cause of anaerobic digester failure [29]. However, the optimum F/M ratio value depends on the type of waste. The maximum biogas production from fish offal waste was obtained when the F/M ratio was 0.2 [13]. Prashanth *et al*. [11] studied the F/M values range from 0.18 to 2.0 of food waste and found that the highest value was between 0.57-0.68. The biogas yield from anaerobic digestion of Chinese cabbage waste increased from 591 to 677 ml/g_{vs} when the F/M ratio was increased from 0.50 to 2.00 [12]. The biogas yield from activated sludge obtained from the wastewater treatment plant in the refinery was similar to the results obtained from previous studies investigating the anaerobic digestion of activated sludge from the wastewater treatment plant in the municipality. The production of biogas from waste activated sludge collected from an urban wastewater treatment plant amounted to 120-190 ml/g_{vs} [30, 31]. Compared with other waste types, the biogas production obtained from RAS was lower than biogas production obtained from sugar beet waste (271.26 ml/g_{vs}), Chinese cabbage (591-677 ml/g_{vs}), walnut peel (226 ml/g_{vs}) and food waste [12, 32, 33]. It could be explained that the unbalanced carbon to nitrogen (C/N) ratio of waste activated sludge limited the potential of biogas production from mono-digestion of waste activated sludge [34].

3.3 Phytotoxicity

Germination index (GI) is given by the following criterion: GI values lower than 50% are highly phytotoxic, between 50-80% are moderately phytotoxic, higher than 80% are no-phytotoxic, and higher than 100% have phytonutrient or phytostimulant effects [35]. GI is shown in Figure 3. A negative effect on the germination and the root growth of the three plants was detected in the inoculum and the refinery activated sludge (RAS); the GI values of the inoculum and RAS ranged from 45% to 80% for all three plants. The inoculum was moderately phytotoxic for all the three plants. RAS was moderately phytotoxic for *Lycopersicon esculentum* and *Brassica rapa*, while

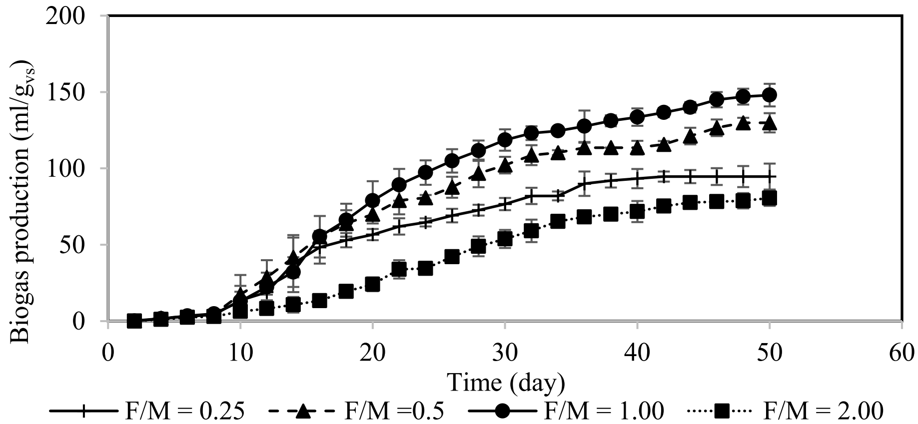
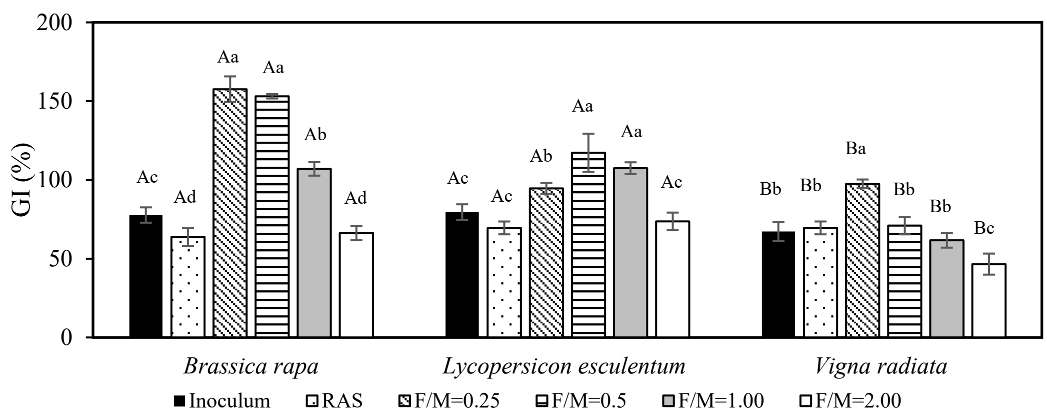


Figure 2. Cumulative biogas production in different F/M ratios during the experimental period
Note: mean value±SD of triplicate samples

Table 3. The value of CODs and VS removal, cumulative of biogas production and methane production, and CH_4 content for different F/M ratio.

F/M	Removal efficiency (%)			Cumulative Biogas production (ml/g$_{vs}$)	Cumulative methane production (ml/g$_{vs}$)	Max. CH_4 content
	TCOD	SCOD	VS			
0.25	60.96±4.29^a	82.67±2.52^a	61.21±2.80^a	98.64±8.50^c	22.71±1.75^c	32.20±5.50^a
0.50	62.17±3.97^a	82.72±2.97^a	65.77±3.33^a	125.64±6.30^b	27.85±1.70^b	35.50±8.20^a
1.00	65.60±4.97^a	83.78±1.78^a	69.10±3.65^a	147.98±7.40^a	51.41±1.78^a	42.00±5.90^a
2.00	43.33±5.03^b	73.45±3.20^b	35.50±3.12^b	84.09±5.10^d	16.43±2.65^d	25.36±7.40^b

Note: Different superscript letters in the same column indicate significant differences at $p<0.05$.



Note: A,B = significant differences ($p<0.05$) among plant species.
a,b,c,d = significant differences ($p<0.05$) among biosolid types within the same plant species

Figure 3. Germinated index of *Brassica rapa*, *Lycopersicon esculentum* and *Vigna radiata* seeds germinated in aqueous extracts of the inoculum, RAS and biosolid from the anaerobic digesters in different F/M ratio

RAS was highly phytotoxic for *Vigna radiata*. The wastewater from refining processing contains a high amount of hydrocarbons. After a series of treatment methods (physical, chemical and biological treatment), the organic content is extremely reduced. Consequently, the main component of refinery activated sludge from the refinery wastewater treatment plant is a residual hydrocarbon that may be a toxic compound for plant growth [28]. Similar to sewage sludge, the GI values of *Lepidium sativum L*. was 20-40% for sewage sludge, but the average GI value was 81% for sewage sludge compost [36]. The GI value of *Sinapis alba* increased from 60 to 80 % when the concentration of sewage sludge in soil was increased from 10-25% [37]. The GI values of biosolids from anaerobic digester with F/M at 0.25, 0.50 and 1.00 ratios were significantly higher than RAS ($p<0.05$). This outcome suggested that the process of anaerobic digestion could decrease phytotoxicity as the anaerobic microorganism could degrade some toxic or organic compounds. Elements such as calcium, copper, iron, magnesium, nitrogen, phosphate, phosphorus, potassium, sodium and zinc are found in RAS [38]. Some of them are essential elements for plant growth. Thus, increasing the amount of RAS via increasing F/M ratio (0.25-1.00) in the anaerobic digestion system could stimulate root elongation and seed germination of *Lycopersicon esculentum* and *Brassica rapa* (GI>100) due to the increased levels of phytonutrients. However, increasing the excess amount of RAS (F/M ratio = 2.00) could inhibit seed germination and root elongation (GI<80). This can be explained by RAS composition. Beside hydrocarbons and essential elements, refinery activated sludge also contains heavy metals such as Ag, Cr, Mn and Hg, which are toxic even at low concentrations [26]. A negative effect on the germination and the root growth of *Vigna radiata* was detected in all F/M ratios. The GI values of *Vigna radiata* was significantly lower than those of *Lycopersicon esculentum* and *Brassica rapa* ($p<0.05$).

Usually, sludge from the wastewater treatment system is used as raw material in the composting process. The level of phytotoxicity may be reduced under the composting process. GI values showed an increasing trend from the beginning of the composting process, which was also reported in the composting of animal manures [39], and co-composting of sewage sludge with the organic fraction of municipal solid waste [40]. The GI value increased above 80% at the end of the composting process in the case of sewage sludge, thereby reducing phytotoxicity. It should be noted that many factors can affect germination and that percentage of germinated seeds is not a guarantee of proper application or high crop quality yield [37].

4. Conclusions

The outcomes of this research showed that the efficiency of treatment and yield of biogas production increased when the F/M ratio increased from 0.25 to 1.00 and the efficiency of treatment and yield of biogas production decreased when the F/M ratio increased from 1.00 to 2.00. The highest final biogas yield (147.98±0.74 ml/g_{vs}) was observed in the digester with an F/M ratio of 1.00, followed by 0.50, 0.25 and 2.00, respectively. The optimum F/M ratio for the anaerobic digestion of RAS was 1.00, which gave the highest biogas yield and treatment efficiency. For phytotoxicity, anaerobic digestion could reduce the phytotoxic level, reflected in increases in GI values when compared with the raw substrate (RAS). An increasing amount of RAS with increasing F/M ratio (0.25-1.00) could stimulate *Lycopersicon esculentum* and *Brassica rapa* growth (GI>100), but an excessive increase in the amount of RAS (F/M ratio = 2.00) inhibited plant growth (GI<80). A negative effect on the germination and the root growth of *Vigna radiata* was found for all F/M ratios. The results indicated that refinery activated slude can potentially be used for biogas production under anaerobic digestion. The results obtained from this lab-scale experiment provide fundamental data crucial to the design and operation of an anaerobic digester for refinery activated sludge and to maximize biogas production and treatment efficiency. Apart from the F/M ratio, several factors influence the behavior of bacteria, such as temperature, C/N ratio, digester type, anaerobic digester size and pH. Then, in order to increase biogas efficiency in the future work, these factors should be examined. Further phytotoxicity research is required to ensure that bio-solids from the anaerobic digestion of refinery waste are safe to use in soil treatment and modifications.

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References

[1] Hu, G., Li, J. and Zeng, G., 2013. Recent development in the treatment of oily sludge from petroleum industry: A review. *Journal of Hazardous Materials,* 261, 470-490.

[2] Ahmadi, M., Tamimi, Z., Jaafarzadeh, N., Teymouri, P. and Maleki, R., 2016. Characteristics and disposal options of sludges from an oil refinery wastewater treatment plant in Iran, 2013. *Archives Hygiene Sciences*, 5(1), 39-46.

[3] Hasani, F. and Nabhani, N., 2016. Waste management system in petroleum refinery. *International Journal of Advanced Biotechnology and Research*, 7(3), 1446-1452.

[4] Berktay, A. and Nas, B., 2007. Biogas Production and Utilization Potential of Wastewater Treatment Sludge. *Energy Sources, Part A: Recovery, Utilization, and Environmental Effects*, 30(2), 179-188.

[5] Demirbas, A., Taylan, O. and Kaya, D., 2016. Biogas production from municipal sewage sludge (MSS). Energy Sources, *Part A: Recovery, Utilization, and Environmental Effects,* 38(20*)*, 3027-3033.

[6] Roy, R., Haak, L., Li, L. and Pagilla, K., 2016. Anaerobic digestion for solids reduction and detoxification of refinery waste streams. *Process Biochemistry*, 51(10),1552-1560.

[7] Haak, L., Roy, R. and Pagilla, K., 2016. Toxicity and biogas production potential of refinery waste sludge for anaerobic digestion. *Chemosphere*, 144, 1170-1176.

[8] Miron, Y., Zeeman, G., Van Lier, J.B. and Lettinga, G., 2000. The role of sludge retention time in the hydrolysis and acidifcation of lipids, carbohydrates and proteins during digestion of primary sludge in CSTR systems. *Water Research*, 34(5), 1705-1713.

[9] Wang, Q., Liang, Y., Zhao, P., Li, Q.X., Guo, S., Chen, C., 2016. Potential and optimization of two-phase anaerobic digestion of oil refinery waste activated sludge and microbial community study. *Scientific Reports*, 6, https://doi.org/10.1038/srep38245.

[10] Ferreiro, N. and Soto, M., 2003. Anaerobic hydrolysis of primary sludge: influence of sludge concentration and temperature. *Water Science and Technology*, 47(12), 239-426.

[11] Prashanth, S., Kumar, P. and Mehrotra, I., 2006. Anaerobic degradability: Effect of particulate COD. *Journal of Environmental Engineering-ASCE*, 132(4), 488-496.

[12] Kafel, G.K., Bhattarai, S., Kim, S.H. and Chen, L., 2014. Effect of feed to microbe rations on anaerobic digestion of Chinese cage waste under mesophilic and thermophilic conditions: Biogas potential and kinetic study. *Journal of Environmental Management*, 133, 293-301.

[13] Hadiyarto, A., Budiyono, Johari, S., Hutama, I. and Hasyim, W., 2015. The effect of F/M ratio to the anaerobic decomposition of biogas production from fish offal waste. *Waste Technology*, 3(2), 58-62.

[14] Sindelar, O., Adamcova, D., Zloch, J. and Vaverkova, M.D., 2020. Phytotoxicity of sewage sludge from selected wastewater treatment plant-new opportunities in sewage sludge treatment. *International Journal of Recycling of Organic Waste in Agriculture*, 9, 75-83.

[15] Oleszczuk, P., 2008. Phytotoxicity of municipal sewage sludge composts related to physico-chemical properties, PAHs and heavy metals. *Ecotoxicology and Environmental Safety*, 69, 496-505.

[16] APHA, 2005. *Standard Methods for the Examination of Water and Wastewater*. 21st ed. Washington D.C: American public Health Association.

[17] Anuar, N.K., Man, H.C., Idrus, S. and Daud, N.N.N., 2017. Biochemical methane potential (BMP) from anaerobic co-digestion of sewage sludge and decanter cake. *IOP Conf. Series: Materials Science and Engineering*, 368, https://doi.org/10.1088/1757-899X/368/1/012027.

[18] Tiquia, S.M., Tam, N.F. and Hodgkiss, I.J., 1996. Effects of composting on phytotoxicity of spent pig-manure sawdust little. *Environmental Pollution*, 93, 249-256.

[19] Luo, Y., Liang, J., Zeng, G., Chen, M., Mo, D., Li, G. and Zhang, D., 2018. Seed germination test for toxicity evaluation of compost: its roles, problems and prospects. *Waste Management*, 71, 109-114.

[20] Hao, Z., Yang, B. and Jahng, D., 2018. Combustion characteristics of biodried sewage sludge. *Waste Management*, 72, 296-305.

[21] Karim, K., Klasson, K.T., Hoffmann, R., Drescher, S.R., DePaoli, D.W. and Al-Dahhan, M.H., 2005. Anaerobic digestion of animal waste: Effect of mixing. *Bioresource Technology*, 96(14), 1607-1612.

[22] Zhang, R., El-Mashad, H.M., Hartman, K., Wang, F., Liu, G., Choate, C. and Gamble, P., 2007. Characterization of food waste as feedstock for anaerobic digestion. *Bioresource Technology*, 98(4), 929-935.

[23] Mao, C., Feng, Y., Wang, X. and Ren, G., 2015. Review on research achievements of biogas from anaerobic digestion. *Renewable and Sustainable Energy Reviews*, 45, 540-555.

[24] Hobbs, S.R., Landis, A.E., Rittmann, B.E., Young, M.N., Parameswaran, P., 2018. Enhancing anaerobic digestion of food waste through biochemical methane potential assays at different substrate: inoculum ratios. *Waste Management*, 71, 612-617.

[25] Speece, R.E., 2008. Anaerobic biotechnology for industrial wastewater treatment. *Environmental Science and Technology*, 17(9), 416-427.

[26] Pramanik, S.K., Suja, F.B., Zain, S.M. and Pramanik, B.K., 2019. The anaerobic digestion process of biogas production from food waste: prospects and constraints. *Bioresource Technology Reports*, 8, https://doi.org/10.1016/j.biteb.2019.100310.

[27] Leung, D.Y.C. and Wang, J., 2016. An overview on biogas generation from anaerobic digestion of food waste. *International Journal of Green Energy*, 13(2), 119-131.

[28] Ali, A.M., Abu-Hassan, M.A., Ibrahim, R.R., Zaini, M.A.A., Abdulkarim, B.I., Hussein, A.S., Su, S.M. and Halim, M.A.I.M., 2017. Characterization of petroleum sludge from refinery industry biological wastewater treatment unit. *The International Journal of Engineering and Science*, 6(9), 61-65.

[29] Polprasert, C., 1996. *Organic Waste Recycling-Technology and Management*. 2nd ed. West Sussex: John Wiley and Sons.

[30] Athanasoulia, E., Melidis, P. and Aivasidis, A., 2012. Optimization of biogas production from waste activated sludge through serial digestion. *Renew Energy*, 47, 147-151.

[31] Maamri, S. and Amrani, M., 2014. Biogas production from waste activated sludge using cattle dung inoculums: Effect of total solid contents and kinetics study. *Energy Procedia*, 50, 352-359.

[32] Liu, J.H., Zhang, W.D., Liu, S.Q., Zhao, X.L., Yin, F., Liu, J., Xu, L., Chen, Y.B. and Yang, H., 2014. The effect of food-microorganism (F/M) ratio on gas properties in batch biogas fermentation with walnut peel. *Advanced Materials Research*, 937, 291-296.

[33] Keramati, M. and Beiki, H., 2017. The effect of pH adjustment together with different substrate to inoculum ratios on biogas production from sugar beet wastes in an anaerobic digester. *Journal of Energy Management and Technology*, 1(2), 6-11.

[34] Yang, Q., Wu, B., Yao, F., He, L., Chen, F., Ma, Y., Shu, X., Hou, K., Wang, D. and Li, X., 2019. Biogas production from anaerobic co-digestion of waste activated sludge: co-substrate and influencing parameters. *Reviews in Environmental Science and Biotechnology*, 18, 771-793.

[35] Emino, E.R. and Warman, P.R., 2004. Biological assay for compost quality. *Compost Science and Utilization*, 12(4), 342-348.

[36] Siles-Castellano, A.B., López, M.J., López-González, J.A., Suárez-Estrella, F., Jurado, M.M., Estrella-González, M.J. and Moreno, J., 2020. Comparative analysis of phytotoxicity and compost quality in industrial composting facilities processing different organic wastes. *Journal of Cleaner Production*, 252, https://doi.org/10.1016/j.jclepro.2019.119820.

[37] Fijalkowski, K. L. and Kwarciak-Kozlowska, A., 2020. Phytotoxicity assay to assess sewage sludge phytoremediation rate using guaiacol peroxidase activity (GPX): A comparison of four growth substrates. *Journal of Environmental Management*, 263, https://doi. org/10.1016/ j. jenvman.2020.110413.

[38] Johnson, O.A. and Affam, A.C., 2019. Petroleum sludge treatment and disposal: A review. *Environmental Engineering Research*, 24(2), 191-201.

[39] Huang, J., Yu, Z., Gao, H., Yan, X., Chang, J., Wang, C., Hu, J. and Zhang, L., 2017. Chemical structures and characteristics of animal manures and composts during composting and assessment of maturity indices. *PLoS ONE*, 12(6), https://doi.org/10.1371/journal.pone. 0178110.

[40] Zhang, D., Luo, W., Li, Y., Wang, G. and Li, G., 2018. Performance of co-composting sewage sludge and organic fraction of municipal solid waste at different proportions. *Bioresource Technology*, 250, 853-859.