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Co-Digestion of Palm Oil Mill Effluent and Refined Glycerin Wash Water for Chemical Oxygen Demand Removal and Methane Production

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Abstract: Problem statement: Refined Glycerin Wash Water (RGWW) from the oleochemical industry contains high Chemical Oxygen Demand (COD) and requires proper treatment before disposal. Unfortunately the wash water also contains high concentration of sodium chloride (NaCl) that could cause inhibition to the normal biological treatment process. However, there is feasibility of co-digesting the RGWW and Palm Oil Mill Effluent (POME) for its treatment and methane recovery. **Approach:** A large 500 m^3 semi-commercial closed digester tank was used to study the effect of co-digesting POME and RGWW under mesophilic condition at different RGWW percentage. The digester performance in terms of COD removal efficiency and methane production rate and stability based on total Volatile Fatty Acids (VFA) accumulation, Mixed Liquor Volatile Suspended Solid (MLVSS) and pH were evaluated. **Results:** At 1.0% of RGWW co-digested, both COD removal efficiency and methane production rate showed satisfactory results with higher than 90% and 505 m^3 day^{-1}, respectively. However, once the percentage was increased to a maximum of 5.25%, COD removal efficiency remains high but the methane production rate reduced significantly down to 307 m^3 day^{-1}. At this stage, the digester was already unstable with high total VFA recorded of 913 mg L^{-1} and low cells concentration of 8.58 g L^{-1}. This was probably due to the effect of plasmolysis on the methanogens at high concentration of NaCl in the digester of nearly 4000 mg L^{-1}. **Conclusion:** Co-digesting of RGWW with high NaCl content and POME is satisfactory for COD removal but not for increasing the methane production.

Key words: Palm oil mill effluent, glycerin wastewater, anaerobic treatment, co-digestion, methane

INTRODUCTION

The upstream and downstream palm oil processing industry is rapidly expanding in Malaysia in order to meet the increasing demand of the world's oil and fats market. Despite huge economics return to the country, the industry also generates huge volume of liquid waste. The liquid waste known as Palm Oil Mill Effluent (POME) and Refined Glycerin Wash Water (RGWW) are generated at a rate of 0.5-0.75 tone of POME per tone of fresh fruit bunch processed from the palm oil mill and 1 tone per day of glycerol residues from the methyl ester production plant[1,2]. The liquid RGWW is basically produced from the refining process of the glycerol residue. These wastewaters although known to contains high amount of organic substances which is suitable for anaerobic treatment process for methane production, they also contains other chemicals which may affect the performance of the anaerobic treatment and methane production rate. For the case of RGWW, it also contains high level of sodium chloride (NaCl) and soap because the glycerol residue itself

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contains up to 64.3% of salt and 6.6% of fatty acids soaps[2]. In these wastewaters the high COD concentration in the effluent is mainly contributed by the soluble organics such as glycerin and soap while the NaCl is from the reaction of acid (HCl) and base (NaOH).

In many studies, the treatment technology for high salt or saline effluent has been studied for COD removal in laboratory scale digesters and pilot scale digesters[3-12]. Sodium ions (Na^+) appears to be essential for methanogenic bacteria due to its roles in a chemiosmotic coupling mechanism and 230 mg Na^+ L^{-1} or 10 mM was suggested as the optimal concentration for acetolastic methanogens in waste treatment process[3]. However, salts (NaCl) concentration above 1% which is regarded as high saline waters could cause loss of cell to dehydrate due to osmotic pressure[6]. In a study, the removal efficiency for soluble COD decreased from 94.8% at 150 mg L^{-1} of Cl^- to only 63% at 5,000 mg L^{-1} of Cl^-[10]. High COD removal efficiency of up to 94% has been reported for anaerobic treatment of saline wastewater by *Halanaerobium lacusrosei*[5]. In a Specific Methanogenic Activity (SMA) study, researchers found that at 25°C, high NaCl content of 30 g L^{-1} and above, the bacteria could not be acclimatized even in 50 days which may be due to osmotic stress which inhibited the reaction pathways of the degradation process[9]. This is supported with another study which found the critical salinity level was 3% for the methanogens in the mesophilic anaerobic digesters[7]. On the contrary, in a modern Submerge Anaerobic Membrane Reactor (SAMBR), researchers found the biomass could rapidly acclimatize to salinity of up to 40 g L^{-1} with 40-60% of Dissolved Organic Carbon (DOC) removal and satisfactory methane production rate[11]. In SAMBR or anaerobic Baffled Reactor (ABR), granule sludge is developed in the system which has better protection for the methanogens against inhibition conditions. The sludge granules in the ABR system usually contained a large amount of organics, amorphous materials and crystals of Fe_2O_3, FeS, $CaCO_3$, filamentous bacteria and extracellular polymeric substances[12]. In a study on sludge sample obtained from anaerobic digester treating POME, the filamentous bacteria is known as *Methanosaeta concilii*[13].

Recent introduction of Clean Development Mechanism (CDM) which enables Certified Emission Reduction (CER) to be traded to Annex 1 countries has attracted many industries including oleochemical industry which produces wastewater with high COD and NaCl to investigate the feasibility of adopting such concept in their wastewater treatment plant. The advantages include reduction of the operating cost from CER, reduction of the green house gas emission, better waste management and reduction of air pollution by converting the aerobic process to the closed anaerobic process. The idea of anaerobic digestion offers several advantages and an ideal solution for organic waste treatment for the production of useful methane gas as a valuable product, low volume of sludge generation which can be used as fertilizer and it is a low energy requirement process[14]. In Malaysia, anaerobic treatment is a very popular treatment method for the Palm Oil Mill Effluent (POME) by using either open lagoon or closed anaerobic tank systems[1,15]. The anaerobic degradation process of organic matters occurs in four metabolic stages namely hydrolysis, acidogenesis, acetogenesis and methanogenesis and there are two groups of bacteria that responsible for the complete conversion of organic substances to methane gas which is the acidogens and methanogens[16,17]. In many studies, co-digestion was adopted to improve the biogas yield in anerobic treatment of organic wastes such as sisal pulp and fish wastes[18], sludge and fruit and vegetable wastes[19], pre-treated barley waste and activated sludge[20] and grease trap and sewage sludge[21]. However none of the studies utilized the wastewater from the glycerin residue refining process co-digested with POME. Therefore the aim of this study is to investigate the feasibility of co-digesting POME and RGWW with high NaCl for COD removal and methane gas production for renewable energy.

MATERIALS AND METHODS

The system set-up and operation: Figure 1 shows the set-up of the system complete with a holding tank and a sludge settling tank. The digester system was equipped with sampling port, temperature and pH probes, mixing pump, biogas mass flow meter and POME mass flow meter. The POME was obtained directly from the mill by pumping and the RGWW was obtained from an oleochemical company located in Kuantan, Pahang, approximately 200 km away from the site. The wastewater was transported to the plant in either 1 m^3 plastic tank or 30 m^3 tanker. The basic characteristic of the RGWW delivered is as follow; NaCl 2.86-3.26%, soap 3.89-4.84%, low level glycerin 0.32-3.26% and total COD 63,500-84,000 mg L^{-1}. The POME was pumped from the mill and stored in the holding tank. The volume of RGWW required according to the percentage was then added into the holding tank and mixed homogenously prior to feeding using a centrifugal pump.

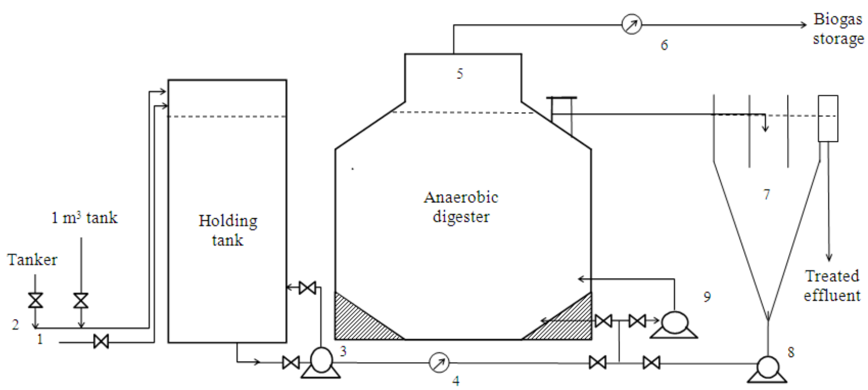

Fig. 1: Schematic diagram of the closed digester complete with a holding tank and a sludge settling tank. (1): POME inlet; (2): Refined glycerin wash water inlet; (3): Feeding pump; (4): Endress+Hauser mass flow meter; (5): Biogas chamber; (6): Endress+Hauser biogas mass flow meter; (7): Sludge settling tank; (8): Sludge recycling pump; (9): Mixing pump

Table 1: The feeding profiles of the mixture of RGWW and POME in terms of co-digestion percentage, COD concentration, feeding rate, OLR and pH

Operation period Days	Percentage of RGWW co-digested volume (%)	Feeding rate of the mixture fed[b] m^3 day^{-1}	COD range of the mixture[b] mg L^{-1}	OLR kgCOD m^{-3} day^{-1}	pH range of the mixture[b] -
1-13	1.0	39.4-51.5	47.9-64.1	5.0	4.2-4.9
14-22	2.0	30.6-49.0	51.0-81.3	5.0	4.2-4.6
23-33[a]	3.0	50.0	26.7-36.0	2.7-3.6	4.5-5.1
34-60[a]	4.0	20.0-50.0	30-95.6	2.6-5.1	4.5-5.7
61-77	5.0	26.5-32.5	74.9-94.9	5.0	4.5-5.0
78-85	5.25	28.9-35.5	74.0-90.8	5.25	4.6-5.0

[a]: During these periods high rainfall was recorded which resulted in diluted POME and the OLR could not be maintained at 5.0 kgCOD m^{-3} day^{-1} due to maximum HRT that could be applied was only 10 days; [b]: Mixture refers to the mixture of POME and RGWW

The digester was fed daily at different organic loading rate (OLR) and tested at maximum OLR of 5.25 kgCOD m^{-3} day^{-1}. The sludge from the settling tank was recycled at a rate of 6 m^3 day^{-1} throughout the period under study.

Chemical analyses: Chemical Oxygen Demand (COD), total Volatile Fatty Acids (VFA), alkalinity, Mixed Liquor Volatile Suspended Solid (MLVSS) and NaCl were performed according to the APHA Standard Methods for the Examination of Water and Wastewaters[22]. The POME fed was measured using an electromagnetic flow meter (PROline promag 50, Endress+Hauser, Germany) and the biogas was measured using a thermal mass flow meter (T-Mass AT70, Endress+Hauser, Germany). The methane concentration was measured using a calibrated portable methane gas analyzer (XP-314A, Shin-Cosmos Electric Co. Ltd, Japan). The pH was measured using HANNA pH/ORP/Temperature meter (HI 991002, HANNA Instrument, Romania).

The feeding profiles: Table 1 shows the feeding profiles in terms of the percentage of RGWW co-digested (%), the mixture feeding rate (m^3 day^{-1}), the COD range of the mixture (mg L^{-1}), the pH of the mixture and OLR applied (kg COD m^{-3} day^{-1}) to the system throughout the period under study. The total period of study was recorded for 85 days in which RGWW was co-digested at different percentages while the OLR was kept at 5.0 kg COD m^{-3} day^{-1} except on the days 23-33 where the feeding rate was fixed at 50 m^3 day^{-1} which equivalent to 10 days of HRT. During that period heavy rainfall was recorded and resulted in diluted POME and the feeding rate was fixed in order to avoid shock loading to the system and washout of microorganisms especially the methanogens. The COD of the mixture was recorded between 26,700 and 36,000 mg L^{-1} in which lower than normal values. The percentage of RGWW co-digested was initially fixed at 1.0% in order to acclimatize the microorganisms to a new environment before being steadily increased to 2.0% from days 14-22, 3.0% from days 23-33, 4.0% from days 34-60, 5.0% from days 61-77 and lastly to 5.25% from days 78-85. Although the pH value for the RGWW was extremely alkaline (pH 12.0-13.0), it has no influence on the pH of the mixture as clearly observed in Table 1. The pH range of the mixture was recorded between 4.2 and 5.7 which are normal to this anaerobic treatment process.

RESULTS

Table 2 shows the results for the methane (CH_4) composition in biogas and the yield at different percentage of RGWW co-digested throughout the study period of 85 days. The percentage was slowly increased from 1-5.25% at the increment of 1% except for the last stage where increment was only 0.25% due to problem of NaCl accumulation. The results of CH_4 composition in biogas and yield is presented in the range and mean with its corresponding standard deviation (SD). The theoretical yield of CH_4 based on the COD could be calculated from the formula $CH_4 + O_2 \rightarrow CO_2 + H_2O$ to give the yield of 0.25 kg CH_4 kgCODremoved^{-1} or 0.35 m^3 CH_4 kgCODremoved^{-1}[23]. As clearly observed, the CH_4 composition in the biogas and yield reduced from 59% and 0.15 kgCH_4 kgCODremoved^{-1}, respectively to 47% and 0.09 kgCH_4 kgCODremoved^{-1} at the last stage of RGWW co-digested. At 1% of RGWW co-digested, the yield was 0.15 kgCH_4 kgCODremoved^{-1} which represents only 60% of the theoretical yield. At 5.25% RGWW co-digested the yield dropped down to 0.09 kgCH_4 kgCODremoved^{-1} which is only 36% of the theoretical yield.

Table 3 shows the performance of the digester and its stability recorded throughout the study period in terms of COD removal efficiency, production rate of biogas and methane, total VFA, alkalinity, pH and MLVSS. Throughout the study, the COD removal efficiency recorded satisfactory result of above than 90% removal which indicates suitable treatment method of the co-digestion mixtures (POME and RGWW). The biogas and CH_4 production rate on the other hand shows a declining trend towards the end of the study. Initially the production rates were 859 and 505 m^3 day^{-1} respectively, for biogas and CH_4 but reduced down to 576 and 307 m^3 day^{-1} at the last stage of the study period. The total VFA was observed to increase once RGWW co-digestion percentage was increased but the alkalinity measured as mg $CaCO_3$ L^{-1} remained stable except in days 23-33 when diluted POME was utilized due to heavy rainfall. The pH was recorded stable between 6.9 and 7.1 as a result of satisfactory alkalinity buffering capacity available in the digester to counter the effect of high VFA concentration. The cell concentration in terms of MLVSS fluctuated between 8.14 and 8.83 g L^{-1} inside the digester.

Table 2: The methane percentage and methane yield recorded

RGWW Co-digested (volume %)	Methane gas composition (%)[a]		Methane yield (kgCH_4 kgCODremoved^{-1})[b]	
	Range	Mean ± SD	Range	Mean ± SD
1.0	54-67	59±5	0.14-0.18	0.15±0.02
2.0	51-62	58±3	0.11-0.14	0.13±0.01
3.0	50-65	57±4	0.08-0.17	0.10±0.03
4.0	50-70	60±6	0.05-0.16	0.10±0.03
5.0	36-50	42±5	0.07-0.13	0.09±0.01
5.25	43-50	47±3	0.08-0.11	0.09±0.01

[a]: Volume methane by volume biogas percentage; [b]: Theoretical methane yield is 0.25 kgCH_4 kgCODremoved^{-1}

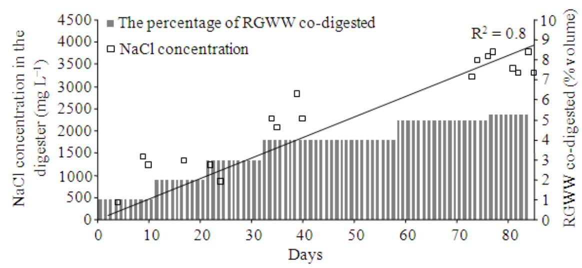

Fig. 2: The sodium salt (NaCl) accumulation in the digester at different percentage of Refined Glycerin Wash Water (RGWW) co-digested

Figure 2 shows the accumulation of sodium salt (NaCl) inside the digester at different percentage of RGWW co-digested. Initially when the RGWW was first introduced into the system, the concentration of NaCl accumulated was low at only 500 mg L^{-1}. However due to the accumulation effect, the NaCl concentration increased once higher percentage of RGWW was co-digested. At the end of the study, the NaCl concentration inside the digester increased to nearly 4000 mg L^{-1}. The NaCl accumulation inside the system followed the linear relationship (r^2 = 0.8) with the RGWW % co-digested. The contribution of NaCl inside the system was from RGWW as POME usually does not contain NaCl.

DISCUSSION

The performance of the digester: The CH_4 composition in biogas and yield at different percentage of RGWW co-digested is shown in Table 2. The digester performances and in terms of COD removal efficiency, biogas and methane production rate are given in Table 3. As clearly observed, the COD removal efficiency remains high of above 90% removal throughout the period under study which reflects good treatment performance of the digester. This result is consistent with previous study on POME without salinity addition[1,13] and other wastewater with salinity addition[5,10] In this study the percentage of RGWW co-digested was increased to a maximum of 5.25% and remarkably the high COD removal efficiency remained unchanged. This may be explained due to present of high biodegradability COD in the POME and RGWW which can be easily utilized by the microorganisms. In a study, the biodegradability rate constant decreased linearly with increase in fraction of particulate COD which confirmed the higher biodegradation rate of soluble COD by the microorganisms in comparison to particulate COD[24] Although it was reported that high salt concentration could interfere the COD measurement[6] the sample was satisfactorily diluted and the concentration of NaCl in the sample was low enough to cause interference to the COD reading. Thus, a reliable COD result was obtained.

Unlike COD removal efficiency performance, the CH_4 composition in biogas, CH_4 yield and CH_4 production rates were found to reduce as RGWW co-digestion percentages was increased with time as clearly shown in Table 2 and 3. Initially at 1% of RGWW co-digested, the biogas and CH_4 production rates were recorded high at 859 and 505 m^3 day^{-1} respectively, but reduced significantly once 5.25% of RGWW was co-digested. The biogas and CH_4 production rate was recorded only 700 and 400 m^3 day^{-1} respectively, at 3.0% of RGWW co-digested. The yield of CH_4 was also recorded low at only 0.1 kg COD m^{-3} day^{-1}. At this stage the feeding rate was fixed at 50 m^3 day^{-1} corresponding to Hydraulic Retention Time (HRT) of 10 days and too short for growth of methanogens in the digester. It was reported the *Methanosarcina* spp. exhibit a faster doubling time of 0.5-2.0 day on acetate than *Methanosaeta* spp. of 1.0-12.0 days[25]. Obviously with shorter HRT of 10 days, the available time was insufficient for *Methanosaeta* spp. which is commonly found in the modern anaerobic digesters. The quality of CH_4 in biogas was not much affected when RGWW percentage was increased from 3.0-4.0% which highlights the availability of active methanogens in the system for CH_4 production. This explains the positive effect of applying low OLR due to the utilization of diluted POME due to heavy rain. Unfortunately, the CH_4 yield still recorded low value at only 0.1 kg COD m^{-3} day^{-1} which is only 40% of the theoretical yield. The declining trend of CH_4 production and yield continued when OLR was fixed at 5.0 kg COD m^3 day^{-1} and RGWW co-digestion percentage was further increased to 5.0 and 5.25%. At this stage the CH_4 quality in biogas and production rate further reduced down to 47% and 307 m^3 d^{-1}, respectively. At this stage the CH_4 yield also dropped down to 0.09 $kgCH_4$ $kgCODremoved^{-1}$. This indicates a poor digester's performance in terms of methane gas production rate, quality and yield. This phenomenon could be explained by lost and reduction of survival protozoa and methanogenic archaea after exposing with high salt concentration resulted less sludge tendency for flocculates and plasmolysis occurred[26]. The good performance of the digester judged by COD removal, biogas and methane production rate was observed when the RGWW co-digested with low strength POME (diluted) during rainfall period.

The system stability: The digester stability could be observed by monitoring the key parameters such as total VFA accumulation, alkalinity accumulation, pH and the cell concentration in the treatment system (MLVSS)[27]. These values are shown in Table 3. Since RGWW also contains high concentration of NaCl, its accumulation in the digester was also monitored and shown in Fig. 2. The alkalinity in the digester was recorded between 2157 and 2798 mg L^{-1} throughout this study, except slightly lower during the heavy rainfall period. The total VFA accumulation in the digester was recorded increased with time as higher percentage of RGWW was applied to the system.

Table 3: The digester performance and stability recorded in terms of COD removal efficiency, production rate of biogas and methane, total VFA, alkalinity, pH and MLVSS

		Digester performances Mean ± SD			Digester stability			
Operation Days	RGWW co-digested (%)	COD removal efficiency (%)	Biogas production rate[a] m^3 day^{-1}	Methane production rate m^3 day^{-1}	Total VFA period mg L^{-1}	Alkalinity mg L^{-1} $CaCO_3$	pH -	MLVSS g L^{-1}
1-13	1.0	94±2	859±81	505±44	374±70	2599±194	6.9±0.1	8.14±1.2
14-22	2.0	95±2	717±49	418±42	600±174	2594±289	6.9±0.1	8.83±1.7
23-33[a]	3.0	95±1	497±42	283±22	813±134	2157±119	7.0±0.1	8.25±1.0
34-60[a]	4.0	95±2	581±70	347±54	800±126	2538±328	7.1±0.1	8.58±1.6
61-77	5.0	97±1	560±57	323±30	858±109	2798±240	7.1±0.1	8.45±1.9
78-85	5.25	96±1	576±104	307±58	913±80	2400±224	7.1±0.1	8.58±1.5

[a]: Biogas refer to the mixture of CO_2 and methane gases and traces of H_2S (negligible for calculation)

Although the system was stable within optimal pH range for anaerobic treatment (6.9-7.1) and high concentration of MLVSS (8.14-8.58 g L^{-1}) in the digester, the NaCl accumulation in the digester increased with time as higher percentage of RGWW was co-digested. The sodium ion (Na^+) inhibition on methanogenesis has been widely reported in many studies[3,28]. In this study, the Na^+ inhibition is clearly seen from the increasing level of the VFA in the system and reduction of CH_4 quality and quantity produced. The fact that the COD removal efficiency was not affected by the increasing concentration of NaCl in the digester suggests the feasibility of POME treatment despite high level of NaCl recorded. This is consistent with the previous study on high removal efficiency in the high salinity environment on COD[7] and ammonia nitrogen[10]. In the case of anaerobic salt bacteria, *Halanaaerobium lacusrosei* better performance was observed[5]. Satisfactory dissolved organic carbon (DOC) removal efficiency was also observed when the cell was able to rapidly acclimatize to high salinity environment[11].

In this study, although COD removal efficiency was not severely affected by high NaCl accumulation (4000 mg L^{-1}), the biogas and CH_4 production rate reduced significantly. Consequently it also effected the CH_4 quality and yield as well. Previous studies reported that methanogenic bacteria could stand with the salinity of less than 10,000 mg L^{-1} and the higher salt concentration affected the digester performance. Sludge grown under higher salt concentration less tendency for flocculates and immediate release of cellular constituents resulted in increased soluble COD[26], high lipid and RNA content in the sludge[29] and also affects in nitrification process[30]. Low salt tolerant was observed from this system and it was believed it is heavily depend on the types of the digester used, the types of effluents applied and operational conditions for the treatment of high salt containing wastewater. It is interesting to note that some of the studies showed satisfactory biogas production despite high salinity environment[7,11]. In this study, the quality and quantity of CH_4 produced reduced as the salinity increased with time. This could be explained from the system design. In the Up-flow Anaerobic Sludge Bed (UASB)[7] and Submerge Anaerobic Membrane Reactor (SAMBR) system[11], granular sludge compacted with bacteria and methanogens rapidly developed in the reactor. In this study, conventional stirred tank reactor was utilized and microorganisms exist in flocks formation rather than granular sludge which obviously very sensitive to the nonconductive environments such as high NaCl concentration. Granular sludge formation provides better condition for methanogens[31] and in such environments the methanogens are protected in the granules so that the process could generate satisfactory methane production.

CONCLUSION

The present study demonstrated the feasibility of co-digesting POME and RGWW for the removal of COD from 1% RGWW to 5.25% RGWW. A maximum percentage of RGWW co-digested, satisfactory COD removal efficiency of above 90% was obtained even though high NaCl concentration was measured in the digester. Initially the CH_4 production rate and yield recorded was satisfactory at 505 m^3 day^{-1} and 0.15 $kgCH_4$ $kgCODremoved^{-1}$, respectively but reduced down to 307 m^3 day^{-1} and 0.09 $kgCH_4$ $kgCODremoved^{-1}$ which was due to high NaCl accumulation in the digester of nearly 4000 mg L^{-1}. In this study, the co-digestion of high NaCl RGWW and POME is satisfactory for COD removal but not for increasing the CH_4 production. In the future, this research will be focused on higher CH_4 production and yield.

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