Enhancement anaerobic digestion and methane production from kitchen waste by thermal and thermo-chemical pretreatments in batch leach bed reactor with down flow

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Abstract

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The effects of thermal (autoclave and microwave irradiation (MW)) and thermo-chemical (autoclave and microwave irradiation – assisted NaOH 5N) pretreatments on the chemical oxygen demand (COD) solubilisation, biogas and methane production of anaerobic digestion kitchen waste (KW) were investigated in this study. The modified Gompertz equation was fitted to accurately assess and compare the biogas and methane production from KW under the different pretreatment conditions and to attain representative simulations and predictions. In present study, COD solubilisation was demonstrated as an effective effect of pretreatment. Thermo-chemical pretreatments could improve biogas and methane production yields from KW. A comprehensive evaluation indicated that the thermo-chemical pretreatments (microwave irradiation and autoclave- assisted NaOH 5N, respectively) provided the best conditions to increase biogas and methane production from KW. The most effective enhancement of biogas and methane production (68.37 and 36.92 l, respectively) was observed from MW pretreated KW along with NaOH 5N, with the shortest lag phase of 1.79 day, the max. rate of 2.38 l·day^{-1} and ultimate biogas production of 69.8 l as the modified Gompertz equation predicted.

Keywords: autoclave; microwave irradiation; kitchen waste; methane; modified Gompertz equation

Food waste is the best biodegradable organic fraction from municipal solid waste that achieved from different sources including food processing plants, domestic and commercial kitchen waste, cafeterias and restaurants (Kiran et al. 2014). Considering that the kitchen wastes (KWs) are high level of moisture and easily biodegradable characteristics, anaerobic treatment is preferred over thermal or thermo-chemical conversation technologies (Ariunbaatar et al. 2015). One of the biogasification technologies is a leaching bed reactor (LBR). LBRs can use wastes without any pretreatment such as particle size reduction (Koppar, Pullammanappallil 2008) and results in low costs, low water, energy requirements and to recover biogas at high rates (Dogan et al. 2008). Researchers have stated that enhance biodegradability and methane yield can be obtained when a pretreatment is applied before anaerobic digestion in reactor. Also the KW pretreatment is possible to decrease the necessary retention time of KW for digestion and the final quantity of the KW. The thermal pretreatment (autoclave, micro-

wave irradiation (MW)) and thermo-chemical pre-treatment (autoclave and MW- assisted NaOH 5N) are relatively recent approach for thermal and thermo-chemical degradation of the KW that enhance heating methods. Compared to conventional heating, MW is an interesting method due to its environmental and energy conservation attributes, since it removes heat losses that happen in energy transmission during normal heating (Shahriari et al. 2013). Marin et al. (2010) probed effect of high temperature and pressure MW pretreatment of source-separated KW. They indicated enhancement of solubilisation of source-separated KW in terms of soluble chemical oxygen demand (SCOD), biodegradability and biogas production. Shahriari et al. (2013) studied effects of single and dual stage mesophilic anaerobic digestion of KW. They stated that digester staging without MW pretreatment resulted maximize methane production and volatile solids stabilization efficiencies at the shortest hydraulic retention times. Anaerobic digestions of autoclaved and untreated source segregated food waste were compared in semi-continuously fed mesophilic reactors and were indicated that methane yield was 5–10% higher for untreated food waste (Tampio et al. 2015). Sawayama et al. (1997) autoclaved mixed kitchen garbage (175°C, 40 bar, 1 h) increased CH_4 yield by 30%. Although in the last decade the number of papers on MW and autoclave has considerably increased in the literature (Tampio et al. 2015; Marin et al. 2010; Shahriari et al. 2013) but there are no studies reporting on the use of MW domestic and autoclave as thermal and thermo-chemical pretreatment steps on the KW in one- stage batch leach bed reactor with down flow (LBR DF) to enhance the anaerobic digestion of KW. Therefore, the main objective of this study is the evaluation of the effects of the MW and autoclave pretreatments (MWP and ATP) as thermal pretreatment and MW and autoclave-assisted NaOH 5N (MWNP and ATNP) pretreatments as thermo-chemical pretreatment on the enhancement of the mesophilic anaerobic digestion and also increasing biogas and methane production of KW using one- stage batch LBR DF.

MATERIAL AND METHODS

Sample and pretreatment conditions. A total of 10 kg of KW was collected from restaurant located on the main campus of Bu-Ali Sina University (Hamedan, Iran). the KW was contained cooked rice (25 ± 2 % w/w), cooked pasta (17 ± 2 % w/w), cooked ground beef (15 ± 2 % w/w), apple (14 ± 2 % w/w), cabbage (14 ± 2 % w/w) and lettuce (15 ± 2 % w/w). The various components of KW were manually chopped to achieve a uniform size of 1–5 mm. All pretreatment methods were conducted just before preparing the feedings for LBR DF. All pretreatments were applied to the raw KW (without dilution with sewage). In the thermal pretreatments, mixed KW was autoclaved (Model OSK, manufacture Ogawa Seiki Co., LTD. Japan) at $T = 120$, $p = 1$ bar and $t = 30$ min (based on study of Ma et al. 2011) and for MW pretreatment, mixed KW was placed in the MW oven (Model: R-677, Sharp Electronics, UK) at $T = 80°C$, $P = 630$ W and $t = 300$ s (based on study of Alizadeh et al. 2018). For the alkaline pretreatment of KW, NaOH 5N was added as an alkaline agent to adjust the pH value of the mixed KW to around 7.5 and the alkaline pretreatment was performed in batch, in two 5 l reactors, at temperature of 23 ± 2°C under anoxic condition. A mechanical stirrer was used for continuous mixing of the components. In the thermo-chemical pretreatment tests, the KW of alkaline pretreated was autoclaved ($T = 120°C$, $p = 1$ bar and $t = 30$ min) and other test, the KW of alkaline pretreated was placed in the MW oven ($P = 630$ W and $t = 300$ s). The pretreated samples were cooled in sealed plastic containers to minimize volatilization losses and all analyses were carried out on the samples at room temperature (23 ± 2°C). The KW of thermo-chemical pretreated was placed in LBR DF and during tests, the pH of the LBR DF was adjusted to around 7.5 with NaOH 5N if pH values below 7.0 were observed.

Experiment set up. The LBR DF was made by stainless steel with total and working volume of the LBR DF that was 20 and 12 l, respectively. The LBR DF has been constructed by modifying leach bed which has fixed bed reactor. Schematic modified of the LBR DF is indicated in Fig. 1. In order to initiating the digestion reactions in each test, KW pretreated (MWP, ATP, MWNP and ATNP) was diluted with anaerobic digester sludge (4.16 l) coming from a mesophilic anaerobic digester domestic wastewater treatment plant (Renoeable Energy Organization, Tehran, Iran) before feeding the reactor. The reactor operated at 37 ± 1°C and biogas production was daily measured.
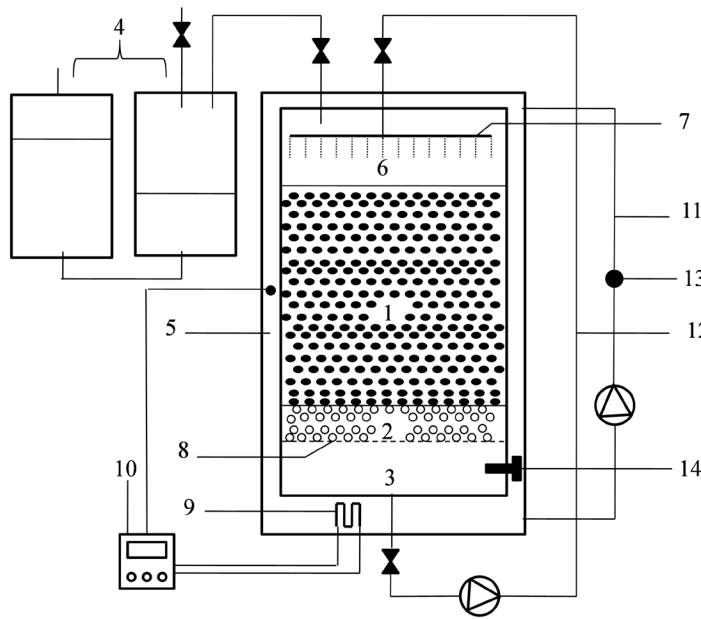
Fig 1. Schematic view of leach bed reactor with down flow (LBR DF)

1 – reactor filed with KW; 2 – gravel bed; 3 – leachate collection; 4 – system for measurement of biogas quantity; 5 – thermal isolation; 6 – head space; 7 – sprinkler; 8 – stainless steel mesh; 9 – electrical heater; 10 – thermostat; 11 – hot water circulate; 12 – leachate circulate; 13 – pH meter; 14 – leachate thermometer; ✕ – valve; ⊘ – peristalic pump

Analyses and calculations. Total solid (TS, (% w/w)) and volatile solid (VS, (% w/w)) contents (before and after all pretreatments and influent and effluent LBR DF in all tests) were calculated accoWrding to weight difference after drying of the samples for 24 h at 105°C and then for 2 h in a muffle furnace at 550°C, respectively (APHA 1995). Removal efficiencies of TS and VS were obtained according to equations (LI et al. 2015):

$$\text{TS}_{removed}\% = \frac{\text{TS}_{initial} - \text{TS}_{final}}{\text{TS}_{initial}} \times 100 \qquad (1)$$

$$\text{VS}_{removed}\% = \frac{\text{VS}_{initial} - \text{VS}_{final}}{\text{VS}_{initial}} \times 100 \qquad (2)$$

Total chemical oxygen demand (TCOD (g/l$_{sample}$)) and soluble chemical oxygen demand (SCOD (g/l$_{sample}$)) analyses (before and after all pretreatments and influent and effluent LBR DF in all tests) were determined using the closed reflux colorimetric method (APHA 1995). During all tests, the pH values were measured with a pH meter (Model pH-230SD, Lutron Electronic Enterprise Co., Taiwan). The biogas was analysed for methane in a gas chromatograph (GC-14B, Shimadzu, Kyoto, Japan). The biogas volume was measured daily using water displacement method.

Data analysis. Non-linear model (modified Gompertz equation) was used to estimate the performance parameters such as lag phase duration time and predicting the final cumulative biogas production. The correlation coefficient (R^2),

root mean squared error (RMSE) content indicate the accuracy of the model. Kinetic of biogas and methane production in batch condition were assumed that had correspondence to growth rate of methanogenic bacteria in LBR DF. The modified Gompertz equation as follows (WANG et al. 2015):

$$B = B_0 \exp\left\{ -\exp\left[\frac{R_m e}{B_0}(\lambda - t) + 1\right] \right\} \qquad (3)$$

where: B – cumulative biogas or methane production (l) at retention time t (day); B_0 – ultimate biogas or methane production (L); R_m – maximum biogas or methane production rate (l). λ value – lag phase time (day), and e is 2.7183. Kinetic constant of B_0, R_m and λ was determined using non-linear regression with help of Mathlab 8.1® software. The non-linear model was fitted base on to minimize the RMSE between the predicted and measured values. In this study, VS values were measured before and after all pretreatments and influent and effluent LBR DF in all tests

RESULTS AND DISCUSSION

Effects of thermal and thermo-chemical pretreatments on TS and VS of the KW

In present study, the KW was cut in uniform size of 1–5 mm which increased effective surface area. The moisture contents at all thermal (MWP and ATP) and thermo-chemical (MWNP and ATNP) tests were as 74.5–78.68%. These values were in the

Table 1. Kitchen waste characteristics before and after all pretreatments and influent and effluent LBR DF in all tests

Parameter	TS (%, w/w)	VS (%, w/w)	SCOD (g·l^{-1}$_{sample}$)	TCOD (g·l^{-1}$_{sample}$)	TCOD/SCOD (%)	TS$_{removed}$ (%)	VS$_{removed}$ (%)
Influent LBR DF in control	23.03	21.72	35.6	58	61.37	–	–
Before MWP	21.9	21.04	31.77	42.43	74.87	–	–
After MWP	21.72	20.76	57.74	64.72	89.21	0.82	0.47
Before ATP	25.49	23.53	31.93	43.16	73.98	–	–
After ATP	23.16	21.26	96.67	119.65	80.79	9.1	6.7
Before MWNP	23.66	22.75	28.42	37.87	75.04	–	–
After MWNP	23.18	22.12	60.92	66.72	91.03	2.02	2.76
Before ATNP	25.71	23.78	30.67	40.99	74.82	–	–
After ATNP	24.17	22.02	97.34	118.4	82.21	5.98	7.4
Effluent LBR DF in control	17.78	16.32	26.61	46.87	56.77	22.79	24.86
Effluent LBR DF in MWP	16.7	13.76	43.91	53.59	81.93	23.11	33.71
Effluent of LBR DF in ATP	17.86	15.65	82.54	96.28	85.84	22.88	26.38
Effluent LBR DF in MWNP	15.3	13.03	45.36	54.57	83.12	23.73	33.96
Effluent of LBR DF in ATNP	18.59	16.12	83.37	96.32	86.55	23.08	26.79

TS – total solid; VS – volatile solid; SCOD – soluble chemical oxygen demand; TCOD – total chemical oxygen demand; LBR DF – leach bed reactor with down flow; MWP – microwave irradiation pretreatment; ATP – autoclave pretreatment; MWNP – microwave-assisted NaOH 5N preteretment; ATNP – autoclave-assisted NaOH 5N pretreatment

range reported by Kim et al. (2008) as 70–90% for organic waste from different countries. Characteristics of the KW before and after thermal, thermochemical pretreatments and control (without pretreatment) are shown in Table 1. According to Table 1, TS and VS contents decreased after MWP, ATP, MWNP and ATNP. Dogan et al. (2008), Tampio et al. (2014) and Marin et al. (2010) stated that decreasing TS and VS contents were due to dilution by steam condensation during the MW radiation and autoclave. Also, the solid organic components are converted to soluble organic components during retention time which indicates that the hydrolysis was effective in the LBR DF and most of the solid particulates of the KW solubilized during retention time. According to Table 1, the TS$_{removed}$ and VS$_{removed}$ contents increased after all pretreatments and effluent LBR DF in all tests. These increasing are due to degradation of solid organic components by microorganisms during retention time (Dogan et al. 2008).

TCOD and SCOD variations

As shown in Table 1, values of the SCOD, TCOD and the SCOD / TCOD ratio (COD solubilization)

increased for all pretreatments compared with control test. By observing SCOD and TCOD values in the leachate samples collected from the LBR DF, the efficiency of the LBR DF (solubilization efficiency) can be determined in terms of SCOD/TCOD ratio. SCOD/TCOD ratio can effectively reflect pretreatment performance (Li et al. 2013). According to Table 1, the most extensive effects of solubilization efficiency occurred with thermo-chemical pretreatment (MWNP and ATNP respectively), followed by thermal pretreatment (MWP and ATP respectively). Increasing solubilization of organic solids made the soluble compounds more available for the anaerobic microorganisms, thus the biogas and methane productions were increased (Ariunbaatar et al. 2015). The autoclave and MW irradiation can damage the cell membranes of vegetable materials and food scraps because of the increasing temperature and internal pressure enhance solubilization (Cheng et al. 2010). Also Kuglarz et al. (2013) reports that the raise of temperature to 100°C might led to a partial evaporation of volatile organic compounds that cause decreasing soluble COD. In this study, temperature of MW pretreated of KW was less than 100 and prevented evaporation of the volatile organic compounds and caused increasing in soluble COD. As a result of decreas-
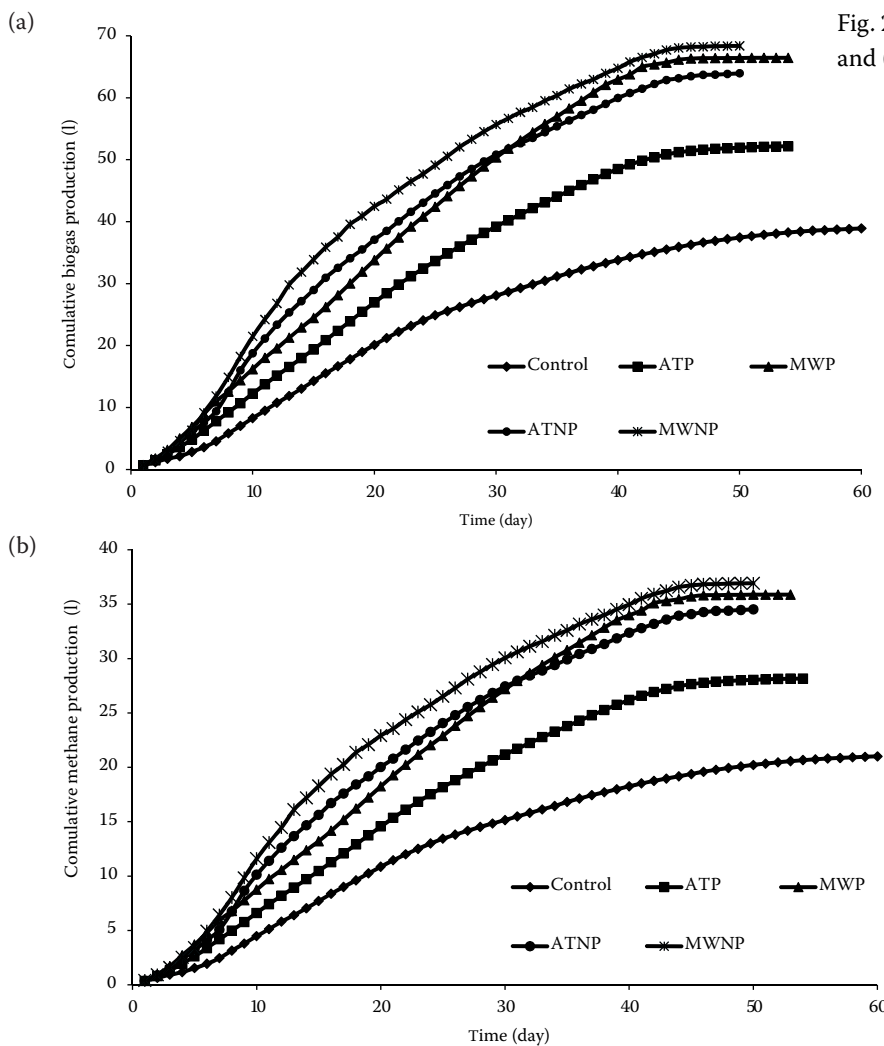
Fig. 2. (a) Comulative biogas production and (b) cumulative methane production

ing evaporation of volatile organic compounds in the MW pretreatment, the TCOD and SCOD values were more than ATP and ATNP and control test. A number of papers have reported that ATP and MWP could increase biogas production as a resulted of an increase in COD solubilization (Shahriari et al. 2013; Marin et al. 2010).

Variations of pH, biogas and methane production

pH is effective factor in anaerobic digestion stages that can be described as an indicator of the stability of a digester. In present study, during thermo-chemical pretreatment tests, the pH of the LBR DF was adjusted to around 7.5 with NaOH 5N and in thermal pretreatment tests, the pH values decreased respectively from 4.5 to 4.0 and 4.2 to 4.0 in MW and autoclave pretreatment of tests. Then

the pH values increased and at the end of tests, pH values reached to 7.1 and 7.3, respectively. In control test, the pH values decrease from 5.8 to 4.6 and then increased to 7.2. This decrease in pH can be explicated to degradation of macromolecules (oligosaccharides and amino acids) into acidic compounds that resulted in accelerating acidification (Papadimitriou 2010; Shahriari et al. 2013). Also, high temporary pressure of CO_2 may decrease pH values (Papadimitriou 2010). In addition, low pH values of the leachate might be due to composition of the KW which mainly includes acidic fruit waste. The increasing in the pH value of the leachate could be due to proteins desorption or acidic compounds volatilization (Bougrier et al. 2007). Fig. 2 shows cumulative biogas and methane production in control, ATP, MWP, ATNP and MWNP. According to the experimental results shown in Fig. 2a,b, the highest cumulative biogas and methane production were respectively obtained as 68.37 and

Table 2. The biogas and methane yields in present study

Condition experiment	Retention time (day)	Biogas production (l)	Methane production (l)	Increase of biogas compared to control (%)	Increase of methane compared to control (%)
Control	60	38.9	21.01	–	–
ATP	55	52.16	28.15	25.42	25.36
MWP	55	66.44	35.87	41.45	41.42
ATNP	50	63.93	34.52	39.15	39.13
MWNP	50	68.37	36.92	43.1	44.12

for abbreviations see Table 1

as 36.92 L in MWNP test. In this study, the ATNP and MWNP reduced retention time to 50 days. In present study, all tests produced low values of biogas and methane at the beginning of test, which progressively increased during retention time. The MWP, ATP, MWNP and ATNP accelerated the hydrolysis of the complex organic components and cause destruction of cells membranes, release of enzymes and organic compounds presented in protoplasm. Kuglarz et al. (2013) stated that the released organic compounds into the liquid phase usually cause increase SCOD/TCOD ratio and biogas production. According to Fig. 2 and Table 2, the highest cumulative biogas and methane production were obtained in the highest effective pretreatment in term of SCOD/TCOD in the MWNP test and less cumulative biogas production was obtained in the less effective pretreatment in term of the SCOD/TCOD in control test. Increasing values of soluble compounds obtained from the pretreated of the KW are caused increasing of cumulative biogas and methane production. Also, increasing of cumulative biogas and methane production related with the greater biological accessibility in the anaerobic digestion process (Papadimitriou 2010). The cumulative biogas and methane production in control test is less than other tests. The reasons decreasing biogas and methane production could be the mechanisms used in each pretreatment method and undesirable refractory compounds such as carboxylic acids, furans and phenolic compounds (Tahrzadeh, Karimi 2008). A number of papers have reported that ATP and MWP could increase biogas and methane production. Tampio et al. (2014) autoclaved (160°C, 6.2 bar) FW and reported that methane yields were 5–10% higher for untreated FW than for autoclaved FW but Menardo et al. (2011) autoclaved swine slurry at 120°C and showed an increase in CH_4 yield of showed an in-

crease in CH_4 yield 115% and also Sawayama et al. (1997) autoclaved mixed kitchen garbage (175°C, 40 bar, 1 h) increased CH_4 yield by 30%. Marin et al. (2010) indicated that MW irradiation at ramp 7.8°C·min⁻¹ increased biogas production as 16% (about 1,700 ml). Shahriari et al. (2012) demonstrated that MW irradiation at 145°C with a ramp of 2.7°C·min⁻¹ increased biogas production (about 650 ml) compared to control. Rafique et al. (2010) showed that thermo-chemical pretreatment has high effect on biogas and methane potential at 70°C with increase of 78% biogas and 60% methane production. While thermal pretreatment also showed enhancement at 100°C having 28% biogas and 25% methane increase. This enhancement of the anaerobic digestion process is due to the reduction of the hemicellulosic fraction (Ariunbaatar et al. 2014). In present study, the values of biogas and methane productions are more than other studies. The discrepancy between the biogas produced in this study with other studies can be attributed by KW characteristics, pretreatment conditions and LBR DF performance.

Modeling and influential parameters on biogas and methane production

In order to further evaluate the effectiveness of the all pretreatments, empirical non-linear regression models (Eq. 3) were fitted to the data. The non-linear regression results are shown in Table 3. According to the non-linear regression results obtained using ATP, MWP, ATNP and MWNP, they can be seen from Table 3 that the lag phases were decreased in KW digestion with the addition of thermo-chemical pretreatments. The thermo-chemically pretreated KW had least lag phases and in biogas and methane production, the exponential

Table 3. Parameters estimated of biogas and methane from non-linear regression for all tests

Pretreatment	Estimated B_0 (l)	Experimental B_0 (l)	R_m (l·day^{-1})	λ(day)	R^2	RMSE*
Control	39.73 (21.45)	38.90 (21.01)	1.15 (0.62)	3.11 (3.11)	0.9979 (0.9979)	0.014 (0.014)
ATP	56.10 (30.29)	52.16 (28.15)	1.59 (0.85)	2.99 (2.99)	0.9986 (0.9986)	0.012 (0.012)
MWP	72.80 (39.31)	66.44 (35.87)	2.01 (1.08)	2.87 (2.87)	0.9975 (0.9975)	0.017 (0.011)
ATNP	66.29 (35.8)	63.93 (34.52)	2.04 (1.13)	2.10 (2.04)	0.9951 (0.9949)	0.022 (0.012)
MWNP	69.80 (37.69)	68.37 (36.92)	2.38 (1.28)	1.79 (1.79)	0.9934 (0.9934)	0.026 (0.013)

*values in parentheses are related to methane gas; for abbreviations see Table 1

phases were respectively started approximately at 1.7 and 3.11 day from the beginning of the digestion period. This was attributed to the acetic acids concentration and SCOD/TCOD %, which were increased after thermo-chemical pretreatment compared to without thermo-chemical pretreatment, which would have increased methanogensis and methane production potential (Li et al. 2013). This would indicate that MWP and MWNP could effectively accelerate the KW digestion and increase the biogas and methane production. ATP, MW, ATNP and MWNP increased SCOD/TCOD (%). The highest increase in KW digestion was observed with the MWNP that obtained higher biogas and methane production. Compared to the KW digestion with ATP and MWP, ATNP and MWNP yielded higher biogas and methane production which is indicated thermo-chemical pretreatment (MWNP) has a better effect on methane production and SCOD/TCOD (%) than thermal pretreatment alone (ATP and MWP) (Li et al. 2013; Rafique et al. 2010). According to this present study results and discussion (Table 1), the SCOD/TCOD % would be one of the most important factors to be applied in the evaluation of the biodegradation, biogas and methane enhancement of a pretreatment strategy.

CONCLUSION

Generally, thermo-chemical (ATNP and MWNP) and thermal (ATP and MWP) pretreatments improved the anaerobic digestion of the KW by disrupt the recalcitrant structures of wastes that caused increase biogas production. Comparing the increases in biogas and methane production, non-linearly estimated lag phase periods and estimated maximum methane production rates of all pretreatment conditions, MWNP and MWP would be indicated respectively as a more effective pretreatment approach than other pretreatment conditions to enhance the biogas and methane production from KW. The most effective enhancement of biogas and methane production was observed respectively 68.37 and 37.6 l for MWNP test and 63.93 and 35.8 l for MWP test.

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