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Effect of Hydrothermal Pretreatment for Enhanced Biogas Production Using Micro-algal Biomass

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Abstract

The filamentous algal biomass, Hydrodictyon reticulatum (H. reticuatum) was used to evaluate the biogas production after hydrothermal pretreatment in batch anaerobic digestion. The effect of hydrothermal pretreatment at range of 50 ~ 280 ^{o}C was tested with filamentous algal biomass. The solubilization by pretreatment showed a higher degree of disintegrated filamentous algal biomass and the soluble COD was increased 12.8 fold than untreated sample at 280 ^{o}C. Solubilization due to hydrothermal pretreatment increased the specific biogas production and volatile solids reduction rates. Using the hydrothermal treatment approach, the specific methane production were 161.8, 169.1, 178.4, 191.7, 274.9, 285.2 and 364.4 mL/g-VS of the pretreated at raw, 50, 100, 120, 180, 230, and 280 ^{o}C, respectively. The volatile solids reduction was greater with increased temperature applied, and the degradation increased 69.7% at 280 ^{o}C from 45.8 % VS reduction of raw filamentous algal biomass.

Keywords*: We would like to encourage you to list your keywords in this section*

1. Introduction

Recently, there has been increasing interest in assessing the energy potential of biofuels obtained from micro-algae (González-Fernández *et al*., 2011). Algae biofuels are an attractive option (Sigh *et al*., 2011) due to its rapid growth rate comparatively, low land usage and high carbon dioxide (Jorquera *et al*., 2010). Because of the other resource of biomass energy resources such as derived from terrestrial crops (first generation) and derived from lignocellulosic crops (second generation) were included the needs of arable land, large amount of water and low biomass productivities (González-Fernández *et al*., 2012). However, microalgae as a third generation of biomass resource based of biofuels are good materials to overcome the mentioned problems. Up to now, numbers of studies have been published to investigate the utilization of microalgae to produce of biodiesel, bioethanol and biohyderogen as a feedstock (Guldhe et al., 2014; Ho *et al*., 2013; Zhu *et al*., 2014). However, biofuel production from microalgae feedstock has several problems to be

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resolved before it can become an economical and environmental ways of alternative energy (Ward *et al*., 2014). The problems face high energy required for conversion process and the process of disposing the residual biomass is difficult even after beneficial lipid extraction since the defatted biomass still contains higher levels of organic matter, such as carbohydrate and protein (Park *et al*., 2013). Anaerobic digestion can be a solution to these problems because this technical process can mineralize algal biomass (Sialve *et al*., 2009). Biogas production from algae by anaerobic digestion is one of the most environmental and beneficial technology (Lee *et al*., 2014). However, anaerobic digestion of microalgae is limited by lard cell wall (Chen *et al*., 1998). According to the report of Golueke *et al*. (1957) digested sludge provided a noticeable green color during anaerobic digestion because of the persistence of chlorophyll, which in an intracellular material. This result showed that cellular lysis was not completed during digestion. Therefore, pretreatment of the algal biomass is needed because the complex cell wall structure of microalgae, composed by cellulose, hemicellulose and pectin, makes bacteria to attack difficult (Passos *et al*., 2013). González-Fernández *et al*, (2012) studied that comparison of ultrasound and thermal pretreatment of micro algal biomass to enhance the biogas production. Passos et al., (2013) employed microwave and thermal treatment to improve the disintegration and digestibility of microalgae. Hydrothermal pretreatment of lignocellulosic biomass was usually evaluated for enhanced bioethanol production (Kumar *et al*., 2009; Mood *et al*., 2013). Because the advantages of hydrothermal pretreatment for lignocellulosic biomass are no addition, hydrate cellulose, and remove hemicellulose and part of lignin (Chandra *et al*., 2012). However, little information is available regarding filamentous algal biomass of hydrothermal pretreatment for anaerobic digestion.

The objective of this study was to investigate the solubilization of filamentous alga, *Hydrodictyon reticulatum (H. reticulatum)* using hydrothermal pretreatment. An additional aim of this work was to study the effects of different temperature for pretreatment to algal feed on anaerobic digestion

2. Materials and Methods

2.1. Hydrothermal Pretreatment of Filamentous Algal Biomass

The filamentous alga used in this study, H. reticulatum, was supplied by the Korean Research Institute of Chemical Technology in Daejeon, Korea, and the characteristics of filamentous algal biomass presented in Table 1. The hydrothermal reactor was used to investigate the effect of hydrothermal pretreatment on the biogas improvement property and dewatering performance of the filamentous algal biomass, *H. reticulatum*. Experiments were performed using a 1000 mL autoclave reactor consisting of a reactor body, a heater, and a steam condenser which was operated under N_2 gas. A 100 mL of *H. reticulatum* feed stock was mixed with an equal amount of water and leaded into the reactor. The operating temperatures and pressures ranged from 50 to 280 and the reaction time was 30 min. Typically the temperature was from 180 to 320 that present subcritical water. The components within the reactor were vigorously mixed using an agitator rotating at 200 rpm.

Soluble COD (SCOD) release was used as a direct measurement of filamentous algal biomass cell disintegration. When the filamentous algal biomass was pretreated by hydrothermal equipment, the intracellular contents were released in the aqueous phase. Increased SCOD after pretreatment was an indicator of the solubilization efficiency. All samples for SCOD were filtered through a 0.45 μm membrane and then used for measurement.

Table 1. Characteristics of Filamentous Algal Biomass

	SCOD	NH_3-N	T-N	TS	VS	VS/TS
Concentration (mg/L)	44.45	2.70	140.00	5,860	5,330	0.91

2.2. Biochemical Methane Potential (BMP) Test

Batch digestion was performed in a series of BMP assays by incubating algal biomass inoculated with anaerobic bacteria for a period about 35 days based on Angelidaki *et al.* (2009). The active inoculum operating at methophilic conditions was obtained from the anaerobic digester at the Jungrang municipal wastewater treatment plant in Seoul, Korea.

Two rounds of batch experiments were conducted under mesophilic conditions. The first round focused on the effect of Substrate to inoculums ratio (S/I ratio). The second round compared the biogas productivity and VSR depending on the different temperature of pretreatment. Batch anaerobic digestion was performed in 160 ml serum bottles with a working volume of 100 ml. The data for both cumulative gas production and volatile solids reduction (VS reduction) were obtained during the digestion.

A nutrient/mineral/buffer (NMB) medium prepared according to Young and Tabak (1993). Duplicate units of the digestion setup were used for all pretreatment schemes in this research. Biogas production from inoculums and medium was recorded and used as the control. Substrate and inoculum were used at a ratio of 0.5:1 using the VS mass except the S/I ratio experiment.

2.3. Analytical Methods

The concentration of Total Solid (TS), Volatile Solid (VS), Total Nitrogen (TN), Total Phosphate (TP), ammonia nitrogen, and COD were analyzed according to the standard methods (APHA, 2005). Total COD (TCOD) and Soluble COD (SCOD) were determined for solubility of filamentous micro algal biomass. The samples were centrifuged at 4000 rpm for 5 min and the supernatant was filtered through a 4.5 μm pore size membrane filter and then used for the analysis of SCOD. Biogas composition was determined by gas chromatography (Young Lin instrument 6800 series, Korea) using a thermal conductivity detector, by injecting gas samples into a packed column (hayeSep 3 m 1/8 in. 100/120). The carrier gas was Helium in split less mode (column flow: 19 mL/min). The oven temperature was 35 ^{o}C with a retention time of 1.5 min. Injector and detector temperatures were 150 and 180 ^{o}C, respectively. The gas composition was measured toward the end of the experiment.

3. Results and Discussion

3.1. Substrate to Inoculum Ratio Test

The maximum methane productivity and the higher rate of methane productivity were reached at a S/I ratio of 0.5, regardless of the filamentous algal biomass tested (Figure 1). At the different S/I ratios, the final methane productivity were 163.6, 137.9, 118.5 and 105.8 mL / g-VS for S/I ratios of 0.5, 1.0, 3.0 and 5.0, respectively. Eskicioglu and Ghorbani (2011) published that the specific biogas production rate in BMP tests significantly decreased with decreasing inoculums to substrate ratio (ISR). They observed that all BMP tests had similar specific methane yields at the end of experiment. In other words, ISR did not affect the ultimate biodegradability of corn whole stillage. However, in case of the report by Zheng *et*

al., (2010), the cumulative biogas production at the end of digestion for the ISR value of 2.0, 1.0 and 0.5 were 70.38, 126.57 and 153.51 mL, respectively. Thus, the biogas production was depended on the substrate concentration or ISR. Because of the low ISR (0.46, w/w) accumulated high concentration of total volatile fatty acids and pH decreased during the batch anaerobic digestion reaction (Eskicioglu *et al.*, 2011). In this study, we adopted the substrate to inoculums ratio of 0.5 (w/w) for the filamentous algal biomass methane production experiment belong to different temperature of pretreatment.

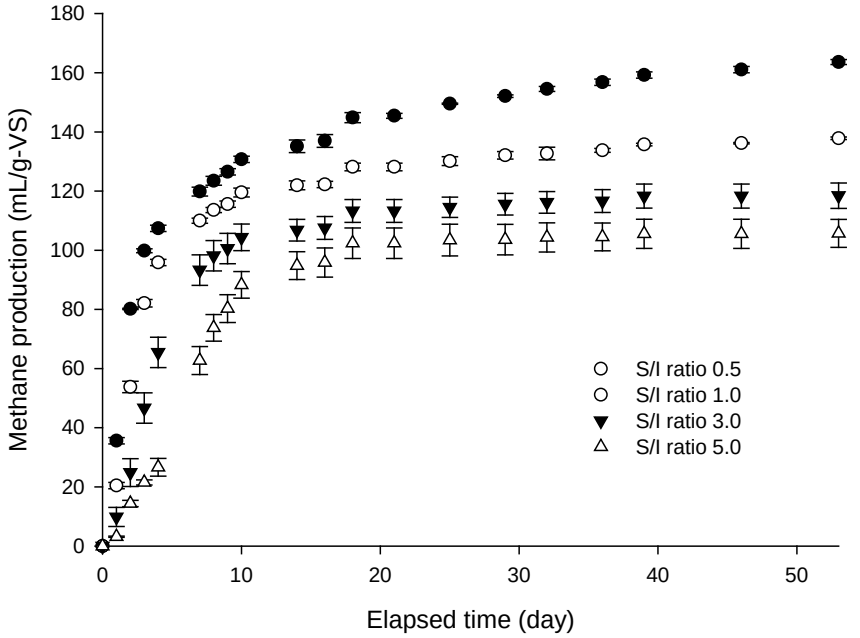

Figure 1. Methane Production of Depending on the Substrate to Inoculum Ratio in Batch Anaerobic Digestion

3.2. Solubilization of Filamentous Algal Biomass

Hydrothermal pretreatment focused on filamentous algal biomass to improve anaerobic digestion in this study. The biomass was subjected to hydrothermal pretreatment with the six different pretreatment temperatures. Two ranges of thermal pretreatment for anaerobic digestion batch experiments were conducted under mesophilic conditions. The first range was typical thermal pretreatment as 50, 100 and 120 ^{o}C, and the second was hydrothermal pretreatment range as 180, 230 and 280 ^{o}C. Typically the temperature was from 180 to 230 ^{o}C that present subcritical waster. SCOD increased along pretreatment temperatures. González-Fernández *et al* (2012) assumed that thermal decomposition of organic material happened in several stages and the solubilization of organic matter belong to the pretreatment time at mild pretreatment temperatures such as 70 and 90 ^{o}C. However, pretreatment temperature is more important than reaction time of solubilization for the organic sludge at the high temperature of pretreatment such as 175 to 200 ^{o}C (Haug *et al.*, 1978). As it can be seen in Figure 2, TCOD was as decreased as SCOD increased. At the end of pretreatment time, 30 min, SCOD increased by 5.3, 5.1, 5.3, 7.3, 11.8 and 12.8 fold for 50, 100, 120, 180, 230 and 280 ^{o}C pretreated temperature, respectively. The results of solubilization in this study obtained similar with Keymer *et al.* (2013) report. They used high temperature and pressure to improve bio gas production of microalgae in anaerobic digestion. The algae biomass and lipid extracted algae fed in high temperature pretreatment equipment during

about 30 min for solubilization of algae biomass and lipid extracted algae. The reaction temperature was 170 °C. After pretreatment, high pressure thermal hydrolysis after lipid extracted of algae sample increased 10 fold of SCOD. Thus the hydrothermal pretreatment is expected particularly effective method at solubilization of organic materials (Wilson and Novak, 2009).

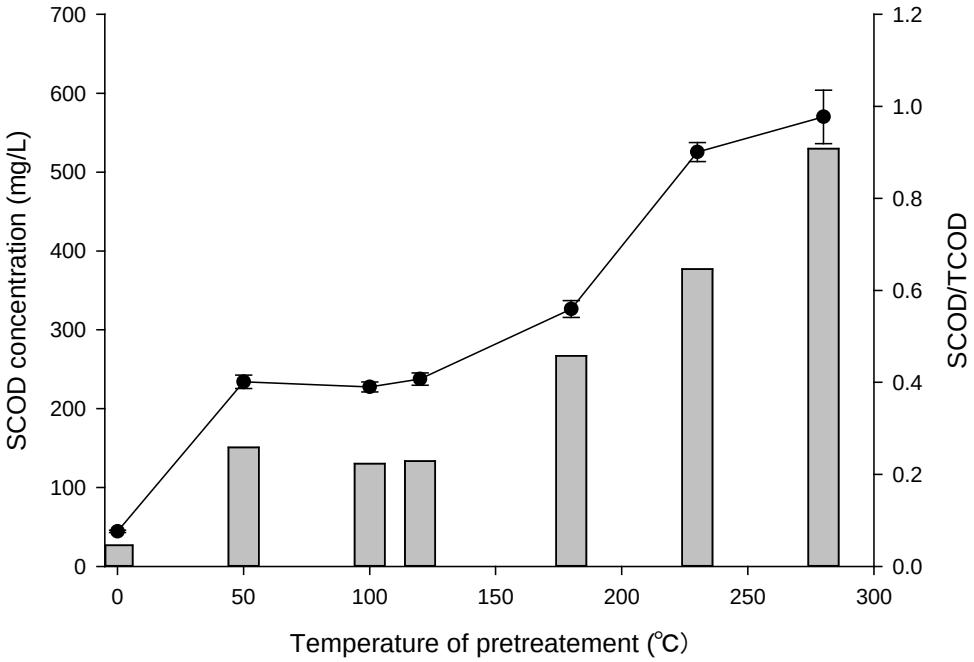

Figure 2. Solubilization of Algal Biomass along Different Pretreatment Temperature

3.3. Effect of Pretreatment for Biogas Production

The assessment of raw and pretreated algal biomass as substrate for the anaerobic digestion was evaluated by the BMP test over a period of 35 days (Figure 3). BMP assay were conducted to determine the specific conditions required for optimal methane production of algal biomass using different pretreatment temperature from 50 to 280 °C. The filamentous algal biomass, *H. reticulatum*, was subjected to thermal pretreatment of different temperatures before the BMP assays. Methane production rate of all kinds of substrates was maximal during the first 8 days and decreased after that. Raw *H. reticulatum* substrate produced methane yield on VS basis of 161.8 mL/g-VS whereas pretreated samples at 50, 100, 120, 180, 230, and 280 °C resulted in 169.1, 178.4, 191.7, 274.9, 285.2, and 364.4 mL/g-VS, respectively. According to the previous study of Chen and Oswald (1998), methane production of algal was increased up to 33% at applied 110 °C pretreatment temperature for 8h. However, another researcher, De Schamphelaire and Verstraete (2009) reported that pretreatment of mixing microalgae was not effective to increase biogas at 80 °C for 2.5h. González-Fernández et al. (2012) reported that the thermally treated microalgae biomass showed a different behavior depending on temperature applied.

To compare the methane production of digestion of different pretreatment temperatures, according Fig. 3, there are two kinds of patterns depending on the temperatures of pretreatment. The first pattern of low temperature range (50 ~ 100 °C) was appeared low methane production increasing rate (10.27 % at 100 °C). The second pattern of high temperature range (120 ~ 280 °C) was reached about 125% at 280 °C of raw *H. reticualtum* biogas production. Thus, low temperature (50 ~ 120

°C) pretreatment applying to enhance biogas production was not effective. However, when was applied high temperature (180 ~ 280 °C) to increase methane production, it increased depending on the temperature rising (Figure 4). In other words, hydrothermal pretreatment was effective to improve methane production in anaerobic digestion condition. The results of methane production depending on different temperatures were similar with solubilization of filamentous algal biomass (Figure 3).

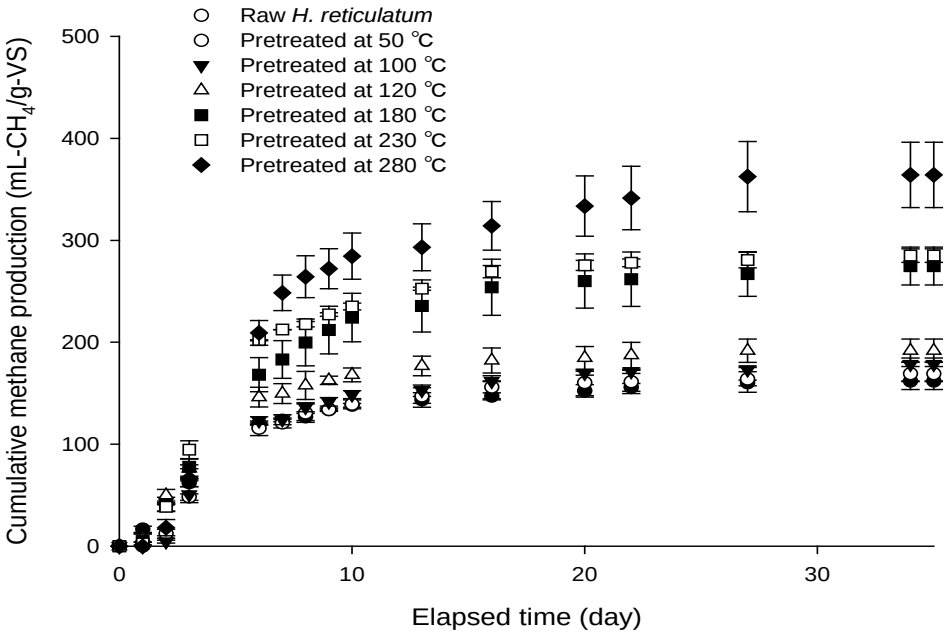

Figure 3. Methane Production of Algal Biomass depending on Pretreatment Temperature

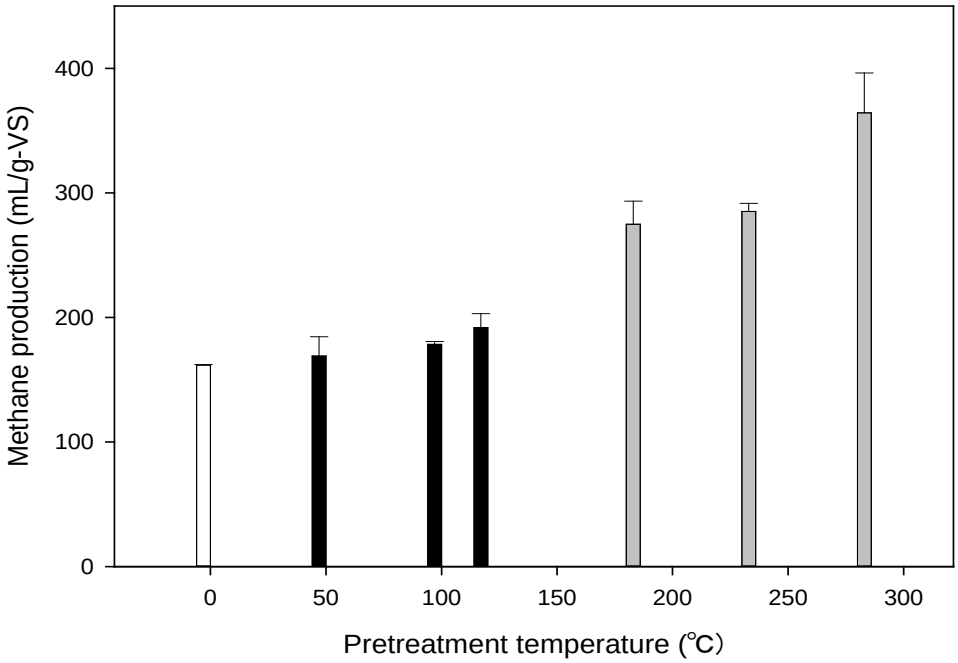

Figure 4. Methane Production Depending on Pretreatment Temperatures at the End of Experiment

3.4. Volatile Solids Reduction (VSR)

Figure 5 shows the VS reduction versus the thermal pretreatment temperatures. VS reduction is an important parameter for assessment of biodegradation of organic matters. The results of the VS reduction after anaerobic digestion batch test were achieved 45.8, 54.4, 57.5, 61.7, 66.6, 68.0 and 69.7% with the applying pretreatment temperatures at raw, 50, 100, 120, 180, 230 and 280 ^{o}C, respectively. The VS reduction was increased with depending on the temperatures of pretreatment increasing only except at 280 ^{o}C. Elango *et al*., (2007) studied VS reduction using municipal solids waste (MSW) and domestic sewage of high strength effluent in the continuous anaerobic digestion. They obtained the results of VS reduction were 73 and 87% with only initial range and after the continuous feeding of substrate, respectively. The activities of anaerobic microorganisms increase with the increase of temperature, reflecting stable degradation of substrate (Elango *et al*., 2007). Ratanatamskul *et al*. (2014) reported VS reduction of food waste and sewage sludge. VS reduction of 68.9, 53.8 and 44.2% were achieved with the applying hydraulic retention time at 27, 22 and 19 days, respectively.

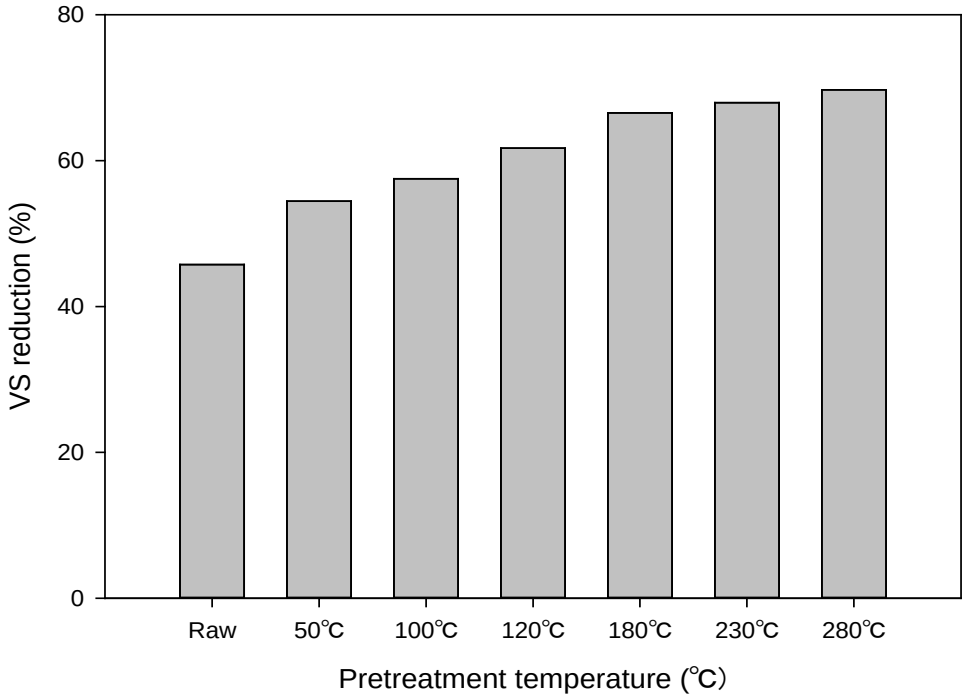

Figure 5. VS Reduction depend on the Pretreatment Temperatures at the End of Experiment

4. Conclusion

This study demonstrated that filamentous algal biomass, *H. reticulatum*, is a potential substrate at different S/I ratio and thermal pretreatment for anaerobic digestion. In batch anaerobic digestion carried out at different S/I ratio, the ultimate methane production decreased from 163.6 to 105.8 mL/g-VS when the S/I ratio increased from 0.5 to 5.0. These results showed evident influence on methane production. The influence of thermal pretreatment depending on different temperatures were conducted 161.8, 169.1, 178.4, 191.7, 274.9, 285.2, and 364.4 mL/g-VS at raw, 50, 100, 120, 180, 230, and 280 ^{o}C, respectively. The hydrothermal pretreatment was effective to improve methane production. The results of the VS reduction after anaerobic digestion batch test were achieved 45.8, 54.4, 57.5, 61.7,

66.6, 68.0 and 69.7 % with the applyed pretreatment temperatures at raw, 50, 100, 120, 180, 230 and 280 ^{o}C, respectively.

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