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RESEARCH NOTE

Microwave Pretreatment of Fresh Water Hyacinth (*Eichhornia crassipes*) in Batch Anaerobic Digestion Tank

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ABSTRACT

The purpose of this research was to study the effect of microwave power in pretreatment of fresh water hyacinth on biogas production. The variations of microwave power levels were 240; 400; 560 and 800 Watt (W). The variations of microwave heating time were 5; 7; and 9 min. The unpretreated fresh water hyacinth was used as control. The research results showed that the microwave pretreatment of fresh water hyacinth improved biogas production. Microwave pretreatment had a positive impact on anaerobic biodegradability of fresh water hyacinth. Almost all pretreated fresh water hyacinth produced biogas more than unpretreated fresh water hyacinth. The maximum biogas production from fresh water hyacinth was obtained at 560 W for 7 min of microwave pretreatment. At this condition, pretreated fresh water hyacinth produced 75.12 mililiter biogas per gram of Total Solid (mL/g-TS). The unpretreated fresh water hyacinth produced 37.56 mL biogas /(g-TS). The highest value of biogas production kinetic constants including biogas yield potential (A), the maximum biogas production rate (U) and the duration of lag phase (λ) were 78.23 mL/(g-TS); 2.26 mL/(g-TS.day); 4.60 days, recpectively.

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1. INTRODUCTION

It is well known that use of fossil fuels has caused global warming. Therefore, renewable clean energy is required for replacing fossil fuel with renewable energy sources to reduce the CO_2 emission [1] . Another issue is the energy crisis. Since the last few decades, fossil fuels are one of the most important parts of human life. Continuous use of fossil fuels and making them as the primary energy cause dwindling, because fossil fuels are not renewable. Energy crisis originates from this. Various biomasses have been identified as alternate source of energy fuels [2]. These biomasses range from various kinds of bio-wastes, e.g. food wastes, municipal wastes, agricultural wastes etc, and energy crops, e.g. edible as well as non-edible oilseeds to various aquatic plants. Biogas is an alternative source of energy that can be developed with the appropriate and relatively simple technology. Biogas is a gas mixture resulting from the activity of methanogenic bacteria in anaerobic conditions or fermentation of organic materials [3]. Biogas consists of a mixture of CH_4 (55-70 %), CO_2 (25-50 %) , H_2O (1-5 %), H_2S (0-0.5 %), N_2 (0-5 %) and NH_3 (0-0.05 %) [4].

One of the potential biomass materials that can be used as raw material for biogas production is water hyacinth. Water hyacinth (*Eichornia crassipes*) is a free floating aquatic weed that lives in waters that have a stagnant stream. Water hyacinth is often found in rivers, lakes and storages. These plants multiply rapidly both generative and vegetative. By means of vegetative propagation, they can be double folded within 7-10 days. In addition, water hyacinth is considered as a lignocellulosic biomass. Lignocellulose material is composed of cellulose, hemicellulose and lignin [5]. Water hyacinth has high cellulose content that makes it a potential source for biogas production. Several studies have been conducted to assess the potential of water hyacinth as raw material for biogas production [6-14].

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However, water hyacinth is difficult to be biodegraded, because hydrolysis of cellulosic material is a slow process and can be a major rate determining step in anaerobic digestion process [11, 15]. Numerous studies have been conducted by researchers to improve biomass conversion efficiency and biogas yield [10]. This study includes a variety of pretreatment methods on lignocellulosic materials. Several studies have been published that use physical methods (mechanical and thermal), chemical (ozonolisis, acid hydrolysis and alkaline hydrolysis) and biological (fungi and enzymatic hydrolysis), or a combination of all these three methods [16-21]. Bruni et al. [22] studied the use of mechanical treatment, hydrothermal, chemical and enzymatic process to improve biogas production. Hendriks and Zeeman [5] stated that the heat effect on lignocellulosic biomass occurs at a temperature of 150 - 180^{o}C where hemicellulose and lignin first began to dissolve. The efficiency of pretreatment can be evaluated through the material dissolved, increase of anaerobic biodegradation and operational costs.

In conventional thermal processing, heat is transferred to the material through convection, conduction or radiation of heat from the surfaces of the material. In microwave, energy is delivered directly to the maerial through molecular interaction within the electromagnetic field. Microwave radiation of electromagnetic energy is converted into heat energy. Microwave technique has many advantages, as it penetrates into the material, deposits energy and can be generated throughout the volume of the material.Transfer of heat are rapid and uniform in heated material. The use of microwave energy reduces the heating period of the materials [23]. In recent years, a number of researchers have concentrated on microwave technique, because it can reach the desired temperature quicker than conventional heating and less energy is consumed during the process [24, 25]. Microwave irradiation can alter the structure of cellulosic biomass, including increasing the specific surface area, decreasing the polymerization and crystalline cellulose, the hydrolysis of hemicellulose and lignin depolymerization [26]. Thermal pretreatment with microwaves can destroy complex structure of lignocellulosic material. Eskicioglu et al. [27] compared the pretreatment using microwave and conventional heating on waste activated sludge; both pretreatment methods equally provided an easy route to dissolve, but with microwave heating, the production of methane increased (± 16 % after 15 days at mesophilic digestion conditions and temperature 96^{o}C). Therefore, microwave pretreatment can be used as an approach over conventional heating for pretreatment of biomass in biogas production .

Zhu et al. [28] reported that microwave pretreatment on rice straw was an effective pretreatment method for increasing the rate of hydrolysis. Jackowiak et al. [29] used microwave pretreatment of wheat straw for biogas production. Research results showed that the maximum methane yield was achieved at a temperature of 150^{o}C with an increase of 28% compared without conventional pretreatment. Sapci et al. [30] conducted a study on biogas production using two methods of pretreatment on wheat straw including microwave irradiation and steam explosion. Biogas yield from wheat straw undergoing explosion of steam–microwave pretreatment increased 20% .There are many researches in biological processes showing that microwave pretreatment changes the nature of the bacteria. *Methanosarcina barkeri* DSM – 804, when exposed to microwave radiation of frequencies ranging from 13.5 to 36.5 GHz showed higher concentration of methane in the biogas, which was 76.3% on the 15th day of incubation in a microwave at the radiation frequency of 31.5 GHz [31]. The effect of microwave pretreatment on biogas production from waste activated sludge [32-35], dairy manure [36]; kitchen waste [37]; food residues [38]; municipal solid waste [39, 40] and comparison of the use of microwave and ultrasonic in palm oil mill effluent [41] have been reported.

Previous researchers have studied the effect of microwave pretreatment on lignocellulosic biomass and sludge. However, there is no researcher investigating the influence of the microwave preatreatment on water hyacinth. The objective of this research was to study the effect of microwave pretreatment at various microwave levels and times on improving the digestion of water hyacinth.

2. MATERIALS AND METHODS

The fresh water hyacinth used for this study was obtained from Citarum river in Bandung (Indonesia). Cattle's rumen was collected from slaughterhouse in Bogor (Indonesia). The characteristics of the fresh water hyacinth were as follows: pH was 6.65, total solid (TS) was 8%, and C/N was 25.93. Sample of fresh water hyacinth (leaves, stem and root) was washed, chopped to small sizes about 2 cm and ground to fine slurry using Cosmos mill type CB 289 G. A commercial domestic microwave oven 2450 MHz frequency, Sharp type 268R (800 W) was used in this study. A series of experiment were performed at different power levels of 240, 400, 560 and 800W. The microwave heating holding times were 5, 7 and 9 min. Laboratory experiments using 600 mL digester (PET bottle) were performed in batch operation mode. Each digester (PET bottle) was fed with 16 g TS of fresh water hyacinth in 200 g sample (pretreated fresh water hyacinth+water). The unpretreated fresh water hyacinth was used as control. The pH condition was 7.00. This was mixed with 16 g cattle's rumen as inoculum. After adding the substrates and inoculum, the biodigester was closed with a rubber stopper and sealed with rubber to create anaerobic conditions in the bottle. In the middle of the rubber stopper a hole was made to insert the hose; it was equipped with valve for biogas measurement.This hose served as a channel for flow of produced biogas, then it was analyzed quantitatively. The produced biogas was flowed through a hose connected to a series of devices that contained water; " water displacement method" was used to determine the volume of biogas [42-46]. Anaerobic digestion was carried out with a retention period of 60 days at room temperature. Cumulative biogas production was monitored throughout the period of study. The schematic diagram of experimental laboratory set up is shown in Figure 1.

2. 1. Modified Gompertz Equation The kinetic data obtained from all digesters were checked for the fitness of modified Gompertz equation [47]. The modified Gompertz equation, assuming cumulative biogas production in batch digester as a function of the growth rate of bacteria was used. The modified Gompertz equation is given by the equation as follows [7, 10, 42-46, 48, 49]:

$$P = A\ x\ exp\left\{-exp\left[\frac{U\ x\ e}{A}(\lambda - t) + 1\right]\right\} \qquad (1)$$

where

P Cumulative biogas production (mL/g-TS) at any time, t

A Biogas yield potential (mL/g-TS)

U Maximum biogas production rate (mL/g-TS.day)

e Mathematic constant (2.718282)

λ Duration of lag phase (days)

t Time at which cumulative biogas production P is measured (day)

The value of A, U and λ was calculated for each digester using POLYMATH software [10, 42, 44, 49].

3. RESULTS AND DISCUSSIONS

3. 1. The Effect of Microwave Power in the Pretreatment of Water Hyacinth on Biogas Production In this study, microwave pretreatment of water hyacinth was performed to improve the production of biogas. The effect of microwave pretreatment on biogas production was studied by varying microwave power levels and heating time. The control was performed using unpretreated water hyacinth. The volume of biogas production from water hyacinth at various microwave power levels and times for all digester is shown in Figure 2. From Figure 2, it can be seen that almost all pretreated samples produced biogas higher than unpretreated sample (control), except the pretreated water hyacinth at 800 W/9 min. The highest biogas production was obtained from pretreated water hyacinth at 560 W/7 min.

The use of microwave power variations in the pretreament of fresh water hyacinth resulted in different biogas production for each sample. The effect of microwave power in the pretreatment of fresh water hyacinth on biogas production is shown in Figure 2.

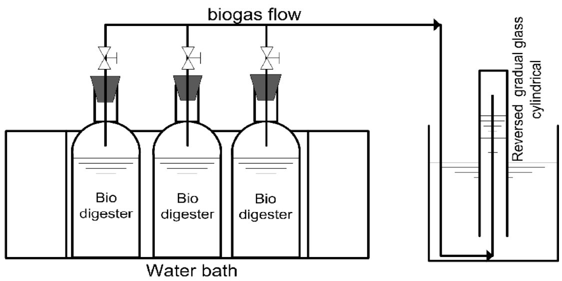


Figure 1. The shematic representation of laboratory scale biodigester [42]

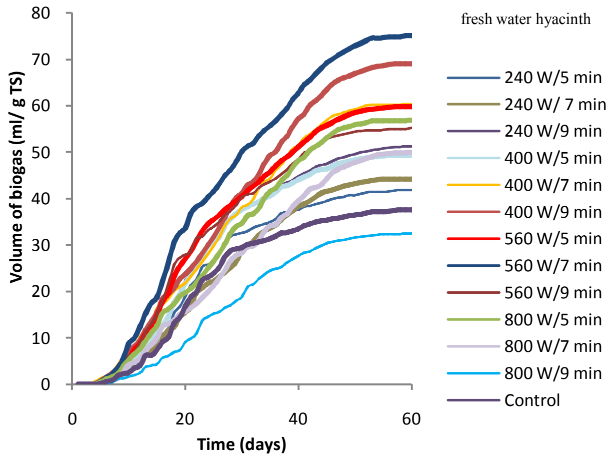
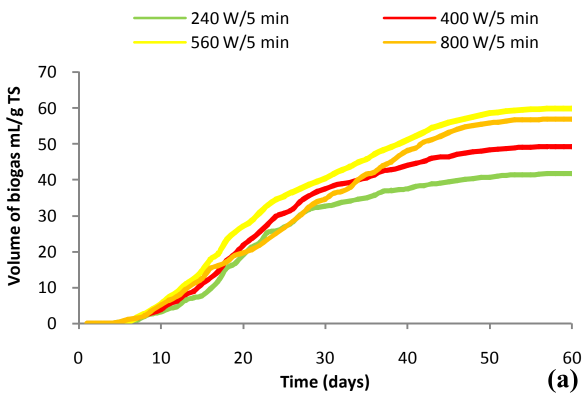


Figure 2. Biogas production of pretreated and unpretreated fresh water hyacinth under various conditions



(a)

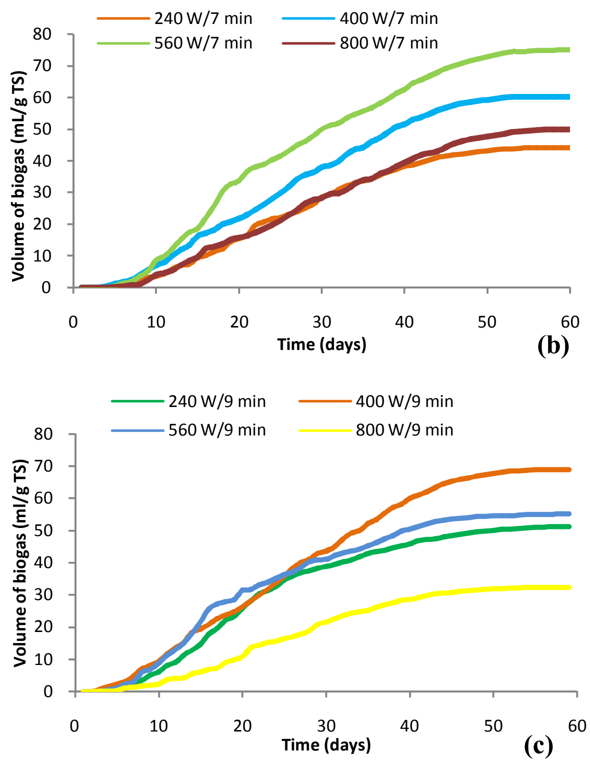


Figure 3. The effect of microwave power on the pretreatment of fresh water hyacinth on biogas production at microwave heating time of (a) 5 min, (b) 7 min and (c) 9 min

Figure 3(a) shows that the production of biogas increased when fresh water hyacinth was pretreated using microwave power 240 up to 560 W for 5 min. But, when using 800 W, production of biogas decreased though the amount was higher than that of 400 W. Figure 3(b) shows that same thing happened when heating time was 7 min; production of biogas increased when using microwave power 240 up to 560 W. However, biogas production decreased for pretreated samples using microwave power 800 W. The maximum biogas production was obtained at 560 W. The pretreatment of fresh water hyacinth with heating time of 9 min, increased production of biogas when using microwave power 240 up to 400 W as shown in Figure 3(c). But, production of biogas decreased when using microwave power 560 up to 800 W. The minimum of biogas production happened when using microwave power of 800 W.

In microwave pretreatment process, the complex structure of lignin is broken down into its constituent structures simpler. Interactions of water hyacinth and microwave causes the separation of cellulose microfibrils wrapper attached to the cellulose and hemicellulose and lignin in the wall of cell. Though microwave energy may not be enough to break chemical bonds, but some of the hydrogen bonds can be weakened or broken [50]. At higher microwave power more molecules become biodegradable and bacteria can penetrate easily and use digestible food for the anaerobic digestion process. Even though, the increase in pretreatment microwave power was not always followed by an increase in the production of biogas [25]. Water hyacinth as a lignocellulosic material has two types of surface area: external and internal. The external surface is related to the size and shape of the particles, while the internal surface relates to the capillary structure of the fiber. The interaction of microwaves with the material at the time of pretreatment will produce thermal effects. Heat effect is the response of polar molecules and ions to changes in the direction of the electric field generated by electromagnetic waves at microwave frequencies. The higher power used to generate microwave energy can generate higher electric field. The higher strength of electric field can generate higher amplitude of the waves. Rotational speed of polar molecules have a linear relationship with the amplitude of the microwave. The higher amplitude causes the polar molecules to rotate faster, so heat is formed rapidly [51]. The water are polar molecules. The rapid motion of water molecules of water hyacinth creates heat and causes swelling at the time of microwave pretreatment so can extend internal surface [52]. Moreover, a higher water content in the water hyacinth provides much more solvent to dissolve or break down lignin and hemicellulose during microwave pretreatment [53]. Hemicellulose and lignin can be dissolved in water at a temperature of 180°C under neutral pH conditions. The solubility of hemicellulose and lignin is not only dependent on the temperature but also the water content and pH [54, 55]. Lignin is located between the cells and in the cell walls and serves as the glue for binding between cells. Lignin limits hydrolysis rate and expansion, because lignin protects and inhibits the substrate digestion. With more lignin dissolved or split apart, bonds of cellulose and hemicellulose are reduced [56]. Some researchers report that there are several factors that affect the hydrolysis process including water content, internal and external surfaces and lignin content in biomass. Zhang and Lynd [57] mentioned that the cellulose will be detached if the internal surface of the pores become larger than the external surface, where it is a case in lignocellulosic biomass. The larger internal surface and reduced or loss of lignin substrates by increasing the number of pores will facilitate the existence of cellulose for hydrolysis by bacteria [58-60]. Therefore, it will speed up the process of hydrolysis. In the process of solving, the hydrolysis carried out by anaerobic bacteria which release specific enzymes and breakdown large molecular units that can not be absorbed directly [61]. The rapid hydrolysis rate will lower the adaptation phase or the minimum time required to start the production of biogas (λ) [52]. As a result, the cumulative biogas production potential (A) and the maximum production rate (U) was lower. Thus, microwave pretreatment is helpful for bacteria in the formation of biogas. The pretreated fresh water hyacinth at microwave power 560 W /7 min produced the highest number of cumulative biogas compared to unpretreated sample (control).

The increase of power and heating time of microwave pretreatment are concomitant with the temperature rise in microwave oven. The production of biogas from pretreated fresh water hyacinth decreased when using microwave power of 560 W for 7 min. The same thing was experienced by Sapci [62]. Sapci [62] states that the increase in temperature of microwave oven from 200 to 300°C in the pretreatment of straw lowered the concentration of cellulose and hemicellulose. Same observation was also reported by Chen and Kuo [63] where the temperature of 180° C was identified as suitable microwave condition at the time of degradation of cellulose. Above this temperature, the specific microwave energy effect on the rate of cellulose degradation is increased significantly [64]. Similarly, pretreatment of municipal solid waste at a temperature of 175°C, decreased biogas production due to the formation of the inhibitor substance [39]. A number of researchers reported on activated slugde waste and other organic materials that undergo chemical, thermal pretreatment and the combination of them can form complex components that inhibit or hinder anaerobic digestion [32, 65, 66]. Pretreatment of biomass at temperature of 290 °C resulted in large amounts of hemicellulose and cellulose to be broken or damaged. But besides destruction of hemiselulose structure, product inhibitors are also produced as phenol compounds, heterocyclic components, furfural and hydroxyimethylfurfural. These compounds are inhibor or toxic to microorganisms for bioconversion processes [26]. Pretreatment of municipal solid waste at a temperature of 175°C, decreased biogas production due to the formation of inhibitor substance [39].

Generally, the results showed that pretreatment of fresh water hyacinth using microwave had a positive impact on the cumulative amount of biogas produced after anaerobic digestion. The amount of cumulative biogas produced from pretreated of fresh water hyacinth was higher than that resulted for water hyacinth without microwave pretreatment. This is similar to results of previous studies who reported that the amount of biogas produced from samples undergone microwave pretreatment was higher compared to unpretreated sample [27, 29, 31, 37-39, 41, [67].

3. 2. Kinetics of Biogas Production

The production of biogas is assumed to be a funtion of bacterial growth in batch digesters. Modified Gompertz equation relates cumulative biogas production and the time of digestion, biogas yield potential (A), the maximum biogas production rate (U) and the duration of lag phase (λ) [7, 10, 42-44, 48, 49]. The value of A, U and λ are listed in Table 1.

Table 1 shows that at 560 W/7 min the optimum condition of microwave pretreatment was obtained to produce the maximum amount of biogas production. At optimized condition, the lag time for biogas decreased to 4.6031 from 8.9717 days. The biogas production rate (U) for 800 W/9 min was the lowest with 1.1203 mLg TS day and the highest at 560 W/7 min with a value of 2.2653 mL/g TS day. The maximum amount of biogas was obtained at 560 W/7 min with a value of 78.2300 mL/g TS. Figure 4 shows modified Gompertz equation for experiment 560W/7 min and for unpretreated sample. From Figure 4, it can be seen that modified Gompertz equation fits best to kinetic data.

4. CONCLUSIONS

The microwave pretreatment of fresh water hyacinth improved biogas production.The best condition of microwave pretreatment of fresh water hyacinth was observed at microwave power of 560 W and heating time of 7 min.

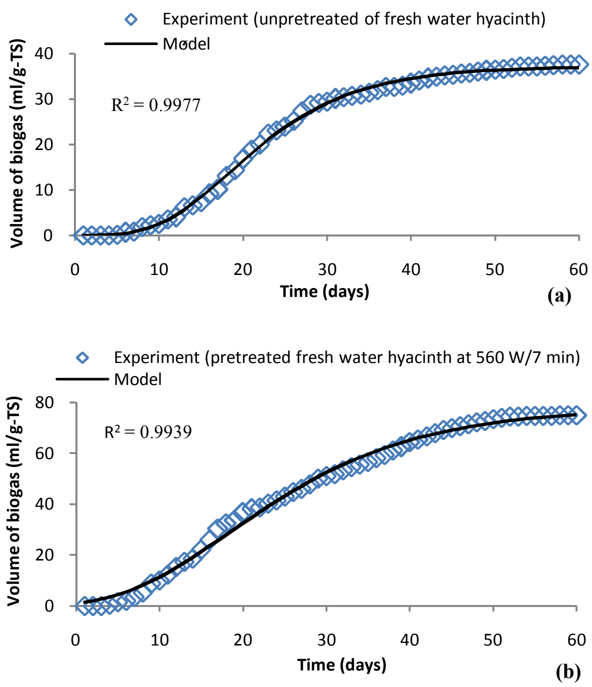


Figure 4. Modified Gompertz equation for (a) unpretreated water hyacinth and (b). pretreated water hyacinth at 560 W/7 min

TABLE 1. Summary of kinetic data of fresh water hyacinth

Variable of digesters	A (mL/g TS)	U (mL/g TS.day)	λ (day)	R^2
240 W, 5 min	41.5279	1.8160	8.8045	0.9979
240 W, 7 min	47.0887	1.4141	8.0367	0.9987
240 W, 9 min	51.2745	1.9521	7.1021	0.9980
400 W, 5 min	49.6371	1.9549	7.1604	0.9990
400 W, 7 min	65.9328	1.7631	6.4874	0.9970
400 W, 9 min	75.7895	1.9701	6.3286	0.9974
560 W, 5 min	61.4109	1.9689	5.4552	0.9955
560 W, 7 min	78.2300	2.2653	4.6031	0.9939
560 W, 9 min	55.8511	1.9568	5.0984	0.9929
800 W, 5 min	61.9641	1.6941	6.6821	0.9969
800 W, 7 min	55.5178	1.3867	7.2104	0.9972
800 W, 9 min	34.2757	1.1203	9.8580	0.9986
Control	37.1617	1.6422	8.9717	0.9977

At the optimum condition of microwave pretreatment, the initial lag phase of anaerobic digestion decreased.The pretreated fresh water hyacinth had higher volume of biogas compared to unpretreated water hyacinth. Therefore, the microwave pretreatment had positive impact on batch mode anaerobic digestion of water hyacinth.

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Microwave Pretreatment of Fresh Water Hyacinth (*Eichhornia crassipes*) in Batch Anaerobic Digestion Tank

RESEARCH NOTE

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چکیده

هدف از این پژوهش، بررسی اثر قدرت مایکروویو در پیشتیمار گل سنبل آب شیرین در تولید بیوگاز بود. تغییرات سطح قدرت مایکروویو ۲٤۰، ٤۰۰، ٥٦۰ و ۸۰۰ وات بودند. تغییرات زمان حرارت مایکروویو ٥، ۷ و ۹ دقیقه بود. سنبل آب شیرین پیشتیمار نشده به عنوان شاهد استفاده شد. نتایج تحقیق نشان داد که تولید بیوگاز از سنبل تازه پیشتیمار شده در مایکروویو بهبود یافت. پیشتیمار مایکروویو، تاثیر مثبتی بر زیست تخریب پذیری بی هوازی سنبل آب شیرین داشت. حداکثر تولید بیوگاز از سنبل آب شیرین پیشتیمار شده در مایکروویو در ٥٦۰ وات به مدت ۷ دقیقه به دست آمد. در این شرایط، سنبل آب شیرین پیشتیمار شده به میزان ۷٥/۱۲ میلی لیتر بیوگاز به ازای هر گرم از جامد کل تولید کرد. در حالی که سنبل آب شیرین پیشتیمار نشده ۳۷/٥٦ میلی لیتر بیوگاز به ازای هر گرم از جامد کل تولید کرد. بیشترین مقدار ثوابت سینتیکی تولید بیوگاز از جمله بازده بیوگاز، ماکزیمم نرخ تولید بیوگاز و مدت زمان فاز تاخیر به ترتیب ۷۸/۲۳ میلی لیتر بیوگاز به ازای هر گرم از جامد، ۲/۲٦ میلی لیتر بیوگاز به ازای هر گرم از جامد در هر روز و ٤/٦۰ روز به دست آمد.

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