Biogas Production Potential of the Microwave, H_2O_2/MW and $S_2O_8^{2-}$/MW Pretreated Wastewater Sludges

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Abstract

The wastewater sludge stabilization by anaerobic digestion is sufficient to reduce the organic content of the sludge, so that it can be safely disposed of without causing odor problems and pathogen contamination, while producing energy in form of biogas. Efficiency of anaerobic digestion and biogas/methane production can be enhanced by pretreating the sludge prior to anaerobic digestion. In this study, the effects of microwave (MW), combined hydrogen peroxide/microwave (H_2O_2/MW) and combined persulfate/microwave ($S_2O_8^{2-}$/MW) pre-treatments on the digestion efficiency and biogas production potential of wastewater sludges were investigated. The unpretreated (control) and the pretreated sludge samples were mixed with the inoculum with an inoculum to substrate ratio (I:S) of 1:1 (w/w on VS basis). The reactors were anaerobically digested at 37°C for 40 days. The MW and H_2O_2/MW pre-treatments applied to sludge samples prior to anaerobic digestion speeded up the hydrolysis step and improved the biodegradability of the organics by increasing their solubility. Application of MW and combined H_2O_2/MW pre-treatments increased the biogas yields by 52% and 13% and methane yields by 64% and 38%. In the $S_2O_8^{2-}$/MW pre-treatment, the concentration of $S_2O_8^{2-}$ created an inhibitory effect on the methanogens.

1. INTRODUCTION

The increasing amount of sewage sludge production in wastewater treatment plants (WWTP) has become a serious problem. To overcome the rising problem, the sewage sludge disposal amount should be diminished by the application of varied sludge treatment and pre-treatment methods. The sludge stabilization must be applied to wastewater sludges for the safely disposal of sludges by eliminating pathogen contamination and odor problems.

Anaerobic digestion (AD) is the most known method for sludge stabilization. Anaerobic digestion helps sludge stabilization by reducing odors, pathogens and the sludge amount. AD also provides the recovery of renewable energy with the biogas production [1–3]. Anaerobic digestion is a conversion process of biodegradable material in microorganisms into biogas in the absence of oxygen, and it comprises mainly methane (CH_4) and inorganic end-products like carbon dioxide (CO_2). Anaerobic digestion of organic material occurs in four stages, hydrolysis, acidogenesis, acetogenesis, and methanogenesis [4–6].

The performance of anaerobic digestion of sludges can be increased with the application of various physical (thermal, mechanical, ultrasonic, microwave), chemical (alkaline, hydrogen peroxide, ozone oxidation), and biological (enzymatic) pre-treatment methods or their combinations. These pre-treatments improve the biodegradability of sludge solids, biogas/methane production and removal of the micropollutants [3,5,7,8].

Advanced oxidation pre-treatment techniques have gained importance in recent years. Hydrogen peroxide (H_2O_2) and ozone (O_3) applications in sludge have been investigated by many researchers. Although ozone is a very strong oxidant, ozonation is an expensive process that limits large-scale application. For this reason, applications of other chemicals such as hydrogen peroxide (H_2O_2), Fenton (hydrogen peroxide/iron catalyst) has come to the forefront. Hydrogen peroxide is the simplest peroxide and act as a strong oxidant, enhancing the sludge disintegration. H_2O_2 pre-treatment can be improved by the heat application to the sludge samples as a combined pre-treatment method in direct heat or microwave irradiation [9–15].

The persulfate anion is the one of the strongest oxidant of the per oxygen family, and it is more stable than hydrogen peroxide and ozone [9]. The persulfate is generally obtained from the persulfate salts. The sodium form of the salts, sodium persulfate ($Na_2S_2O_8$), is the most commonly used one. It is a white crystalline solid and a reactive oxidant. It can be used for the degradation of organic compounds in contaminated soil and groundwater (Block et al., 2004). The persulfate as a powerful oxidant is used for the environmental purposes like organic pollutant destruction in water, wastewater and sludge, sludge pre-treatment, soil and groundwater amelioration [16–20].

Microwave irradiation is another common sludge pre-treatment method creating a thermal effect for the disintegration of the sludge flocs. Microwave pre-treatment improves the release of organic components such as soluble COD, soluble proteins and soluble carbohydrates in the liquid phase of the wastewater sludges [21,22].

Combined pre-treatment methods give more effective results than the single pre-treatment methods. Therefore, it is expected to reach higher biogas production efficiencies with the application of combined pre-treatment methods to sludge samples prior to anaerobic digestion. However, there are some contradicting studies in literature reporting that the H_2O_2/MW and $S_2O_8^{2-}$/MW pre-treatments resulted with lower biogas yield compared to MW pre-treatment [10,16,23,24].

This study investigates the effects of microwave (MW), combined hydrogen peroxide/microwave (H_2O_2/MW) and combined persulfate/microwave ($S_2O_8^{2-}$/MW) pre-treatments on the biogas production potential of wastewater sludges and the anaerobic digestion efficiency in terms of organic removal rates.

2. MATERIALS AND METHODS

2.1. Sludge Samples

The waste water sludge samples were obtained from recirculation unit of a biological wastewater treatment plant located in Istanbul. The inoculum sludge was supplied from the anaerobic digester of the same plant.

Characterization of the sludge samples used in this study is shown in Table 1.

2.2. Sludge Pre-treatments

Sludge samples were pre-treated with MW, H_2O_2/MW and $S_2O_8^{2-}$/MW pre-treatment methods in this study.

2.2.1. Microwave (MW) Pre-treatment

The microwave pre-treatment was applied to sludge samples by irradiating them for 15 minutes at optimum conditions of 160°C and 2000 kPa in a MW system (Berghoff MWS+3) by using a 5-staged temperature program. The conditions of MW pre-treatment were selected based on the results of a preliminary MW optimization study.

2.2.2. Combined Hydrogen Peroxide and MW Pre-treatments (H_2O_2/MW)

Combined H_2O_2/MW treatment consisted of a preheating stage, H_2O_2 (30% w/w) addition and microwave irradiation stage. First, the sludge samples were heated at 120°C for 15 minutes in the MW system to destruct the biological enzymes in the sludge to avoid the excessive consumption of hydrogen peroxide [25]. After preheating stage, 1 g H_2O_2/g TS was added into the sludge samples and the samples were irradiated in a microwave digester at 160 °C for 15 minutes. The applied peroxide concentration of 1 g H_2O_2/g TS was selected based on the results of a previous study and the literature [26–30].

2.2.3. Combined Persulfate and Microwave Pre-treatments ($S_2O_8^{2-}$/MW)

Combined $S_2O_8^{2-}$/MW pre-treatment was applied to sludge samples by adding 1 g $S_2O_8^{2-}$/g TS and irradiating them in a microwave digester at 160°C for15 minutes. The applied persulfate concentration of 1 g $S_2O_8^{2-}$/g TS was selected based on the results of a previous study and the literature [26,31–33].

2.3. Anaerobic Digestion Period

Anaerobic digestion experiments were performed in six parallel sets of four reactors (total of 24 reactors) including sludges pretreated with different methods. The four main reactor groups were control reactors (unpretreated sludge and inoculum), MW reactors (MW pretreated sludge and inoculum), H_2O_2/MW reactors (H_2O_2/MW pretreated sludge and inoculum) and $S_2O_8^{2-}$/MW reactors ($S_2O_8^{2-}$/MW pretreated sludge and inoculum).

The unpretreated (control) and the pretreated sludge samples were mixed with the inoculum in 120 mL serum bottles (reactors) with an inoculum to substrate ratio (I:S) of 1:1 (w/w on VS basis). The pH values of the reactor contents adjusted to be in the favorable range of 7-7.2 for AD [11,34]. The initial alkalinity concentrations of the reactor contents were adjusted to be around 3000-4500 mg/L as $CaCO_3$ [35,36]. The reactors were sealed and flushed with nitrogen gas for 2 minutes to create an anaerobic environment. The reactors were anaerobically digested at 37°C for 40 days in water baths.

Changes in sludge characteristics were determined weekly throughout the anaerobic digestion process by analyzing one parallel of each reactor.

2.4. Biochemical Methane Production (BMP) Tests

Biogas consists mainly of methane and carbon dioxide, and small amounts of hydrogen sulfide and ammonia, and water vapor [3]. Biochemical methane potential (BMP) test helps to determine the anaerobic digestibility of sludge samples by measuring biogas productions. The BMP assay process was first established by Owen et al. (1979) as a simple and inexpensive procedure to monitor relative anaerobic biodegradability of substrates [37].

The BMP tests in the study were conducted according to the procedure that described by Owen et al. (1979) in serum bottles. Total biogas productions in reactors were measured daily with pressure method by using a manometer (Lutron PM-9107). The biogas compositions were analyzed weekly by using a gas chromatograph (Agilent HP 6850).

3. RESULTS AND DISCUSSIONS

3.1. Sludge Characterization

Sludge characteristics are given in the Table 1.

Table 1. Characteristics of the sludge samples.

Parameter	Unit	Sewage Sludge	Inoculum
TS	g/L	13.5	44
VS	g/L	8.3	20.2
TSS	g/L	13	41
VSS	g/L	8.2	18.4
pH	-	6.6	7.6
COD	mg/L	19576	41805
sCOD	mg/L	255	1106
TOC	mg/L	230	235
TKN	mg/L	880	1935
P	mg/L	310	720
VFA (as acetic acid)	mg/L	3.9	18.8
Alkalinity	mg CaCO3/L	1220	8365

3.2. Sludge Reduction

At the end of the anaerobic digestion period, the total solids (TS) and volatile solids (VS) removal rates were increased with the help of pre-treatments applications. The control reactor, containing unpretreated sludge and inoculum, has initial TS and VS concentrations as 25 g/L and 13 g/L. In the control reactor, TS and VS removal rates were measured to be 15% and 24%. The TS removal rates were obtained as 53%, 28% and 18%, respectively in combined H_2O_2/MW, MW and $S_2O_8^{2-}$/MW pretreated sludge containing reactors. The highest VS removal efficiency was found in reactor including, combined H_2O_2/MW pretreated sludge as 59%. The reactors including $S_2O_8^{2-}$/MW and MW pretreated sludges were resulted with the VS removal rates of 45% and 42%.

Initially, the COD concentrations in the reactors were between in the range of 20000 to 24000 mg/L. The COD removal rates of the reactors containing MW, H_2O_2/MW and $S_2O_8^{2-}$/MW pretreated sludge were found as 58%, 55%, and 41% respectively. The COD removal rate of the control reactor was 18%. Pre-treatments increased the COD removal efficiencies two to three folds.

Wang et al. (2015) used a lower H_2O_2 dose of 0.2 g H_2O_2/g TSS at 100^{o}C and reported that, H_2O_2/MW pre-treatment resulted with 19.35% of COD removal [38]. Zhang et al. (2010), studied the COD removal from landfill leachate by using MW-assisted H_2O_2, peroxymonosulfate (PMS) and persulfate (PS) treatments. They achieved COD removal rates of 43.5%, 80.2% and 97.3% by the applications of MW-assisted H_2O_2, peroxymonosulfate (PMS) and persulfate (PS) treatments, respectively, with a 0.3 mol/L oxidant concentration [39].

The sCOD variation in the reactors during the anaerobic digestion period was represented in Figure 1.

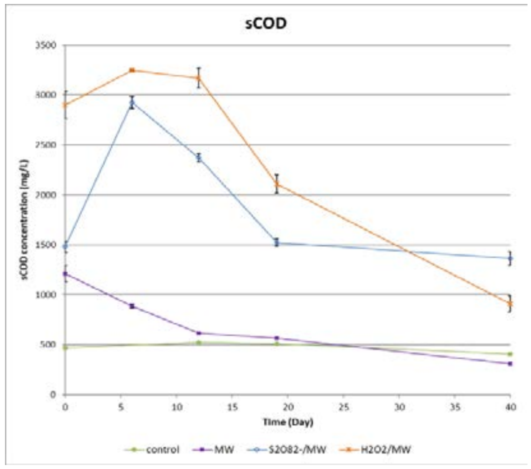

Fig. 1: sCOD variation in the reactors

Initially, the sCOD concentrations in the reactors were between in the range of 470 to 2900 mg/L. The sCOD removal rates of MW and H_2O_2/MW pretreated sludges containing reactors were 74% and 68%. The reactor containing $S_2O_8^{2-}$/MW pretreated sludge was resulted with 8% sCOD removal, while the sCOD removal rate was 14%, in control reactor.

The pre-treatments considerably increased the solubilization of the organics in the sludges. The sCOD of the reactor contents were increased by 618%, 315% and 258% by the application of H_2O_2/MW, $S_2O_8^{2-}$/MW and MW pre-treatments, respectively.

MW and H_2O_2/MW pre-treatments significantly increased sCOD removal. Microwave pre-treatment dissolves the particulate COD by transferring the liquid phase materials from the solid phase. Hydrogen peroxide showed the same effect chemically. Wong et al. (2006) investigated the use of combinations of H_2O_2 pre-treatment and different temperature MW applications for the COD solubilization. 72% and 77% of COD were solubilized into soluble COD with 1 mL H_2O_2 addition at 120°C and 100°C, respectively. 97% and 104% COD solubilization were obtained with the addition of 2 mL H_2O_2 at 120°C and 100°C. Their study showed that the increased temperature of MW may lead to lower COD solubilization [28]. Eswari et al. (2016) resulted that combined MW and hydrogen peroxide pre-treatment (110^{o}C and 0.3 H_2O_2 mg/g SS) obtained COD solubilization up to 50.3% [14].

Although $S_2O_8^{2-}$/MW pre-treatment increased the solubilization of organics about three folds, the sCOD removal efficiency of anaerobic digestion was found to be lower than the efficiency in the control reactor including unpretreated sludge sample. This can be explained with the sudden decrease in pH of the sludge sample after the application of 1 mg $S_2O_8^{2-}$/g TS. The pH of the reactor content decreased sharply to 3.5. and probably affected the activity of microbial population negatively. At the high H_2 partial pressure conditions, propionic acid degrading bacteria can be inhibited and leads to a further decrease in the pH eliminating methane production [40].

3.3. Biogas Production

Biogas productions were slower at the start and the end of the anaerobic digestion. After the acclimatization period (lag phase), an exponential growth of methanogens was observed, and so biogas productions started to increase.

The cumulative biogas production and the biogas yield graphs were given in Figure 2 and Figure 3.

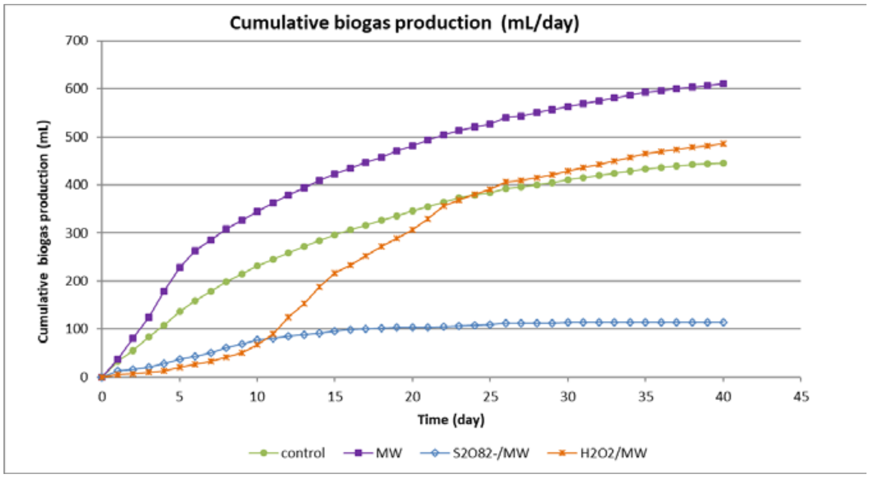

Fig. 2: The cumulative biogas production

As the Figure 2 indicates, MW and H_2O_2/MW pretreated sludge containing reactors produced more biogas than the control reactors. These results showed that the MW and H_2O_2/MW pre-treatment applications improved the biogas production. The cumulative biogas productions were increased by 52% and 13% in reactors pretreated with MW and H_2O_2/MW, respectively.

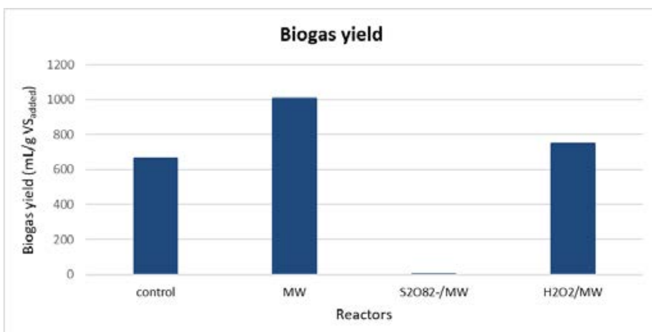

Fig. 3: The biogas yield

The contribution of the inoculum sludge was subtracted from total gas productions in each reactor and the yields were calculated. The highest biogas yield of 1008 mL CH_4/g VS was obtained from MW pretreated sludge in accordance with the cumulative biogas production data. Application of MW and combined H_2O_2/MW pre-treatments to the sludge samples improved the biogas yields in reactors by 52% and 13% and methane yields by 64% and 38%, respectively. The methane contents of the biogas produced in the MW and H_2O_2/MW pre-treatments applied and the control reactors stayed in the range of 55% - 65%, indicating that the methane production was successful. No methane production was achieved by the $S_2O_8^{2-}$/MW pre-treatment.

In this study, the obtained biogas productions were in accordance with the sCOD removals in the reactors. The highest biogas production was obtained from MW pretreated sludges.

In the study of Eskicioğlu et al. (2008), reported that the microwave pre-treatments applied to sludge samples at 65ºC, 75ºC, 85ºC and 175°C prior to mesophilic digestion provided 10.8%, 10.9% 16% and 31% increase in overall biogas production., respectively [41]. In the same way, Alagöz et al. 2015, reported that microwave pre-treatment applied to the sludge samples at 175°C improved the methane yield by 52% [22].

Although, combined H_2O_2/MW pre-treatment was expected to result with higher biogas/methane production than the MW pre-treatment alone, it remained behind the MW pre-treatment. This can be explained with that the residual H_2O_2 or byproducts sourced from the application of H_2O_2 to the sludge samples limited the activity of methanogens, and decreased the biogas production. In accordance with the results of this study, Shahriari et al. 2012 found that the combined H_2O_2+MW pre-treatment produced lower methane production than the MW pre-treatment. They reported that the residual H_2O_2 or byproducts from advanced oxidation can inhibit methanogenesis and decrease biogas production [10]. Accordance with the study, Valo et al. (2004) found that the chemicals addition (H_2O_2 and H_2O_2+$FeSO_4$) can limit the positive effect of high temperature thermal pre-treatments. MW pre-treatment showed higher methane production than H_2O_2 and H_2O_2+$FeSO_4$ combined MW pre-treatments in their study [23].

Persulfate treatment affected the biogas production negatively. This result can be explained with the inhibiting effects of applied 1 g $S_2O_8^{2-}$ /g TS (13.6 g $S_2O_8^{2-}$/L) on the methanogenic bacteria. After the application of $S_2O_8^{2-}$ to the sludge samples, the pH of reactor content decreased sharply to 3.5 and probably caused to the death of methanogenic bacteria. Therefore, the methanogenesis step of the anaerobic digestion process failed. During anaerobic digestion, the content of the biogas, produced in a very low amount, in the reactor was analyzed. There was no oxygen deteriorating the anaerobic conditions. Other inhibition sources, VFA and ammonia were analyzed and seen that the reactors did not have very high concentration of VFAs and ammonia, and the concentrations were in the safe limits for AD process.

Supportively, Isa et al. (1986) showed that sulfate concentrations (Na_2SO_4) up to 5 g of sulfate S per liter did not significantly affect methanogens but, concentrations higher than 10 g of sulfate S per liter created inhibition due to the salt toxicity [42,43]. Zhen et al. (2013), reported that the effect of Fe(II)/$S_2O_8^{2-}$ pre-treatment on anaerobic digestion was dosage dependent, and high dosages had inhibitory effects on the methanogens [24].

4. CONCLUSION

This study investigated the effects of microwave (MW), combined hydrogen peroxide/microwave (H_2O_2/MW) and combined persulfate/microwave ($S_2O_8^{2-}$/MW) pre-treatments on the digestion efficiency and biogas production potential of wastewater sludges. Pre-treatments increased the solubility and the biodegradability of the organics in sludges. Application of MW, combined H_2O_2/MW pre-treatments increased the biogas yields by 52%, 13% and methane yields by 64%, 38% respectively. The $S_2O_8^{2-}$ /MW pre-treatment applied to the sludge samples eliminated the biogas and methane productions by affecting the activity of microbial population negatively.

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