

## Enhancement of Biogas production and organic reduction of sludge by different pre-treatment processes

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### Abstract

Anaerobic digestion (AD) is the most common process for dealing with primary and secondary wastewater sludge. In the present work, four pre-treatment methods (ultrasonic, chemical, thermal, and thermo-chemical) are investigated in Al-Rustumya Wastewater Treatment plant in order to find their effect on biogas production and volatile solid removal efficiency during anaerobic digestion.

Two frequencies of ultrasonic wave were used 30 KHz and 50 KHz during the pre-treatment. Sodium hydroxide was added in different amounts to give three pH values of 9, 10 and 11 in chemical pre-treating processes. The sludge was heated at 60°C and 80°C through thermal pre-treatment experiment. Also, the sludge was treated thermo-chemically at 80 °C and pH 11 prior to anaerobic digestion. Maximum biogas production (6009 ml) was obtained at ultrasonic pre-treatment method with 30 kHz. The volumetric ratio of produced biogas to the initial volume of sludge is about 4:1.

**Keywords:** Anaerobic Digestion, Pre-treatment Methods, Biogas Production, Volatile Solid Removal.

### 1. Introduction

Sludge originated from wastewater treatment processes tends to have concentrated heavy metals and refractory organic compounds as well as potentially pathogenic organisms (viruses, bacteria and etc.). The disposal of sewage sludge has become a critical problem because of the increased cost of sludge disposal. In order to solve this problem, the recycling of sewage sludge has become necessary. Anaerobic is the most common process for dealing with wastewater sludge containing primary and secondary sludge.

Anaerobic digestion (AD) is preferred to be used to reduce the high organic

loading of sludge because of the rapid growth of the biomass that would ensue if the sludge were treated aerobically [1]. Fraser [2] suggested that AD consists of three stages *Hydrolysis*, *Acidogenesis* and *Methanogenesis*. The biological hydrolysis step represents the rate limiting step of the AD process [3]. Many pre-treatment methods were studied to accelerate the rate limiting step and increase the biogas production. For example, Neis et al, [4] studied the enhancement of anaerobic sludge digestion by ultrasonic disintegration. They found that higher removal rates with shorter sludge residence times was established and the

decrease in sludge residence time from 16 to 4 days showed no loss in degradation efficiency. **Sae Eun Oh [5]** investigated the effects of ultrasonic frequency and power on disruption of biosolids in waste activated sludge and examined its effects on methane production of waste activated sludge treated by ultrasonic. His most important conclusions were: first, biosolids disruption in waste activated sludge on ultrasonic treatment was the most effective process at ultrasonic frequency of 40 KHz and power of 0.3watt/ml, second, the ultrasonic exposure of 10 minutes at the 40 KHz and 0.3watt/ml was expected to make a contribution to a higher disruption efficiency of biosolids in waste

activated sludge and third, ultrasonic treatment enhanced the subsequent anaerobic digestion of volatile solids and caused an increase of biogas generation. The literature review of ultrasonic pre-treatment is presented in Table (1).

Solubilization of sludge with alkaline pre-treatment was studied by **Kim et al [7]**. The pH values of sludge were set at 9, 10 and 11 by the addition of 10N sodium hydroxide (NaOH). In the alkaline treatment, the suspended solid concentration continuously decreased with the increase of pH from 9-11. The suspended solid removal efficiency of 20.9% for pH 11 was obtained and was in consistent with the finding of **Chen and Coworkers [8]**.

Table 1, Literature review of ultrasonic pre-treatment (Carrere et al, [6])

Reference	Condition	Results
Tiehm et al, 1997	31KHz, 3.6 W,64S, 22d	Increase in VS removal from 45.8% to 50.3%
Tiehm et al, 2001	41KHz, 150min,8d	Increase of VS removal from 21.5%to 33.7%
Chu et al, 2002	20kHz, 0.33wml <sup>-1</sup> ,20 min, 100d	Increase of CH <sub>4</sub> production from 143 to 292 gKg <sup>-1</sup> TS
Bien et al,2004	20kHz , 180W, 60S, 28d	138% Increase of biogas production
Salsabil et al, 2009	20kHz,50d,Batch 108000kj Kg <sup>-1</sup> TS	84% Increase of biogas production
Erden and Filibeli, 2009	20kHz, 35d, 9690 Kg <sup>-1</sup> TS	44% Increase of biogas production
Elvira et al, 2009	30kWm <sup>3</sup> ,20d	37% Increase in biogas production

**Zhang et al [9]** show that during the two-phase sludge anaerobic digestion, the sludge was hydrolyzed and acidified in the first phase, and then methane was produced in the second stage. Pre-treating sludge at pH 10 for 8 days was reported, by which both sludge hydrolysis and acidification were increased, and the methane production was significantly improved. The effect of different sludge pre-treatment methods on methane yield was compared. The pH 10 pre-treated sludge showed the highest accumulative methane yield (398 ml

/gram of volatile suspended solids), which was 4.4 and 3.5 fold of the blank (untreated) and ultrasonic pre-treated sludge, respectively. An overview of chemical pre-treatment studies are shown in Table (2).

Thermal pre-treatment of sludge was introduced to increase the biogas production. In the study of **Wang et al [11]** the effectiveness of lower temperature pre-treatment (60-100°C) on mesophilic 37°C anaerobic digestion of activated sludge was investigated. They concluded that thermal pre-treatment resulted in a significant

increase (30-50 %) in the methane yield. The effect of pre-treatment at 70°C on mesophilic and thermophilic anaerobic digestion of primary and secondary sludge was studied by **Hariklia et al [12]**. The pre-treatment step showed very positive effect on methane potential and production rate upon subsequent thermophilic digestion of primary sludge. The literature review of thermal pre-treating process is represented in Table (3).

Table 2, Overview of chemical hydrolysis pretreatment studies (Appels et al, [10])

Reference	Reagent	Results
Knezevic et al 1995	NaOH	*No significant improvement in VSS reduction and improved gas production with increased NaOH dosage
Inagaki et al 1997	NaOH	*improvement of digestion by 60%
Tanaka and Kamyaba, 2002	NaOH	*60% increase of overall SS reduction
Carballa et al 2006	CaO	*No significant improvement in anaerobic digestion

**Kim et al [14]** studied the thermo-chemical pre-treatments of sludge by using NaOH as alkali at a temperature of 121°C and pressure of 1.5 atm. The study showed that the impact of the rate limiting step reduced by pre-treatment and digestion efficiency of the sludge was consequently improved. The main scope of this work is to improve the digestion of anaerobic process under different pre-treatment processes of sludge, ultrasonic, chemical, thermal and thermo-chemical.

Table 3, Literature review of impacts of thermal treatments on waste activated sludge mesophilic anaerobic digestion (Bougrier et al. [13])

References	Conditions	Results
Haug et al, 1978	175 C, 30 min CSTR, HRT=15 d	Increase of $CH_4$ production from 115-186 ml/g $COD_{in}$ (62%)
Stuckey and McCarty, 1978	175C, 30min Batch, 25d	Increase of convertibility of COD to $CH_4$ from 48 to 68%
Tanaka et al, 1997	180 C, 60min Batch, 8 d	Increasing of methane production (90%)
Fjordside et al, 2001	160 C CSTR, 15 d	Increase of biogas production (60%)
Gavala et al, 2003	70 C , 7 d Batch	Increase of $CH_4$ production from 8.3 to 10.45 mmol/g VS input
Barjenbruch and Kopplow, 2003	121 C, 60 min CSTR, 20d	Increase of biogas production from 350 to 420 ml/g VSS (20%)

## 2- Materials and Methods:

### 2-1 Materials:

The sludge sample used in our experimental work was taken from thickeners that are found in Al-Rustumya waste water treatment plant (WWTP) located in the north of Baghdad-Iraq, which processes 300m<sup>3</sup>/day of waste water. This sample is basically a mixture of primary and secondary waste activated sludge (WAS). The thickened sample is pre-treated by one of the suggested methods and each 1liter of pre-treated sludge is then mixed with 500 ml of inoculums at the ratio of 2:1(v/v) prior to final digestion as shown in

Fig.1. This mixing of thickened sludge with inoculums is very helpful to accelerate hydrolysis step and increase anaerobic bacteria (i.e. to shorten digestion time) [10]. The inoculums

used is a digested sludge for 14 days at 37°C operating temperature and pH 7. The general characteristics of raw and inoculums of each pre-treatment are shown in Table (4).

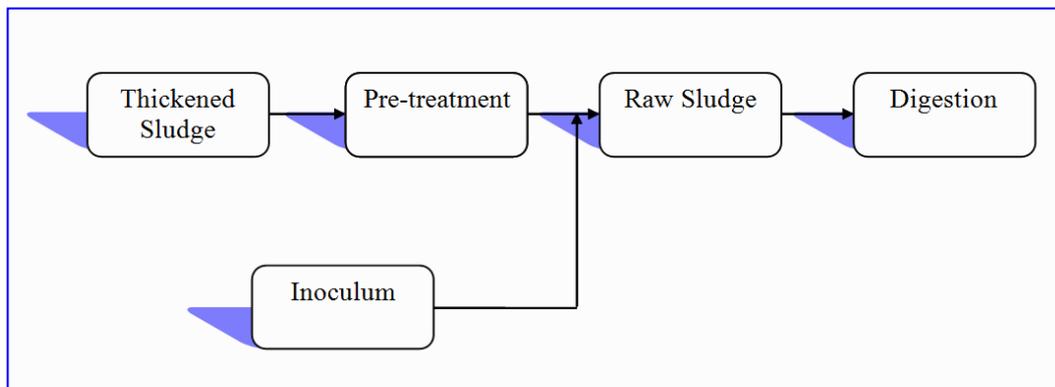


Fig. 1, Final sludge preparing steps

Table 4, Main sludge sample characteristics.

Parameter	Raw Sludge			Inoculum		
	Minimum Value	Maximum Value	Mean value	Minimum Value	Maximum Value	Mean Value
TS (g/ml)	0.128	0.161	0.152	0.106	0.146	0.132
VS (g/ml)	0.049	0.062	0.056	0.039	0.052	0.0462
NVS (g/ml)	0.096	0.112	0.0956	0.066	0.107	0.0862
TSS (g/ml)	0.102	0.139	0.1248	0.084	0.138	0.112
VSS (g/ml)	0.04	0.055	0.0482	0.031	0.045	0.0372
NVSS(g/ml)	0.058	0.095	0.0766	0.051	0.106	0.0748
SCOD(g/l)	2.0	3.45	2.52	3.4	5.6	4.05
pH	6.8	8.2	7.46	6.9	7.4	7.26

### 2-2 Blank Test (Control):

In order to study the enhancement of digestion, the blank test (control) was done. It is anaerobic sludge digestion of 1.5 liter sludge (1liter raw sludge + 0.5 liter of inoculums) at the condition of 37°C, pH 7 and sludge retention time of 14 days.

### 2-3 Pre-treatment of Sludge:

#### 2-3.1 Ultrasonic

Two 1liter of sludge samples were filled in stainless steel bath. One of them was exposed to 30 kHz and the other to 50 kHz frequency for 120 minutes. This was then mixed with inoculums sample in the ratio of 2:1 (v/v) to a final volume of 1.5 liters.

### 2-3.2 Chemical

For alkaline pre-treatment a 3liters sludge sample was filled into three beakers of 1 liter capacity each and pH values of samples were set at 9, 10 and 11, respectively, by the addition of 10N sodium hydroxide. The beakers were mechanically stirred at 250rpm for 2hr. After alkaline pre-treatment, the hydrochloric acid (HCl) solution of 10N was added to set the pH at the required value (pH 7). The pre-treated sludge was then mixed with inoculums sludge in the ratio of 2:1(v/v) to 1.5liter.

### 2-3.3 Thermal

The low temperature pre-treatment was carried out at 60°C and 80°C in order to enhance thermal solubilization of particulate material. 1liter of sludge was heated by magnetic stirrer-heater for 60 min and the beaker was covered with plastic film (to avoid water evaporation) and gently stirred (10 rpm) to ensure temperature homogeneity. After a 60 min heating period, this sludge was cooled down to ambient temperature (25°C) and mixed with inoculums sludge at the ratio of 2:1(v/v) to accelerate the digestion rate; then the final volume of 1.5 liter was digested.

### 2-3.4 Thermo-Chemical

The sludge sample was chemically pre-treated by the addition of NaOH to set the pH to 11 and then thermally heated up to 80°C. This sample was then cooled down to ambient temperature and buffered by the addition of HCl to normal pH 7. 1liter of this sample was then inoculated at the ratio 2:1 (v/v) to a final 1.5 liter sludge sample.

## 2-4 Experimental Procedure

Sludge mixture is digested through 14 days in 2 liter jacketed bioreactors. Organic matter was continuously mixed at a rate of 10 rpm to get

uniform temperature and pH distribution. Produced gas mixture was then washed by concentrated basic solution of NaOH (10N). The acid gases ( $H_2S$ ,  $CO_2$ ) are trapped by NaOH and the remaining gas (methane) flows up to gas collector. The produced pressure through digestion, displace the equivalent volume of water to calibrated cylinder. The collected water volume indicates the produced methane gas volume. Fig.2 shows the schematic experimental apparatus. Each experiment was repeated three times, and they gave results with error of about  $\pm 8\%$ .

Three reactors were used to accomplish anaerobic digestion. Each reactor was made of clear plastic with 11.5 cm in diameter and 20 cm height. The reactor top cover contained six holes for gas outlet, pH sensor, temperature sensor, mixer road, acid and/or base feeding and sludge inlet or outlet.

The sample was drained from bottom sampling hole as shown in Fig. 2.

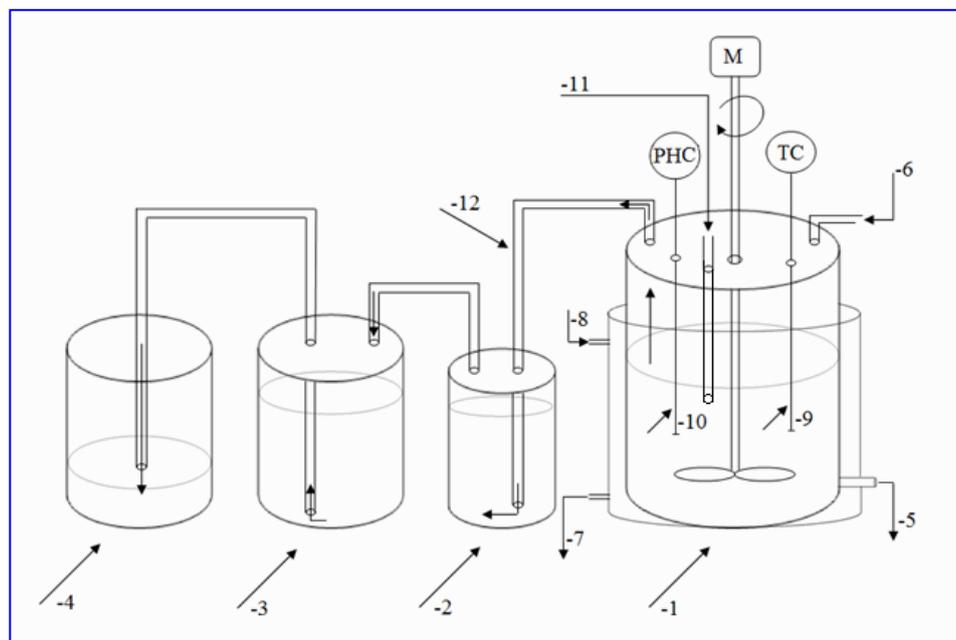
The plastic top cover can be easily opened and closed after each run to clean the reactor. The jacket is made of clear plastic with 13cm diameter. Cooling / Heating water circulated continuously by water bath. Fig. 3 shows the schematic reactor apparatus.

## 2-5 Analysis

Volatile solid (VS) amount is the amount of combustible material in a sample. It is determined by an analytical method called "loss on ignition" which is the amount of matter that is volatilized and burned from a sample exposed to air at 550 °C for 2 hours. The organic (carbon containing matter) is lost and the remaining matter is the mineral or ash component of the original sample. VS amount is usually reported as percent of total solids (TS), where TS are the sum of the VS and ash components [15].

After drying, total solids were incinerated in the same furnace at 550 for 2 hours. The solid remaining volatile solid. Each analysis was done three times a week according to

represents ash content (Non Volatile Solid (NVS)). The difference between total solids and ash content called standard methods of 2540G (APHA, AWWA, WEF [16]).



1- Anaerobic Jacketed Reactor	5- Sampling Hole	9- Temperature Sensor
2- Acid Gas Scrubber	6- Inlet Hole	10- pH Probe
3- Gas Collector	7- Water Outlet Hole	11- Acid/Base Inlet Hole
4- Calibrated Cylinder	8- Water Inlet Hole	12- Plastic Tube Connection

Fig.2, The Schematic experimental apparatus

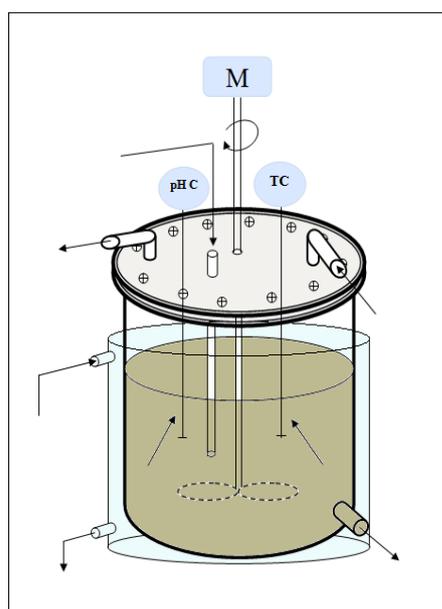


Fig.3, The Schematic reactor apparatus

### 3- Results and Discussions:

#### 3-1 Blank Test:

Fig.4 displays the accumulated biogas through blank digestion test. It showed that accumulated biogas was gradually increased with digestion time.

According to this figure the biogas production was started from the 1<sup>st</sup> day and increased continuously at slow rate to reach the value of 295ml at the final day.

This increase is compatible with the volatile removal fraction. The collected biogas at the end of digestion was 3309 ml. Also it led to low VS reduction through 14 days of digestion. Fig.5 illustrate that the first value of VS starts at 0.053 gm/ml and then roughly

drops to a final value of 0.045 gm/ml after a long time of digestion. Since the VS was the basic food of anaerobic bacteria; therefore its amount continuously decreased during digestion process. This decrease can also be explained if we remember that the anaerobic digestion was carried out through three general steps of hydrolysis, acidogenesis and methanogenesis. Through the hydrolysis step the particulate insoluble organic matter was converted to soluble organics by enzymatic reactions. During acidogenesis step, the dissolved organic materials were converted to amino acids, hydrogen and carbon dioxide and finally the methanogenesis bacteria consume the organic acid to produce methane gas. The continuation of the previous three steps within 14 days of the digestion process leads to a conversion of a large amount of volatile organic compounds to methane gas.

The total VS removal efficiency of sludge, when exposed to biological treating (i.e. AD) reached a value of **15.09%** after conventional digestion at 37°C and pH 7. This value is possibly improved and maximized by using one of the suggested pre-treatment methods.

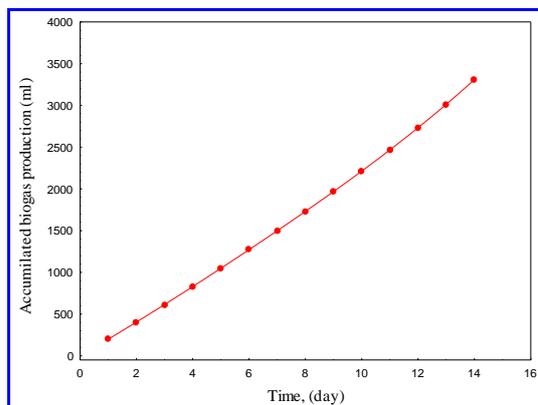


Fig.4, Accumulated biogas during blank test

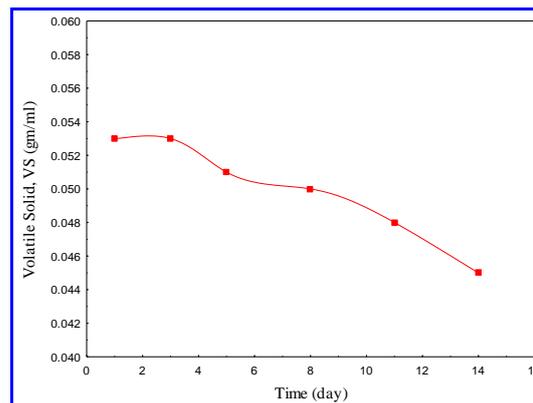


Fig.5, Volatile solid concentration variation through Blank test

### 3-2 Ultrasonic Pretreatment:

Fig.6 represents the accumulated biogas after digestion of sonicated sludge, during 14 days. According to this figure the biogas production was increased continuously for both studied frequencies. For example, the digestion of 30 kHz pre-treated sludge yielded 6009 ml of biogas at the end of fermentation period while the digestion of 50 kHz pre-treated sludge yielded lower volume of biogas which was 4397 ml for the mass sludge sample. By giving attention to accumulated biogas from untreated sludge digestion 3309ml, it could be observed that the significant enhancement was reached after sludge pre-treating. Hence, an increase of 81.6% of biogas production was achieved at lower ultrasonic frequency. Similar results were obtained by the work of **Salsabil et al [17]** and those obtained in the work of **Elvira [18]**, were much higher while higher quantity of biogas was produced by the work of **Bien [19]**. This difference may be due to the variation in the operating condition.

The primary aim of ultrasonication pre-treatment process is to increase the sludge biodegradability to enhance the biogas production at lower SRT in anaerobic digester. This is done by the ability of Ultrasonication to induce cavitations, which lysis the cell walls of microbes and releases the

intracellular components into the aqueous phase. The intracellular biopolymers solubilisation and conversion to the lower molecular weight compounds of sludge through hydrolysis; is a rate limiting step. Therefore, the sonication parameters affect cavitations and accelerate the rate limiting step which effects on the sludge digestion.

The volatile solid variation along the digestion period is shown in Fig.6.

The performance of anaerobic reactors was mainly evaluated in terms of increase in VS reduction and biogas [20]. The higher VS solid removal efficiency resulted from the 30 kHz of pre-treated sludge (57.7%) and next resulted from 50 kHz of pre-treated sludge (33.4%) as shown in Fig.6. In other words, the process of cracking (as is evident in above figure) at 30 kHz is much better than 50 kHz and this is due to the phenomenon of the formation of bubbles as they grow up with the decrease in frequency, and the larger the bubbles, the more powerful and disruptive to the bacteria, organic materials particularly high molecular weight polymers, carbohydrates and lipids. The obtained results from processing by the waves are in compatible with other researchers such as **Tiehm et al [21]** who found that the degree of disintegration was decreased when the frequency increased from 41 to 3217 kHz. **Farooq et al [22]** also found that the use of low power ultrasound was more effective than higher sound frequency in stimulating AD.

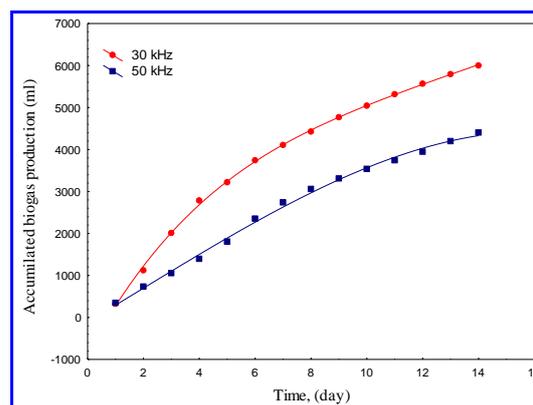


Fig.5, Accumulated biogas after ultrasonic pretreatment

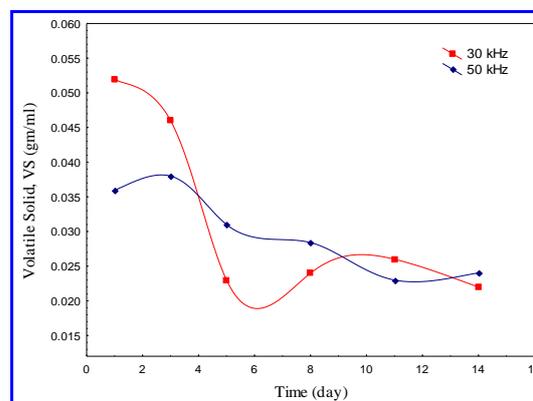


Fig.6, Effect of ultrasonic wave on volatile solid content

### 3-3 Chemical Pretreatment:

The biogas production for different pre-treating pH values is shown in Fig. 7. It illustrates that the biogas accumulation was affected by pH value of alkaline pre-treatment during fermentation process in the range of pH 9-11. The biogas production was increased with the increase of pH value. According to **Katsiris et al [23]**, the NaOH used over pre-treating process was converted to  $\text{Na}^+$  and  $\text{OH}^-$  and when the pH of sludge samples increased, the bacterial surfaces become increasingly negatively charged. This creates high electrostatic repulsion which causes desorption of some part of extracellular polymers. Increasing soluble organic compounds concentration means acceleration of hydrolysis step.

Acceleration of hydrolysis step by alkaline pre-treatment has a benefit of maximizing biogas production as an example. Moreover, the addition of chemical agent through alkaline pre-treatment acts as another technique too. The addition of NaOH to sludge sample saponificated the present lipids and led to increase its lysis. So, alkaline pre-treatment is active when the sludge has a high concentration of lipids. Similar results were obtained by the work of **Zhang et al [9]**. They showed that alkaline pre-treating caused significant increase in biogas production. Much higher biogas yields were observed at pH (11) compared to other conditions.

By comparing the chemically pre-treated sludge with each others, it was found that lower VS removal efficiency was achieved at pH 9 and pH 10 (23.7% and 26.2% respectively) as shown in Fig .8. Significant improvement was done by pH 11 pre-treated digester, which reached 44.1% after 14 days SRT.

Generally, chemical pre-treatment can enhance the subsequent anaerobic digestion by the effect of alkali on microorganism and on the complex organic compounds of high molecular weight. Regarding low VS removal improvement from chemical pre-treatment digester, it does not mean that the pre-treatment could not break down the compounds but it is due to the addition of NaOH. Additional Na+ into the raw sludge resulted in increasing TS content while sludge solubilization was taking place. Therefore, the final TS amount of digested sludge was found to be relatively high. This result differs from that obtained by **Kenzevice [24]** and **Carballa [25]** who found that there was no significant improvement in the value of VS removal efficiency with increasing NaOH dosage. This study

agrees well with the work of **Tannaka[26]**.

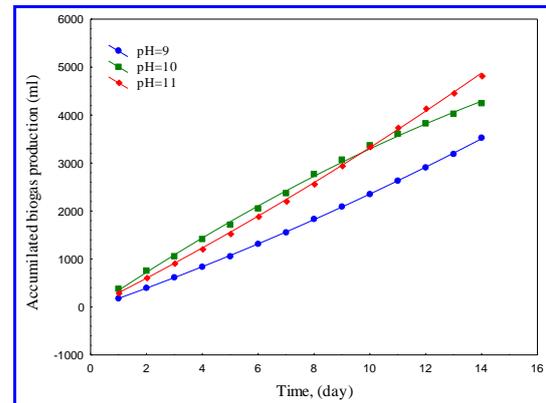


Fig.7, Accumulated biogas after chemical pre-treating

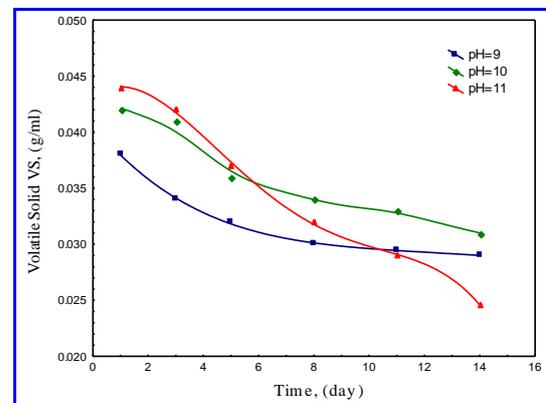


Fig.8, Effect of alkaline pre-treatment on volatile solid content

### 3-4 Thermal Pretreatment:

Accumulated biogas of both thermal pre-treating conditions is shown in Fig. 9. According to this figure, there is no significant increase in total accumulated biogas at the end of digestion period. Depending on Fig. 9, the pre-treatment of sludge by 60°C temperature has resulted in lower biogas accumulation 3643ml as compared to 3749ml at 80°C pre-treating temperature. However, thermal pre-treatment methods improved the organic solubilization (acceleration of hydrolysis step through digestion) in addition to enhancement of sludge dewaterability. The increasing of biogas production after thermal pretreatment was about 10-13% than that of blank

test. Similar results were obtained by the work of **Gavala et al [27]**. While higher increase of biogas production was produced by the work of **Wang et al [11]** of about (30-50)% when sludge was pretreated thermally at about (60-100)<sup>0</sup>C.

Fig. 10 illustrates the modification of VS concentration through 14 days of digestion after both 60 and 80°C pre-treated sludge. According to that figure, the VS concentration of 60°C pre-treated sludge decreased from **0.04 gm/ml** to **0.031 gm/ml** after long period of fermentation process. While VS concentration in 80°C pre-treated sludge passed a wide range of variation starting at the point of **0.044 gm/ml** and reached the point of **0.03 gm/ml** after the same fermentation period. This difference in VS variations indicates that 80°C of pre-treated sludge were digested better than 60°C pre-treated sludge. This is due to the higher consumption of VS by present anaerobic microorganism.

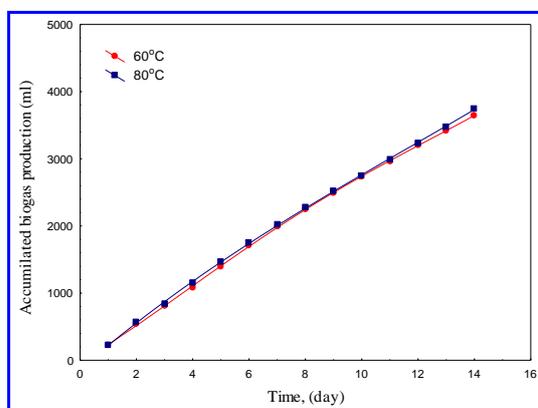


Fig. 9, Accumulated biogas after thermal pre-treating

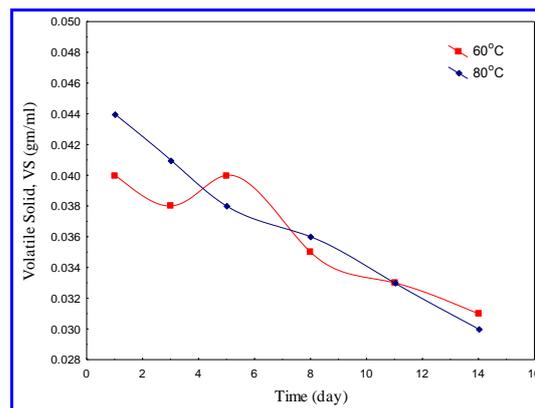


Fig. 10, Effect of thermal pre-treatment on volatile solid content

### 3-5 Thermo-Chemical Pretreatment:

Thermo-chemical pre-treating process is one of suggested techniques led to higher biogas production at the end of experiment. Fig.11 shows the accumulated biogas after sludge pre-treating process which led to accumulation of 5098 ml after 14 days of digestion. About 55.1% of total biogas was accumulated during the second week, so higher SRT was required to increase biogas production as much as possible. **Dogan and Dilek Sanin [28]** showed that thermo-chemical pre-treating process affected the final biogas production during 47 days of solid retention time. The higher biogas was achieved by MW+ pH 12 and MW + pH 11 and for both cases they were 18.9% more than the blank test.

On the other hand, Fig. 12 shows VS concentration drops from **0.055 gm/ml** to **0.026 gm/ml** continuously through disintegration period. The interesting value of total volatile solid removal efficiency of **52.73%** was achieved after two weeks of sludge disintegration and only during the second week of digestion. **34.8%** of total VS content was removed because of high bacterial activity. This amount of removal was reflected on the final biogas production. This result is in consistent with the work of **Kim, et al [14]**.

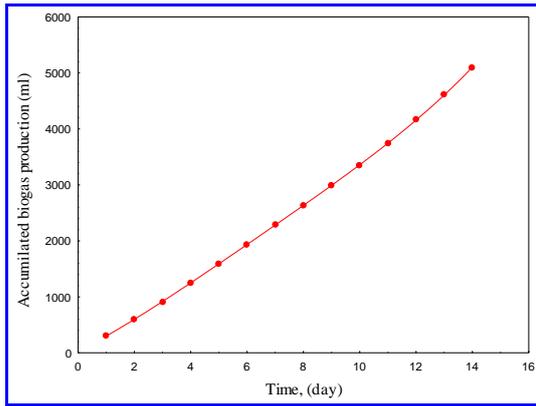


Fig. 11, Accumulated biogas after thermo-chemical pre-treatment

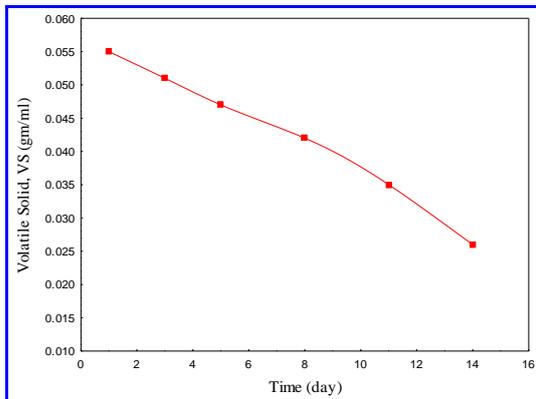


Fig.12, Effect of thermo-chemical pre-treatment on volatile solid content

#### 4-Conclusion:

The following are the main conclusions drawn from this study:

1- All the method, which have been suggested in this paper of pre-treatment acted successfully on different levels to enhance the digestion efficiencies, compared to control (blank test). These methods reduce the digestion time so the volume of the digesters may be reduced and hence less capital cost is required.

2- Arrangements of improving efficiencies of digestion processes depending on percentage increasing of biogas production which has been achieved in this study are described as follows:

Ultrasonic 30 kHz (81.6%) > Thermo-chemical pH (11) + 80°C (54.06%) > Chemical pH (11) (45.91%) >

Ultrasonic 50 kHz (32.9%) > Chemical pH (10) (28.41%) > Thermal 80°C (13.3%) > Thermal 60°C (10.10%) > Chemical pH (9) (6.38%)

3- The increasing of organic waste removal efficiency after pre-treating (without pre-treatment) is arranged as follow:

processes, compared to blank test  
Ultrasonic 30 kHz (42.9%) > Thermo-Chemical pH (11) + 80 °C (37.63%) > Chemical pH (11) (29%) > Ultrasonic 50 kHz (18.24%) > Thermal 80°C (16.72%) > Chemical pH (10) (11.09%) > Chemical pH (9) (8.58%) > Thermal 60 °C (7.4%)

#### Acknowledgment:

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#### Nomenclature:

SCOD: Soluble Chemical Oxygen Demand [ppm]; VS: Volatile Solid [gm/ml]; WWTP: Wastewater Treatment Plant; HRT: Hydraulic Retention Time [day]; SRT: Solid Retention Time [day]; RPM: Revolution Per Minute; TS: Total Solid [gm/ml] VS: Volatile Solid [gm/ml] NVS: Non Volatile Solid [gm/ml] TSS: Total Suspended Solid [gm/ml] VSS: Volatile Suspended Solid [gm/ml] NVSS: Non Volatile Suspended Solid [gm/ml]

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