

# Sodium Hydroxide Solution Pretreatment Effect on Anaerobic Digestion Process under Mesophilic Conditions

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**ABSTRACT:** Biogas, latent energy inside organic components, is a by-product of the anaerobic digestion process. Biogas validity depends on methane composition value. Some organic compounds are not easily accessible to active microorganisms in the anaerobic digestion process due to their nature and structure. These compounds are made available to microorganisms by pretreatment and methane composition is increased. In this study, alkaline pretreatment of solid organic wastes by sodium hydroxide solution was studied through the anaerobic digestion laboratory apparatus. A mixed feed of organic solid waste, previous-stage digested matter, and bovine excrement were considered. Three tests were designed. In the first test, the feed was without pretreatment. Solid organic wastes of the second and third tests were immersed in 0.5 M and 0.1 M sodium hydroxide solution for 24 hours in the laboratory, respectively. The setup was subjected to -0.75 bar<sub>g</sub> vacuum and mesophilic temperature conditions. The mechanical stirrer in digester homogenized substrate medium. The digestion period lasted 40 days. The percentage composition of dry matter, organic matter, humidity, ash, carbon, and nitrogen of the feed and digested were measured. Produced biogas analyzed by gas chromatography. According to the results, produced biogas and methane volume respectively at the 3rd test compare to the 1st test increased 15.52% and 21.31%, and in the 3rd test compare to the 2nd test 19.52% and 2.3 times. Vice versa, produced biogas and methane volumes respectively at the 2nd test compare to the 1st test decreased 3.32% and 63.7%. Sodium hydroxide solution destroys the hard tissue of cellulose lignin and hydrolyzes organic polymers. However, in high concentrations acetogenic and methanogenic microorganisms are inhibited by producing volatile fatty acids and saponification.

**KEYWORDS:** Anaerobic digestion; Solid organic wastes; Pretreatment; Sodium hydroxide solution; Biogas.

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## INTRODUCTION

Increasing population growth has increased the production of organic waste [1, 2]. Disposal of this waste by landfilling or releasing it into the environment causes soil and climate pollution that endangers human life and other living organisms [3, 4]. One of the best waste management and disposal methods is the anaerobic digestion process. This method eliminates the problems of waste disposal mentioned above and produces by-products including biogas and solid or liquid organic fertilizer. These products make the anaerobic digestion method economically justifiable [5]. Process productivity increases depend on several parameters and the most effective is biogas methane composition. Produced biomethane is renewable and green energy with the possibility of replacing fossil fuels. In addition, the increasing growth of industry and technology and fossil fuels consumption as finite and un-renewable energy sources on the one hand, and destructive environmental effects due to their consumption by replacing biomethane in some applications can be modified [6, 7]. Also, the process of anaerobic digestion is a way to convert raw animal manure into liquid and solid nutrient fertilizers with biogas production [8]. Production of biomethane in the anaerobic digestion process is dependent on factors such as food type and composition, pretreatment and residence time, temperature conditions, digester type, mixing, and process pressure [9, 10]. Biogas is embedded energy inside organic compound molecules that are released by the anaerobic digestion process. Anaerobic digestion consists of four stages: hydrolysis, acidification, acetogenesis, and methanogenesis which are carried out under microorganisms' influence. Under anaerobic conditions, microorganisms convert simple molecules of sugars such as glucose into biogas. Starch and cellulose are both chains of glucose units. Starch is used as a plant energy source, so it is easy to break. Cellulose acts as a plant wall and retaining structure and its hydrolysis is difficult [9]. Lignin as a cellulosic wall coating of plants is the most complex hydrocarbon polymer with phenyl propane-forming units and has hydrophobic properties that make it resistant to microbial and enzymatic decomposition [11]. Lignin resistance to decomposition specifically reduced biogas production efficiency in the anaerobic digestion process [12]. One of the first steps in the anaerobic digestion process of these compounds is to select the appropriate pretreatment method to remove lignin and make cellulose available for

hydrolytic enzymes [13, 14]. Pretreatment technologies increase access to cellulosic materials carbon and in general, microorganisms contact the surface with nutrients in the anaerobic digestion process. Pretreatment technologies contain physical, chemical, biological, and/or combination methods [15, 16]. These methods include vapor explosion, hydrothermal process, and acidic and alkaline pretreatment [17, 22]. Alkaline pretreatment is a preferable method. Lignin contents are released by using various alkalis, especially KOH,  $\text{Ca}(\text{OH})_2$  and NaOH [23]. Alkaline treatment reduces the polymerization degree, removes the lignin, and makes cellulose available for degradation by microorganisms and enzymes, causing the soaping process [24, 28]. Alkali high concentrations increase pretreatment cost relative to produced energy from waste and produce Volatile Fatty Acids (VFAs) which inhibit the anaerobic digestion process [29]. The aim of this study is to investigate the alkaline pretreatment effect with sodium hydroxide solution on the anaerobic digestion process and biogas production in a laboratory scale set up under mesophilic conditions.

## EXPERIMENTAL SECTION

### Equipment

Fig. 1 shows a set of application equipment for this research. The main part, the digestion tank has been designed and built for double-wall heating and has been insulated with a layer of glass wool to prevent heat loss. A Memmert D91126 Model water bath was used to heat the fluid. In order for materials to mix inside the digester, a stirrer was used which provides the intensity of current and voltage required for rotation by a DC power supply device, model GWINSTEK, GPS-3303C. A thermocouple is used to measure the temperature of the digester inside the material and the measured temperature can be seen on a screen with an accuracy of  $\pm 0.1$  °C. A 1953 WINTERS model gauge pressure measures intra-digester pressure and biogas produced. A PLATINUM, JB, 60507 vacuum pump was used to create anaerobic conditions and to evacuate the system inside air or produced biogas at the end or during the process. An outlet from the vacuum pump has been connected to the flare for the flame test. For gas phase analysis and related tests, two tanks were used one with a volume of 13 mL and the other with a volume of 2 liters are considered in the system. The produced biogas is discharged from the system with a path connected to the safety valve into a 250-liter polyethylene tank.



Fig. 1: Anaerobic digestion equipment set.

### Materials

Mainly the feed and sodium hydroxide solutions are the main ingredients. The feed was a combination of degradable organic matter including household solid organic waste, digested material of the previous stage, and bovine excrement. Sodium hydroxide solution consisted of 0.5 and 0.1 M concentrations.

### Test method

Three tests were done, feed mixture consisting of shredded household solid organic waste, digested matter, bovine waste, and water was selected at same weight ratios with a total weight of 8 kg. 2nd and 3rd test, before feed mixing, the shredded household solid organic waste was immersed inside sodium hydroxide solutions respectively with 0.5 and 0.1 M concentrations under laboratory ambient temperature for one day. Then filtered and washed with plenty of water. Also, their pH was controlled, which should be almost 7 otherwise for receiving 7pH, washing was repeated. In every test, 200 g of the feed mixture was separated for analysis and the rest was entered into the digester. To create anaerobic conditions, after closing the digester door, the system was subjected to a relative pressure of -0.75 bar through a vacuum pump. Mesophilic conditions were provided by hot water circulation between the digester thermal jacket and the Bain Marie bath at 40 °C.

By means of a mechanical stirrer, the substrate medium was homogenized 3 times per week for 3 hours at an average rotational speed of 10 rpm. The biochemical process of anaerobic digestion with biogas production was done gradually. Normally, during a 40-day period, system pressure due to biogas production was increased and after passing a peak decreased and finally stabilized. During this period, the temperature and pressure of produced biogas were regularly measured and recorded through a thermocouple and gauge pressure installed in the system. At the end of the process, the sampling tank was separated for biogas analyses. Then, biogas was discharged into the 250-liter storage tank by a vacuum pump through a safety valve outlet. The Digester door was opened and 200 g of digested material was separated for relevant tests and analyses. Finally, all contents of the digester were discharged and the system was provided for the next series of tests.

### Samples analysis method

Parameters include identifying and measuring the composition of feed, digested products, and biogas. Assayed parameters of feed and digested products are common and similar, some of which were measured by physical-chemical methods and some by instrumental. Physical chemistry parameters include humidity content, dry matter, ash, and organic matter, which were measured according to the national standards of Iran with numbers 10716, 1677, and 13320 [30-32]. The composition of feed and digested products, including carbon, nitrogen, hydrogen, and sulfur, were also measured using the Costech Model Analyzer, Instrument Elemental Combustion System, and Eager 300 for EA1112. Biogas composition was also measured using gas chromatography (GC) with HP-PLOT Q column DG1DCE40DE.

## RESULTS AND DISCUSSION

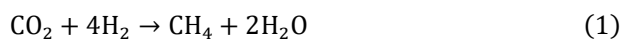
In all three tests, values of humidity content, ash, organic and dry matter of feed, and digested product were measured. Results are shown in Table 1. Materials (feed and digested product) consist of two parts, dry and humidity. By comparing the humidity content of feed and digested product for all three tests and increasing humidity in digested product versus feed is distinguished that part of the feed is converted to water during the biogas production process due to the activity of methanogenic microorganisms (Reaction (1)) [9]:

**Table 1: Humidity, dry matter, organic matter and ash percent composition of feed and digestate.**

Title	1st test		2nd test		3rd test	
	Digestate	Feed	Digestate	Feed	Digestate	Feed
Humidity%	84	75.86	90.71	86.2	89.13	86.62
Dried Matter%	16	24.14	9.29	13.8	10.87	13.38
Organic Matter%	15.81	20.23	7.92	12.50	9.44	11.97
Ash%	0.19	3.91	1.37	1.30	1.43	1.41

**Table 2: feed and digested product nitrogen and carbon composition percent.**

Element Title	1st test		2nd test		3rd test	
	Digestate	Feed	Digestate	Feed	Digestate	Feed
C%	33.67	45.98	38.88	54.93	22.57	42.42
N%	4.64	5.88	2.73	3.7	1.8	4.61



The dried part of feed and digestate consists of organic matter and ash. The organic part has the ability to transform and decompose in an anaerobic digestion process due to microorganisms' activity. Ash can contain tracer elements and compounds required for microorganisms' growth and multiplication for the digestive process, and/or is not degraded and remains a digested part in the digester. Therefore, the reduction in organic matter percentage composition digested into feed will be proportional to the progress rate of the anaerobic digestion process to produce biogas. Also, the reduction rate of ash percentage in digested compared to feed confirms consumption of ash part as required tracer elements and compounds for growth and multiplication of microorganisms in the digestive process. The results of 1st test confirm this but are inconsistent with 2nd and 3rd test results. Considering that the feed organic solid waste section of 2nd and 3rd tests was pre-treated by 0.5 and 0.1 M sodium hydroxide solutions, respectively, these results can be described as follows: large compounds and molecules such as cellulose, proteins, and fats are hydrolyzed and converted into their building blocks by sodium hydroxide solution. High concentrations of sodium hydroxide solution can produce volatile fatty acids in the next step. Even at a later stage, it will cause a soapy process. Active microorganisms in the substrate are not able to degrade compounds in soap. Therefore, these compounds remain in the digested matter and are added to ash because they are not in the group of volatile organic substances [9, 33, 34].

Carbon and nitrogen are the main and essential elements of feed for anaerobic digestion and biogas production. Therefore, identifying and measuring the amount of these elements in feed and digested products will be a measure of feed capacity for biogas production and an improvement rate of the anaerobic digestion process to produce biogas. Carbon is needed for growth and proliferation as well as nitrogen to build cell walls [9, 35, 36]. Feed and digested product elemental analysis results for all three tests are shown in Table 2.

Fig. 2 shows the conversion percentage of carbon and nitrogen in digested products into feed for all three tests. Results show that this rate is higher in 3rd test which was pre-treated with 0.1 M sodium hydroxide solution, than 2nd test which was pre-treated with 0.5 M sodium hydroxide solution, and than the 1st test, which was unprocessed. A high concentration of sodium hydroxide solution causes volatile fatty acids production, lowering pH of the substrate medium and inhibiting biogas production processes. It also leads to a soap process in a further step. Both of these reactions inhibit the biogas production process and as a result elements conversion rate in 2nd test, which is processed with 0.5 M solution, is lower than in 3rd test.

Fig. 3 shows the biogas average percentage composition for all three tests. Methane high percentage combination in 3rd test compared to the other two tests shows that alkaline pretreatment by sodium hydroxide solution optimal concentration makes organic compounds available to active microorganisms in an anaerobic digestion process through hydrolysis of polymers and large molecules and

improves the methanogenic process. In contrast, alkaline pretreatment by high sodium hydroxide solution concentration limits the methanogenic process due to increased concentration of soluble organic acids and soap production. Detergent-containing compounds such as soap actually kill active microorganisms, and thus the high concentration of sodium hydroxide solution acts as an inhibitor in the anaerobic digestion process [37]. The normal volume of collected and measured biogas per feed mass unit for each of tests is shown in Fig. 4. According to measured methane percentage composition (Fig. 3), produced methane normal volume is estimated from biogas normal total volume and is shown in Fig. 4. According to results, produced biogas normal volume per feed mass unit third test, which contains pretreatment organic solid waste by a 0.1 M sodium hydroxide solution respectively is more than the first test where feed was not affected by sodium hydroxide solution and too second test where feed organic solid part was pretreated by 0.5 M sodium hydroxide solution. Also, 3rd test produced methane volume higher than 1st and 2nd tests, respectively. A high concentration of sodium hydroxide solution in 2nd test caused to increase and the accumulation of organic acids soluble then decreased pH so the activity of methanogenic microorganisms decreased or blocked. Also, soap components production follows an increase and accumulation of organic acids soluble and high concentration of sodium hydroxide solution concluded to lyse and death of methanogenic microorganisms. Both of these phenomena decrease biogas and biomethane production. On the other hand, pretreatment with a High concentration of sodium hydroxide solution can destruct organic components so their potential for biogas production decrease.

## CONCLUSIONS

The feed pretreatment technique facilitates an anaerobic digestion process. It makes organic compound molecules more readily available to active microorganisms in the anaerobic digestion quaternary process. In this research, alkaline pretreatment by sodium hydroxide solution has been studied and results follow below:

- Sodium hydroxide solution destroys cellulose lignin hard tissue. Makes molecules available to active microorganisms and causes hydrolysis of large polymers and molecules, which is the first step in the quartet process of anaerobic digestion.

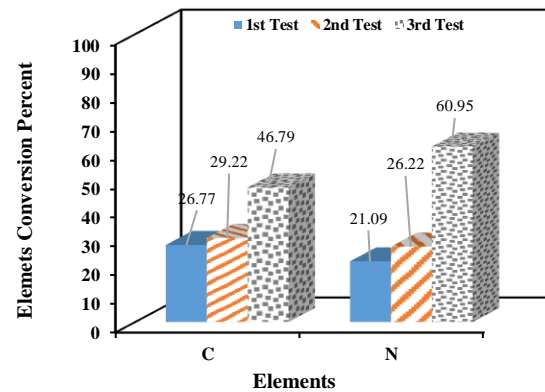


Fig. 2: Comparison of carbon and nitrogen elements percentage conversion in tests.

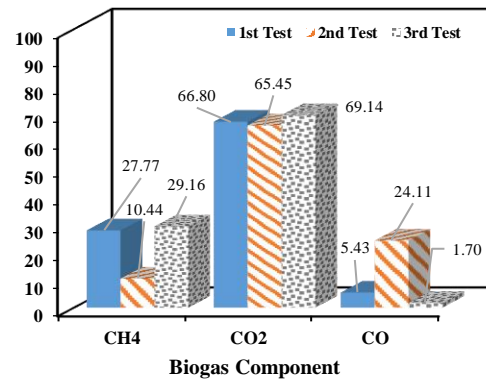


Fig. 3: Produced biogas percent composition in tests.

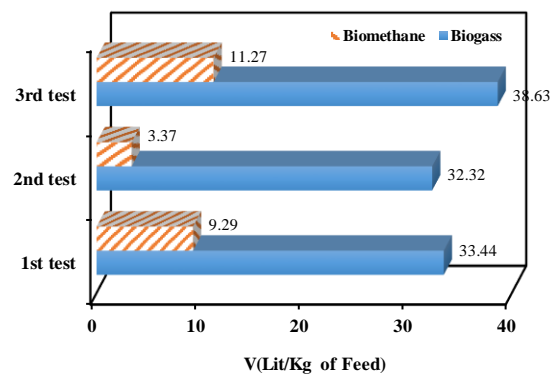


Fig. 4: Produced biogas and biomethane normal cumulative volume per feed mass unit in tests.

- High concentrations of sodium hydroxide solution, in addition to being able to economically increase process overhead costs compared to revenue from biomethane production, form volatile fatty acids, and inhibit acetogenic microorganisms.

- High concentrations of sodium hydroxide solution causes the saponification process of fatty acids and consists of soap compounds. Soap compounds inactivate and kill microorganisms, especially the methanogenic group. It also remains an insoluble compound in digested part and will increase the amount of ash.

- Volume percentage increase of produced biogas and methane in 3rd test, where feed was pre-treated by 0.1 M sodium hydroxide solution, compared to 1st test, which did not have pre-processing stage by sodium hydroxide solution, were 15.52% and 31.31%, respectively and too 3rd test compare to 2nd, which was pretreated by 0.5 M sodium hydroxide solution, was estimated to be 19.52% and 2.3 times respectively.

- Volume percentage reduction of produced biogas and methane in 2nd test compared to 1st due to the dominance of inhibitory effect due to sodium hydroxide solution high concentration was estimated to be 3.32% and 63.7%, respectively.

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