

Anaerobic Digestion of 500 Gallons of Reactor Wastewater Sewage Sludge

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ABSTRACT

Anaerobic digestion is an important biological process used in the treatment of wastewater. The wastewaters in some places such as sewage systems, abattoirs, and agricultural plants have contributed immensely to environmental problems. Anaerobic digestion also applied in the treatment of municipal waste, agricultural waste, sewage sludge, domestic waste, and industrial waste. This has been used in many countries of the world to reduce pollution and as well provide alternative energy source such as biogas. Anaerobic technology has been in existence for decades and has reduced the use of fossil fuels.

This work examined the production of biogas from sewage wastewater otherwise known as sewage sludge. Two 500-gallon capacity tanks were used for the experiment. The sewage wastewater was tested in two categories. The non-inoculated wastewater sewage sludge in reactor 1 and wastewater sewage sludge with cow dung used as inoculum in reactor 2, mixed with water in the ratio of 2:1:1. The anaerobic digestion of wastewater sludge with inoculum produced approximately 1.1 times cumulative volume of biogas yield with regards to the non-inoculated sludge. The initial volume of biogas obtained on the first day in the inoculated sample was increased approximately 39% over the value obtained from the non-inoculated one.

(Keywords: wastewater, sewage sludge, inoculum, biogas, anaerobic digestion)

INTRODUCTION

The technology and yield of biogas depends on the composition and biodegradability of the organic feedstock, microbial growth, pH, and temperature conditions [1]. Anaerobic digestion of municipal wastewater sludge has been widely practiced since the early 1900s and is the most widely used sludge treatment method.

Overall, the process converts about 40% to 60% of the organic solids to methane (CH₄) and carbon dioxide (CO₂) [2]. The chemical composition of the gas is 60-65% methane, 30-35% carbon dioxide, plus small quantities of H₂, N₂, H₂S and H₂O. Of these, methane is the most valuable because it is a hydrocarbon fuel (giving 36.5 MJ/m³ in combustion).

The residual organic matter is chemically stable, nearly odorless, and contains significantly reduced levels of pathogens. The suspended solids are also more easily separated from water relative to the incoming sludge or aerobically treated sludge (such as in outdoor ponds).

While, anaerobic digestion is a proven, effective and highly efficient treatment system, upsets in performance remain common. A correctly run digester will efficiently convert up to 95% of organic material into a low-odor stabilized slurry and produce a renewable resource in the form of biogas that can be flared or used on site. An increase and an optimization of anaerobic digestion applications is required to enhance wastewater treatment sustainability [3]. Indeed, the anaerobic digestion process can be applied for the treatment of various types of wastewaters in a more sustainable way than alternative processes.

Anaerobic digestion (AD) has become an increasingly important industrial process (4). Applications include the treatment of municipal, industrial, agricultural and farming wastewaters. Furthermore, anaerobic digestion is applied for stabilization, quantity reduction, hygienization, and reuse of sludge that originates from conventional wastewater treatment systems (e.g., activated sludge).

On one hand, the anaerobic digestion process has general advantages such as (5):

- The production of biogas that can be used as a green source of energy (e.g. for power generation).
- It can accommodate high COD loads.
- It can be applied for low strength wastewaters provided that the proper reactor configuration is chosen.
- It adapts to remove and/or work in the presence of various toxicant components, provided that adaptation time is allowed for the anaerobic biomass.

On the other hand, the incorrect design of an anaerobic plant for certain applications and/or the inefficient operation of the plant will cause the following disadvantages to become more evident (5):

- Anaerobic biomass growth is slow and the optimum growth is achieved at high temperatures.
- The optimum pH for the process lies in a narrow range near neutrality and the process intermediates make the pH drop below the optimum range.
- The process is sensitive to COD overloads and toxicant shock loads.
- The process effluent is high in COD and nutrients compared to consents stated by legislation.
- The process is complex and difficult to operate compared to other conventional processes.

For certain applications, optimization aims at maximizing the anaerobic process advantages and minimizing or eliminating its disadvantages. This can be achieved by understanding the anaerobic process dynamics and accordingly considering the proper approach for each specific application. The solution can be to take certain actions to upgrade the design and to improve the operation of the anaerobic plant. Also, the solution can be to integrate the anaerobic process with other processes. To understand the process, tools are needed to observe and analyze while certain tools are also needed to study the integration.

Substrate Composition, Hydrolysis, and Degradability in Wastewater

Carbohydrates: Most of the carbohydrates are anaerobically biodegradable [6]. The common polysaccharides and the sugar monomers are easily degraded by anaerobic digestion. Cellulose biodegradation is not easy and less rapid [7]. Still, the rate is sufficient, and the biodegradation of pure cellulose is not a significant rate limiting. However, the cellulose of plants is never pure. Natural cellulose biodegradation is slower due to the protective effect of lignin [8]. Anaerobic digestion is, however, useful for treatment of fruit and vegetable wastewater that contains lingo-cellulose in two step reactors [9]. If the quantity of particulate COD is high, quantification of lignocellulose might be needed. The particulate degradability can be assessed by comparing the acidified COD (i.e. the COD of VFAs in the reactor) with COD_t and COD_s in the influent.

Proteins and Amino Acids: Proteins are often easily hydrolyzed into amino acids; however, they are sometimes coagulated to insoluble forms when exposed to either heat, acids or tannins. Most types of proteins are hydrolyzed and degraded anaerobically [10]. In an anaerobic reactor, protein COD is converted to methane and protein organic-N to NH₄⁺-N. Therefore, the potential of ammonia toxicity needs to be assessed. Generally, the degradation of proteins is not rate limiting. In advanced reactor configurations, (e.g., in an ABR), the reactor performance remains the same during gradual substitution of carbohydrate by protein [11].

Fats and Long Chain Fatty Acids: Fats are polymers of long chain fatty acids (LCFA) linked to a glycerol molecule with ester bonds. The hydrolysis of fats by extracellular lipase enzymes is generally rapid if the fat is soluble. The fats are more soluble if the pH-value is high (pH 8) compared to the pH of acidifying reactors (5.5 - 6.0) where the fat is mostly insoluble and the hydrolysis is slow. Alkaline pre-treatment enhances the hydrolysis and increases the anaerobic digestion rates [12]. Pretreatment of fats with pancreatic lipase enzyme achieves better reductions of fat particulates compared to alkaline pre-treatment only with NaOH [13]. Therefore, both the higher pH and the presence of hydrolytic enzymes need to be considered for efficient hydrolysis and anaerobic degradation of fats. Hydrolysis of fats produces LCFA. The

anaerobic degradation of the LCFA monomers of fat is actually not by fermentation but more similar to the anaerobic oxidation that is done by acetogenic bacteria. The most important products of the anaerobic oxidation of long chain fatty acids (LCFA) are acetate and hydrogen gas (67% and 33% respectively). LCFAs have inhibitory effects on acetogens, aceticlastic methanogens and hydrogenotrophic methanogens (14).

Phenolic Compounds: The phenolic compounds present in wastewater are usually derived from the lignin and tannin of plants. Lignin is apolar and is usually only soluble in alkaline conditions. However, some low molecular weight forms are soluble [15]. The tannins are water soluble compounds. Tannins have ester inter-monomeric bonds that are easily hydrolyzed biologically and acidified during anaerobic digestion. Lignin is generally less degradable than tannins.

Volatile Fatty Acids (VFA): VFAs are easily biodegradable substrates. In the anaerobic process they are the intermediate products between the main processes: acidogenesis, acetogenesis and methanogenesis. Some wastewaters are high in VFA concentration, especially those originating from fermentation processes (e.g. vinasses). VFAs have pKa values between 4.7 and 4.9 and when produced they will cause a drop in the pH. Therefore, high VFA levels in wastewaters are favorable to anaerobic digestion provided that the pH is controlled to the optimum level (around 7). The type of VFA produced by anaerobic bacteria depends on the substrate type [16]. The acidification of LCFA by anaerobic bacteria leads to VFA in the form of acetate only. The acidification of monosaccharides by anaerobic bacteria on the other hand produces acetate C₂, propionate C₃ and butyrate C₄. Thermodynamically, the production of C₂ by anaerobic acidifying bacteria is more favorable than respectively C₃ and C₄. Thus, for acidified wastewaters that mainly contain carbohydrates, it is expected that acetate will be the highest proportion of VFA. Under stress conditions to the process, propionate and butyrate concentrations are expected to increase. For example, at high hydrogen concentration more propionate is produced [17]. Wastewaters with high protein (amino acids) content mainly yield acetate, butyrate, valerate (C₅) and propionate when acidified. The type of VFA produced depends on the type of amino acids degraded (18).

Precipitation

Precipitation can be implemented as mechanism for partial COD removal with suspended solids in a pre-treatment step. However, it has an adverse effect on high rate reactors (e.g. Upflow Anaerobic Sludge Bed (UASB) and Fixed Bed Reactors (FBR)). The entrapment of the solids in the high rate reactors may cause dilution of the methanogen population while the contact time with the wastewater is relatively short. Some components precipitate due to pH changes and addition of calcium:

- Lignin precipitates by lowering pH below 9 or adding calcium.
- Fat precipitates by lowering pH below 8 or adding calcium
- Some proteins coagulate by lowering pH below 6
- Humic acids coagulate by lowering pH below 5 or adding calcium
- Pectin coagulates by adding calcium
- Protein plus tannins form protein-tannin aggregates

Toxicity in Wastewater - Inorganic Toxins

Ammonium: Ammonium nitrogen is present in wastewaters that originally contain high concentrations of proteins or amino acids. Organic nitrogen is also mineralized to ammonium during anaerobic digestion. The toxicity of ammonium is due to the unionized form (free NH₃) [19]. The fraction of free NH₃ is low at a pH value of 7 (about 1% of the (NH₄⁺ NH₃) content) but it is about 10 times higher at pH 8. However, ammonia can be stripped by raising the pH to such alkaline level in a prehydrolysis/acidification step [20]. Anaerobic digestion is found to be still feasible at extreme ammonia concentrations (21).

Sulphur: Wastewater may contain inorganic forms of sulphur like sulphate (SO₄²⁻) and sulphite (SO₃²⁻). During the anaerobic digestion, these compounds are microbiologically reduced to hydrogen sulphide (H₂S). The toxicity of H₂S is again due to the unionized form (free H₂S). The concentration of free H₂S which causes 50% inhibition of methanogenic activity in granular sludge is approximately 250 mg S L⁻¹ (22). For adapted sludge, this 50% inhibition concentration increases to 1000 mg S L⁻¹ [23]. Sulphate is relatively non-toxic. Therefore, the biological

reduction of SO_4^{2-} to H_2S during the anaerobic digestion increases the toxicity of sulphur. In contrast, sulphite, SO_3^{2-} , is more toxic than H_2S . Its biological reduction is very desirable because it will decrease the toxicity of sulphur.

Salt: Salt can cause toxicity problems if the concentration is very high. High concentrations of salts are sometimes present in the wastewaters of industrial processes. Salt toxicity on micro-organisms arises mainly from cations [24]. For acidified wastewater (e.g., from fermentation processes, high concentrations of salt are sometimes needed to neutralize the high concentrations of VFA). In these cases, the possibility of salt toxicity should be considered. The methanogenic toxicity of various kinds of salts is listed in Table 1. The results of this table indicate that monovalent cations are less toxic than divalent cations like calcium (Ca^{2+}). However, the low solubility of Ca^{2+} in the presence of bicarbonate at mild alkaline conditions may result in a low effective concentration in the reactor.

Table 1: The 50% Inhibitory Concentration of Salts to the Methanogenic Activity of Digested Domestic Sludge pH= 7.0, T= 35°C.

Salt	50% inhibitory concentration (mg L ⁻¹)
Mg ²⁺	1930
Ca ²⁺	4700
K ⁺	6100
Na ⁺	7600

Heavy Metals: Heavy metals are sometimes present in wastewaters. Occasionally, it is necessary to add heavy metals as nutrients to wastewater for anaerobic treatment. However, care should be taken to avoid an overdose which can have a toxic effect. The toxicity of heavy metals depends on the soluble concentration. The soluble concentration, of heavy metals would decrease as a result of precipitation reactions with carbonate (CO_3^{2-}) and sulphide (S^{2-}) which are generally present in anaerobic digesters. The precipitation of heavy metals is more effective at increasing pH due to the pH dependency of CO_3^{2-} and S^{2-} .

Acidogens are found to be more sensitive to heavy metals than methanogens in granular sludge [26]. This could be related to the structure of sludge granules in which acidogens at the outside are more exposed to heavy metals. Lin

and Chen (1999) [27] found that the 50% inhibition to methanogens depends on the type of VFA, the type of heavy metal and the HRT.

MATERIALS AND METHOD

Experimental Procedure

The wastewater sewage sludge was weighed in a weighing balance to ascertain the weight. An appropriate weight of water was also measured and poured into a mixing reactor for appropriate mixing before charging it into the 500-gallon tank. The waste water sewage sludge was mix with water in the ratio of 4:1. This ratio was chosen because of high volume of water in the wastewater sewage sludge. The digested cow dung inoculum was also measure and the weight was recorded. The wastewater sewage sludge, inoculum and water were mixed in the mixing chamber at a ratio of 2:1:1. After that, the two were charged in the two different biogas digester (tank) for anaerobic digestion effect.

Energy Content Determination

AOAC (1975) method was used. This was done with bomb calorimeter (model XRY-1A, make: Shanghai Changji, China). It involves igniting the waste sample in oxygen bomb calorimeter (under a high pressure of oxygen gas). The heat energy that was released was absorbed by the surrounding water inside the bomb calorimeter. This gave rise to a temperature increase of the surrounding water and this was used to estimate the energy value of the sample. 1g of the sample was pelleted and turned in the oxygen bomb calorimeter. The heat of combustion was calculated as the gross energy.

$$\text{Energy content} = \frac{E\Delta T - 2.3L - V}{g} \text{ (KJ/Kg)}$$

Where E = energy equivalent of the calorimeter
 ΔT = temperature rise
 L = length of burnt wire
 V = titration volume
 g = weight of sample

Determination of Total Solids [29]

Total solid is made up of the digestible and non-digestible material in the waste. Meynell (1982) method was used. 3g of the raw waste was dried in an oven at 105°C for 5 hours. The dried sample was cooled in a desiccator and then weighed. The weight obtained after all moisture loss is the total solid.

$$\% \text{ T.S} = \frac{B - C}{g} \times \frac{100}{1}$$

T.S = Total solid

B = Weight of crucible + dry residue

C = Weight of crucible

g = Original weight of sample.

Determination of Volatile Solids [29]

The volatile solid is the true organic matter available for bacterial action during digestion. The method of Meynell (1982) was used. The solid residue from the total solid determination was heated in a muffle furnace at 600°C for 2 hours. The heated residue was cooled in a desiccator and weighed.

$$\text{Volatile solid (VS)} = \frac{B - C}{g} \times \frac{100}{1}$$

B = Weight of dried residue from total solid determination

C = Weight of residue after further heating at 600°C

g = Original weight of sample.

Nitrogen/Crude Protein Determination [28]

The micro-Kjedahl method as described in Pearson (1976) was used. This method involves the estimation of the total nitrogen in the waste and the conversion of the nitrogen to protein with the assumption that all the protein in the waste is present as nitrogen. Using a conversion factor of 6.25, the actual percentage of protein in the waste was calculated:

$$\% \text{ crude protein} = \% \text{ Nitrogen} \times 6.25.$$

Digestion

Apparatus Used: Micro-Kjedahl digestion flask (500ml capacity) (Make: Barloworld U.K, model Fk 500/3l) Ohaus weighing balance (0.001g accuracy, model AR3130, Made in England).

Reagents Used: Catalyst mixture (Mixture of 20g potassium sulphate, 1g copper sulphate and 0.1g selenium powder), concentrated tetraoxosulphate (VI) acid.

Procedure: 1g of the ground waste sample was weighed into the Kjeldahl digestion flask. 1g of the catalyst mixture was weighed and added into the flask. 15 ml of conc. H₂SO₄ was also added. Heating was carried out cautiously on a digestion rack in a fume cupboard until a greenish clear solution appeared. The digest was allowed to clear for about 30 minutes. It was further heated for more 30 minutes and allowed to cool. 10 ml of distilled water was added to avoid caking. Then the digest was transferred with several washings into a 100 ml volumetric flask and made up to the mark with distilled water.

Distillation

Apparatus Used: Micro Kjeldahl distillation unit (make: Barloworld, UK model 734205) 100 ml conical flask. (Receiver flask)

Reagents Used: 40% NaOH, Boric acid indicator solution

Procedure: A 10ml aliquot was collected from the digest and put in the flask. A 100ml receiver flask containing 5ml boric acid indicator solution was placed under the condenser of the distillation apparatus so that the tip was 2cm inside the indicator. 10ml of 40% NaOH solution was added to the digested sample through a funnel stop cork. The distillation commenced by closing the steam jet arm of the distillation apparatus. The distillate was collected in the receiver flask (35ml).

Titration

Titration was carried out with 0.01M standard HCl to first pink color.

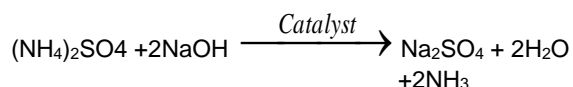
% Nitrogen=

$$\frac{\text{Titration vol.} \times 0.014 \times M \times 100 \times 100}{\text{wt. of sample} \quad 10}$$

Where M= molarity of std HCl

% crude protein = % N x 6.25

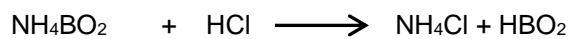
Equation of the Reaction



The ammonia generated was collected in excess boric acid.



After complete ammonia distillation, the ammonium borate solution is titrated with a standard HCl solution. Strong acid (HCl) displaces weak boric acid from its salt.



1 mole of ammonia is equivalent to 1 mole of ammonium borate which is equivalent to 1 mole of HCl.

Knowing the amount of 0.01 M HCl used for the titration, the amount of ammonia bound to borate can be calculated. From this amount, the quantity of nitrogen in the sample can be calculated.

Crude Fiber Content Determination [28]

This determination is done to have an idea of the materials that are indigestible in the waste. It is largely made up of cellulose and small lignin. Crude fiber is obtained as an organic residue left behind after the raw waste has been subjected to standard condition with organic solvents, dilute mineral acids and sodium hydroxide.

The AOAC (1990) method was used. 1g of the sample was weighed (w_1) into a 600ml beaker and 150ml of preheated 0.128M H_2SO_4 was added to it. This was heated for 30 minutes and filtered under suction and washed with hot distilled water until the washings were no longer acidic. The residue was then transferred to a beaker and

boiled for 30 minutes with 150ml of preheated KOH (0.223M). It was filtered and washed with hot water until the washings are no longer alkaline. The residue was washed three times with acetone and dried in an oven at 105°C for 2 hours. It was then cooled in a desiccator, weighed (W_2) and ashed in a muffle furnace (make: Vecstar, model LF3, made in U.K) at 500°C for 4 hours. The ash obtained was cooled in a desiccator and weighed (W_3).

$$\% \text{ Crude fiber} = \frac{W_2 - W_3}{W_1} \times \frac{100}{1}$$

Where:

W_1 = weight of sample

W_2 = Weight of dry residue

W_3 =Weight of ash.

Ash Content Determination [28]

The residue remaining after all the moisture have been removed and the fats, proteins, carbohydrates, vitamins and organic acids burnt away by ignition at about 600°C is called ash. It is usually taken as a measure of the mineral content of the raw waste.

Using AOAC (1990) method, 1g of the finely ground samples were weighed into porcelain crucibles which have been washed, dried in an oven at 100°C, cooled in a desiccator and weighed. They were then placed inside a muffle furnace and heated at 600°C for 4 hours. After this, they were removed and cooled in a desiccator and then weighed.

$$\% \text{ Ash} = \frac{A - B}{C} \times \frac{100}{1}$$

A = Weight of crucible + ash

B = Weight of crucible

C = Weight of original sample

Fat Content Determination [28]

Pearson (1976) method was used. This involves the use of Soxhlet extraction apparatus. This method involves continuous extraction of waste with organic solvent such as petroleum ether for 4 hours or so depending on the volume of sample. To carry out the extraction, the flask was washed and dried in an oven. It was then cooled

in a desiccator and weighed. 1g of the ground waste sample was accurately weighed and transferred into a rolled filter paper and then placed inside the extraction thimble. The thimble was placed inside the extractor. Some quantity of petroleum ether was poured inside the extraction flask (usually three-quarter of the volume of flask). The condenser and the flask were connected to the extractor. The whole unit was placed on a heating mantle for 4 hours after which the petroleum ether was recovered. The oil collected in the flask was dried in an oven at 105°C. It was then weighed, and the percentage fat calculated as shown below:

$$\% \text{ fat} = \frac{C - A}{B} \times \frac{100}{1}$$

C = weight of flask + oil
 A = weight of empty flask
 B = weight of original sample.

Determination of Carbohydrate Content

This was determined by difference which was done by subtracting the sum of % ash, % protein, % fat, % moisture and % crude fibre from 100.

Carbon Content Determination [29]

Walkey-Black (1934) method was used. 0.05g of the finely ground sample was weighed into a 500ml conical flask. 10ml of 1M potassium dichromate was poured inside the flask and the mixture was swirled. 20ml of conc. H₂SO₄ was added and the flask was swirled again for 1 minute in a fume cupboard. The mixture was allowed to cool for 30 minutes after which 200ml of distilled water; 1g NaF and 1ml of diphenylamine indicator were added. The mixture was shaken and titrated with ferrous ammonium sulphate. The blank was also treated in the same way.

$$\% \text{ carbon} = \frac{B - T \times M \times 1.33 \times 0.003 \times 100}{g}$$

Where B = Titration volume (Blank)
 T = Titration volume (Sample)
 M = Molarity of Fe solution
 g = Weight of sample

RESULTS AND DISCUSSION

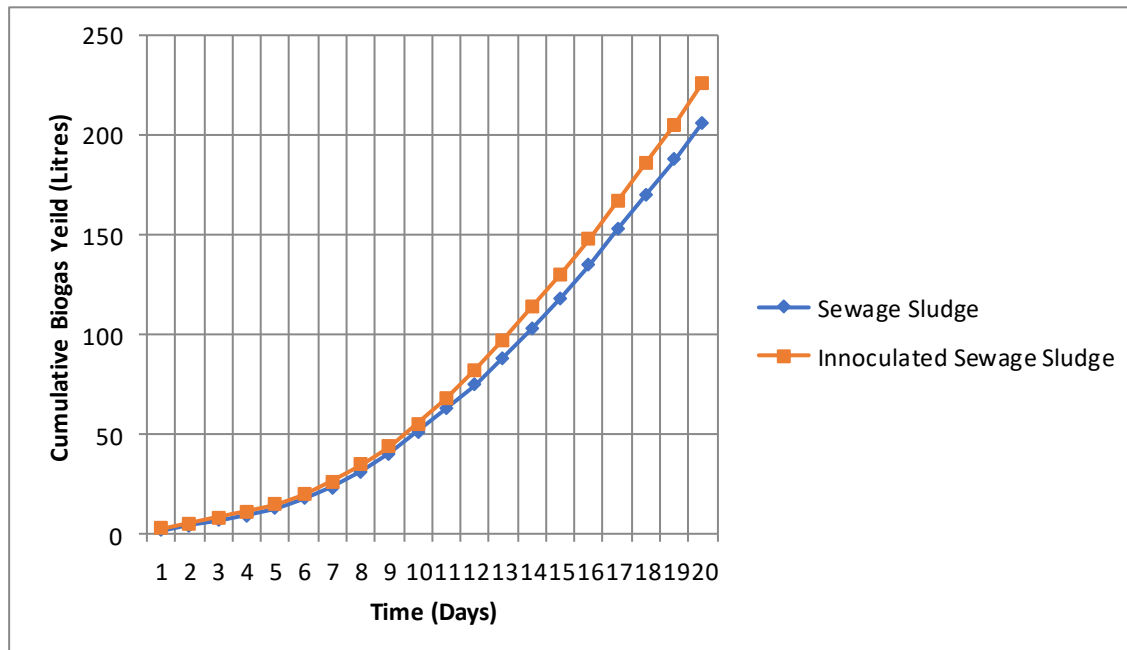


Figure 1: Cumulative Biogas Yield versus Time (Days).

Table 2: Cumulative Biogas Yield for a Test Period of 20 days.

S/N	Sewage sludge	Sewage Sludge with Inoculum
1	1.8	2.5
2	3.9	5.2
3	6.4	8
4	9.1	11.1
5	12.6	14.8
6	17.5	19.7
7	23.3	26.2
8	31	34.4
9	40.3	43.7
10	51.1	55
11	62.6	67.9
12	74.7	82.1
13	88.1	97.5
14	102.6	113.6
15	118.1	130.4
16	134.8	147.9
17	152.8	166.7
18	169.8	185.8
19	188.1	205.6
20	207.5	229.4

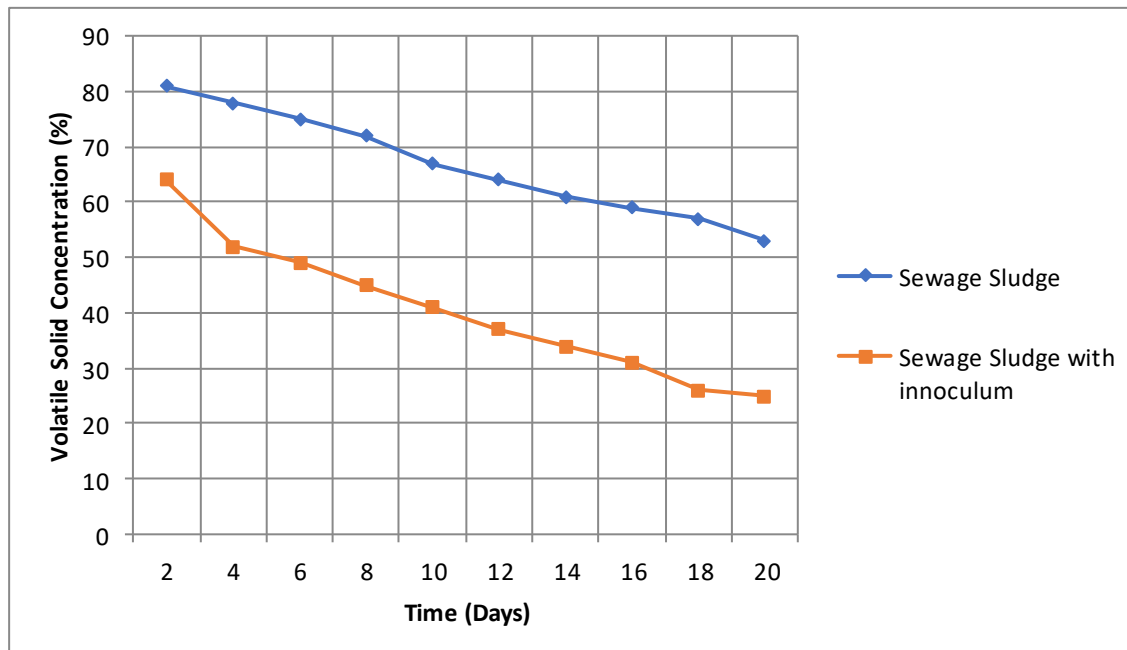


Figure 2: Volatile Solid Concentration versus Time

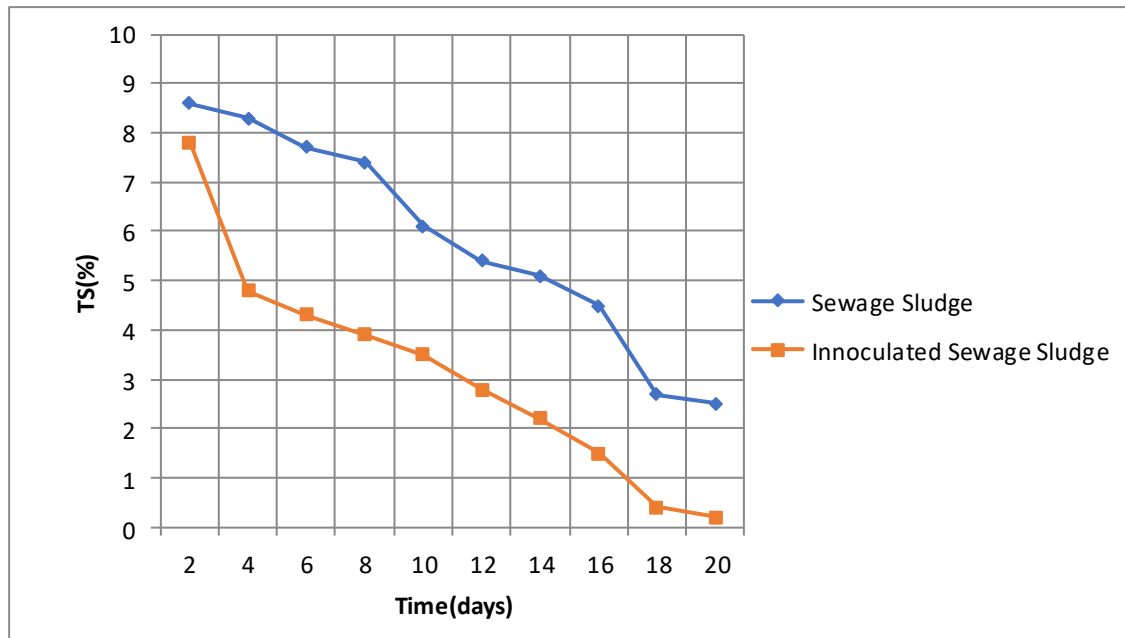


Figure 3: Total Solid Concentration versus Time.

The cumulative biogas yield for the two experiments increased progressively from the first day of charging the digester to day 20 of the test periods. The initial volume of biogas recorded for the sewage sludge was 1.8 litres while the initial volume recorded for the inoculated sewage sludge was 2.5 litres. This value is approximately increased 39% in the value obtained with reference to the non-inoculated one. The more volume recorded for the inoculated sample was due the presence of inoculum which acted as enzyme, speeding up the reaction rate. The feedstock used for the inoculation was digested cow dung which contained a lot of anaerobic bacterial otherwise known as fermentation bacterial. At day 4 the cumulative volume of the sewage sludge was 9.1 litres and the inoculated sludge was 11.1 litres. This was increased 21.9% in cumulative biogas yield showing the positive effect of inoculum in anaerobic digestion of feedstock. Also, at day 10 there was significant increase of 7.7% cumulative yield of biogas for inoculated sample.

Day 14 showed appreciable percentage increased in biogas yield of the inoculated sewage sludge to the non-inoculated one. The value is about 10.7%. The percentage trend of cumulative increased in biogas yield decreased from the initial day to the

last day. It was also observed that the maximum cumulative biogas yield of the sewage sludge with inoculum is about 1.1 times the non-inoculated sample. The volatile solid concentration decreased from 81% to 53% for the sewage sludge without inoculums.

The inoculated wastewater sewage sludge decreased of the volatile solid concentration was from 64% to 29% respectively. The noticeable decreased in the both samples was due to degradation of lignocelluloses content of the sample. Also, it was as a result of degradation of other material contain in the feedstock and as well action of fermentation bacteria. The total solid concentration decreased from 8.2% to 2.3% in sewage sludge and 7.8% to 0.2% in the inoculated tank.

CONCLUSION

Wastewater from different places such as abattoirs, sewage, and some industrial plants are causing great problems to society. These wastewaters are unhealthy to humans, because of the pathogens and the odours emanating from wastewaters. It is also dangerous to some aquatic habitats and if not appropriately taken

care of, may lead to epidemics. Anaerobic digestion is a method of treating this waste and as well obtaining a clean source of energy known as biogas. This work treated anaerobic digestion of a wastewater sewage sludge. The maximum litres of biogas obtained from the non- inoculated tank were 207.5 litres. The charged tank with digested cow dung Inoculum gave 229.4 litres as maximum biogas yield. The volatile solid concentration decreased from 81% to 53% for the sewage sludge. The inoculated wastewater sewage sludge decreased of the volatile solid concentration was from 64% to 29%, respectively.

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