



# Purification of bio-slurry waste using an electrolytic biomass solar cell with Co-generation of Bio-hydrogen Gas

Abdallah Marjan<sup>1</sup>, Aloys Osano<sup>1</sup> and Bakari Chaka<sup>2\*</sup>

<sup>1</sup>The Centre for Innovation, New and Renewable Energy (CINRE) Department, Maasai Mara University, P.O Box 861-20500, Narok, Kenya

<sup>2</sup>Department of Mathematics and Physical Sciences (MPS), Maasai Mara University, P.O Box 861-20500, Narok, Kenya  
bakarichaka@yahoo.com

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## Abstract

Bio-slurry disposal in areas lacking farmyards, where they can be used as organic manure is a challenge. On contrary, there is a dire need for greener processes to increase clean potable water and bio-fuels. This study purposed to electrolyze bio-slurry for its purification while producing bio-hydrogen gas. An electrolytic biomass solar cell (EBSC) of capacity 4,000ml and current density 3.458amps/cm<sup>2</sup> was used. Carbon and steel wool were used as the anode and cathode respectively. The levels of physico-chemicals and bacteria inhibition in the bio-slurry were monitored over a 24-day retention period. Functional groups in the bio-slurry were observed before and after electrolysis while the volumes of bio-hydrogen gas were also monitored. Solid matters in the bio-slurry were effectively reduced by 32.15% while turbidity decreased from 18.92 to 6.85 NTU. The pH value decreased from 8.5 to 6.5 with the electrolysis process. Removal of *E. coli* bacteria was significantly higher than that of *S. aureus* ( $P > 0.05$ ). There were observable changes in the functional groups of the raw and electrolyzed samples, corresponding to the changes in compounds and pH decline. The highest volume of bio-hydrogen recorded was 450.0ml. Bio-hydrogen gas production was largely affected by the temperature and decreased over the retention period.

**Keywords:** Bio-slurry, clean water, bio-hydrogen, electrolysis.

## Introduction

Bio-slurry is the digestate that is left after anaerobic degradation of organic matter into biofuels<sup>1</sup>; most notably biogas. This effluent is quite watery and has little potential for biogas production due to degradation of most carbon matter to biogas. The effluent poses a challenge to most operators of biogas systems. Some operators use bio-slurry as green manure. However, biogas systems located far away from farms and with poor infrastructure have challenges in disposing bio-slurry. The treatment options for this sludge are largely dependent on its bio-chemical composition and anaerobic digester (AD) parameters<sup>2</sup>. Electrolysis of bio-slurry, is a potential method of cleaning this 'waste' by reducing total solids and microbes while generating other biofuels such as bio-hydrogen.

Some of the crucial anaerobic digester parameters that define biomass and bio-slurry include; its pH, alkalinity index, total solids including total dissolved solids (TDS) and total suspended solids (TSS), volatile solids and organic load. Turbidity of bio-slurry is an indicator of high TSS values<sup>3</sup>. The pH of bio-slurry ranges between 7.0-8.0, slightly higher than that of raw biomass. This is due to lack of organic acids in the biomass which are degraded to biogas and carbon dioxide gas. This pH range is quite good as a manure, as it also aids to neutralize excessive soil acidity. Bio-slurry therefore have a higher alkalinity value compared to raw biomass. Whereas the total solids in the bio-slurry are expectedly lower, the TDS

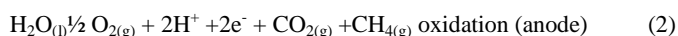
value can be higher than that of the initial biomass. This is due to conversion of most of the volatile matter into biogas and CO<sub>2</sub> gas. The TDS levels is a function of the bio-slurry electrical conductivity, EC, which is a key player in electrolysis of bio-slurry.

Like electrolysis of other wastewater, electrolysis of biomass is an effective method for treating bio-slurry<sup>4</sup>. Products of electrolysis are cleaner (less turbidity and microbial organisms). The process is quite non-spontaneous process, requiring external energy to proceed. Electrolysis of industrial wastewater aims at yielding cleaner water that can be safely discharged into the natural sinks e.g rivers. However, due to variation in chemical composition and AD parameters, electrolysis of bio-slurry targets to clean this biomass while generation gaseous biofuels. From the conventional electrolysis of pure water equations, oxygen and hydrogen gases are produced at anode and cathode respectively. This is illustrated in equations-1, 2 and 3 below;

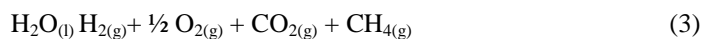
At the cathode, reduction process takes place and hydrogen gas is produced:



Oxidation of the digestate solution takes place:



It is important to note that the process; oxidation and reduction occur concurrently. Together, the overall reaction that we get is;



The amount of methane gas produced from bio-slurry is quite low, and the focus is shifted to bio-hydrogen gas. The gas is equally a very useful bio-fuel. The process of obtaining pure hydrogen gas from anaerobic reactors is rigorous and requires several chains<sup>5</sup>. Other bacteria strains can inhibit bio-hydrogen formation by hydrogenotrophic methanogens<sup>6</sup>. pH is the governing factor which dictates the best ecology for survival of this methanogens<sup>7</sup>. The optimum pH ranges between 6 and 8. Presence of acidogenic bacteria in the substrate or consortia lead to production of acidic products, lowering the pH<sup>2</sup>. This has a negative toll on the hydrogenotrophic methanogens.

Accumulation of waste bio-slurry is a potential habitat for pathogens such as bacteria while also offering a perfect breeding site for mosquitoes. This study aimed at purifying 'waste' bio-slurry using an electrolysis while monitoring the volumes of bio-hydrogen gas co-generated. The levels of indicators of impure water (turbidity, total solids and micro-organisms) were monitored over the electrolysis period undertaken. This is a mutual-benefiting scheme which is 100% renewable operating at ambient conditions.

## Materials and methods

**Design of study:** Fresh bio-slurry from a biogas digester in Maasai Mara University, Narok, Kenya (coordinates 1.0918°S, 35.8498°E) was collected and analyzed. The bio-slurry was analyzed for common water quality parameters present in slurry i.e TDS, TSS, turbidity, pH as well as presence of microbes (gram negative and positive bacteria). These parameters and microbes were continually monitored for a duration of 24 days. The functional groups present in the slurry were analyzed before and after electrolysis. The levels of bio-hydrogen gas produced at the cathode as well as the mixture of gases from the anode was also monitored against the time and temperature of production. A 4,000ml electrolytic cell powered by a 10W solar cell was used.

**Assembly of the electrolytic solar cell:** Waste biomass was filled in a 4,000ml clear reactor fabricated from polymethylmethacrylate (PMMA) material (thickness 2.5mm). The anodic and cathodic chamber were encased with two calibrated (800ml) PMMA domes. The domes were calibrated to monitor the gas volumes (in ml) at the electrodes. A carbon electrode (anode- surface area 4.15cm<sup>2</sup>) and steel wool (cathode-surface area 4.15cm<sup>2</sup>) was completely immersed in the waste biomass contained in the plastic container. Both electrodes were connected to a solar panel (Loom Poly-Crystalline Solar panels, 10W, 12V) as the external electromotive force (emf) of the system. Beehives shelves were placed at the bottom of the reactor to support the electrode chambers. The solar panel was placed at a height of 1 meter above the ground inclined at an angle of 40° to maximize the

solar absorption. The electrolysis was allowed to take place with the pH of the electrolyte and the temperature being recorded after intervals of 3 hours. The average temperature of the retention period was 20.5±1.5°C.

**Methods used:** Physico-chemical parameters of the bio-slurry: For the TSS, the mass of 10.000g bio-slurry solution was weighed. The solution was then passed through a pre-weighed Whatman #41 filter paper. The used filter paper was then dried in an oven at 105°C for 1 hour, cooled in a desiccator before reweighing the filter paper and solution again, up to a constant mass. The difference in weight of the filter paper is the TSS value. TDS were obtained by subtracting TSS from the TS values.

For total solids, TS, 10.000g of sample was weighed, M<sub>1</sub> using an Analytical balance and then placed in an oven conditioned at 105 °C for 6 hours before removing, cooling (in a desiccator) and reweighing to a constant mass, M<sub>2</sub>.

$$\% \text{TS} = \frac{M_2}{M_1} \times 100\% \quad (4)$$

$$\% \text{TDS} = \text{TS}\% - \text{TSS}\% \quad (5)$$

pH, turbidity and temperature values were measured in situ using Universal Multiline P4 WTW (Hanna G-114, Germany). Turbidity was recorded in Nephelometer Turbidity Units (NTU).

**IR Functional Groups:** The bio-slurry was gradually concentrated until all the water was dried. Functional groups were elucidated using FT-IR Shimadzu 119. The Attenuated Total Reflection (ATR) method was preferred using KBr discs.

**Analysis of Microbial Activity:** Antimicrobial studies were conducted for both gram-positive (*S. aureus*) and gram-negative (*E. coli*) bacteria.

**Media preparation:** 5% Muller-Hinton's universal agar was used for media preparation. All the distilled water used was sterilized prior to use. The plates onto which the media was to be dispensed together with the inoculation needles and micro-spatulas were all autoclaved (Wisconsin Aluminum Foundry, UL 6P38, 25X-2 autoclave) for thorough sterilization. The media was autoclaved at 250°F and 15 psi pressure for quarter an hour. The media was then cooled to 113°F and dispensed onto the plates which were later allowed to cool further and incubated at 40°F for one day.

**Antimicrobial sensitivity tests:** The plates were streaked with 6.0mm sterile octo-discs impregnated with the bio-slurry samples. The samples and their plates were evenly spread out according to aseptic regulations to avoid cross-contamination. The plates were then incubated for one day at 98.6°F (Omega I-

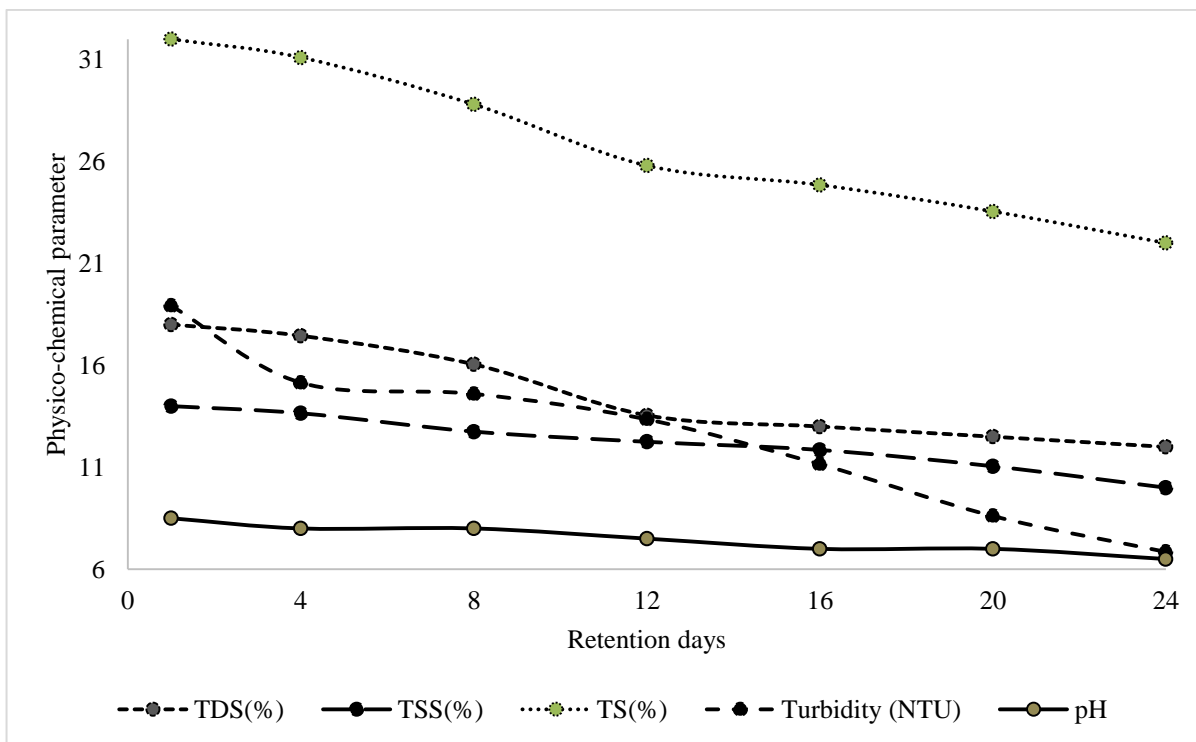
52, PNP 9052 incubator). The plates were then removed and the diameter of the inhibition zones determined.

**Statistical analysis:** 95% level of confidence was used for all the statistical analyses done. Ms Excel (version 2016) and Origin Lab (version 6.5) statistical softwares were used.

## Results and discussion

**Physico-chemical levels of the bio-slurry:** All the parameters analyzed decreased continually with the electrolysis process. The TS values at the onset of the experiment (32.0%) were quite high but within the range of a closed water system (20 to 500mg/L). The bulk of this solids were the TDS due to abundance of inorganic matter in the bio-slurry. The inorganic compounds in the biomass cannot be effectively degraded into methane and carbon dioxide at ambient AD systems<sup>8</sup>. These compounds are thus passed out of AD systems in the bio-slurry and are a key contributor to the TDS values. Some of these compounds (such as bio-metals) and ligands are quite ionic and adversely affected by electrolysis<sup>9</sup> of the bio-slurry. The compounds are thus used up in the process leading to gradual decrease. It is therefore not surprising to observe that the deviation in TDS values were steeper than those of TSS values. The rate of removal of TDS (33%) was higher than that of TSS (28.57%). The rate of reduction of TS in the bio-slurry after electrolysis (31.25%) after 24 days was quite effective with the magnitude of current density employed. Figure-1 summarizes the trends in removal of these parameters in the EBSC.

The rate of removal of TSS (28.57%) was quite lower than that of other industrial effluents such as tea, dairy, tannery or metallurgical effluents<sup>10</sup>. This can be attributed to the amount and nature of organic compounds in bio-slurry, working temperature as well as the current density employed. The turbidity of the bio-slurry was effectively reduced by 36.21% from 18.92 NTU to 6.85 NTU in the same duration. Turbidity is a function of TS<sup>11</sup> and its removal pattern coincided with that of TS. The ultimate turbidity levels were below the recommended WHO levels for unpolluted river water (10 NTU). This is the most observable feature of the bio-slurry as far as clarity of the effluent was concerned. The EBSC can thus be regarded to have successfully cleaned the water from a layman's perspective. The pH values of the system were the most unperturbed gradually decreasing from 8.5 to 6.5. pH is a key factor not only for bio-slurry but also for other wastewater systems. The pH of 8.5 is attributed to loss of organic acids after their conversion to methane and carbon dioxide gas during acetogenesis and acidogenesis processes of biogas formation<sup>12</sup>. The end-product of biogas production (bio-slurry) is thus devoid of these acidic compounds. During electrolysis, there is change in the composition of the bio-slurry as most compounds are either solubilized or used up. The dynamic change in bio-slurry composition solubilize other acidic compounds, therefore decreasing the ultimate pH of the solution. This pH range is still within the recommended pH for potable water systems by WHO (6.5 to 8.0)<sup>13</sup>.



**Figure-1:** Variation in levels of physico-chemicals in the bio-slurry with electrolysis process.

**Reduction in bacteria colonies in the bio-slurry:** The population of both gram positive and negative bacteria in the bio-slurry were effectively reduced with electrolysis of the process. Like methanogenic bacteria (including hydrogenotrophic ones), *S. aureus* bacteria are gram positive in nature<sup>14,15</sup>. The bacteria can also survive in limited amounts of oxygen such as in the EBSC. Therefore, the level of resistance of the *S. aureus* bacteria was more than that of the *E. coli* ones. There was a significant difference in the percentage inhibition of the *S. aureus* compared to the *E. coli* bacteria, especially after the 8<sup>th</sup> retention day ( $P > 0.05$ ). Table-1 illustrates the levels of inhibition of the two bacteria colonies in the EBSC.

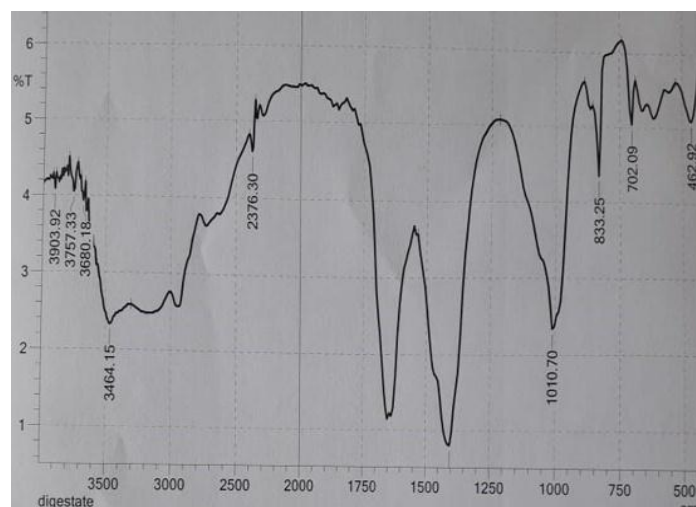
Current density, other physico-chemical parameters and electrolysis time affect the rate of bacteria inhibition by electrolysis or electrocoagulation<sup>16</sup>. In this case, the applied current density (3.458Amps/cm<sup>2</sup>) was quite moderate but was impeded by weather fluctuation. The average temperature of the retention period was 20.5±1.5°C occasioned by clouds especially in the morning hours. The flow of current was therefore quite irregular but continuous for 8 hours every day of the 24 retention days. Fluctuation in current density reduces the contact time of external emf on the bacteria colonies thus reducing the bacteria inhibition. The optimal temperature for electrolysis of wastewater in removal of bacteria has been observed to be 35°C. The optimal pH is between 7-9, with the performance reducing with decreasing pH value. In the above case, performance increased after the 8<sup>th</sup> day, which corresponded to gradual decrease in pH (See the previous section above) but more stable temperature conditions. The performance was more effective between pH 7.0 and 6.5. The electrolysis time for these experiments was quite irregular thus did not have a great impact on the output.

**Variation in functional groups present in the bio-slurry before and after electrolysis:** Change of bio-slurry composition alter the functional groups present in bio-slurry. Some pronounced peaks were effectively sequestered. The peaks of other functional groups became more intensified while others emerged. This is illustrated in Figure-2. Both samples indicated presence of carboxylic acids with OH<sub>RCOOH</sub> (3757.33 and 3749.62cm<sup>-1</sup> respectively), OH<sub>ROH</sub> and C=O<sub>RCOOH</sub> peaks present. The correlation area of the OH<sub>RCOOH</sub> peak increased after electrolysis due to the acidification of the process. This is evident in the pH values which gradually decreased from 8.5 to 6.5. Similar findings were observed in the C=O<sub>RCOOH</sub> peaks. The OH<sub>ROH</sub> stretching free alcohol peak and N-H primary stretching amine in the raw bio-slurry (3680.18 and 3464.15cm<sup>-1</sup> respectively) were quenched by electrolysis implying a decline in the alkanols as the carboxylic acid was increasing. Electrolysis of the bio-slurry solubilized organic acids in the sample, which then neutralized the alkaline alkanols and amines thus reducing their presence.

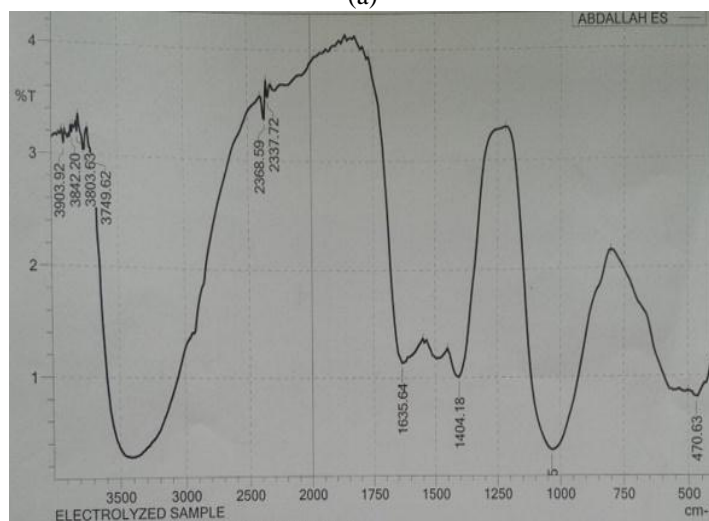
Both samples exhibited presence of thio-groups with stretching sulfate peaks of equal intensity at 1404.18cm<sup>-1</sup> for both samples. Thio-groups are common in several proteins and sulfate ligands<sup>17</sup>. A similar research conducted on sulfate-rich wastewater confirmed persistence of these compounds with electrolysis<sup>18</sup>. The electrolyzed sample also exhibited a strong stretching sulfoxide peak (S=O) near the OH-R peak at 1033.85cm<sup>-1</sup>. Several fingerprint peaks (C-F stretch at 1010.70cm<sup>-1</sup>, C-H 1, 4 disubstituted bending at 833.25cm<sup>-1</sup> and C-H monosubstituted benzylic bend at 702.09cm<sup>-1</sup>) in the raw bio-slurry were sequestered by the electrolysis process. The -C-H stretch at 462.92cm<sup>-1</sup> in the raw bio-slurry was shifted to 470.63cm<sup>-1</sup> in the electrolyzed sample.

**Table-1:** Inhibition of *E. coli* and *S. aureus* bacteria during electrolysis.

Day	Anti-bacteria inhibition level			
	<i>E. coli</i>		<i>S. aureus</i>	
	Octo-disc diameter (mm)	% inhibition	Octo-disc diameter (mm)	% inhibition
1	6.0	0.0	6.0	0.0
4	7.3	21.6	7.5	25.0
8	8.5	41.7	8.0	33.3
12	11.0	83.3	9.0	50.0
16	12.5	108.3	11.0	83.3
20	13.5	125.0	11.8	96.6
24	15.0	150.0	12.5	108.3



(a)

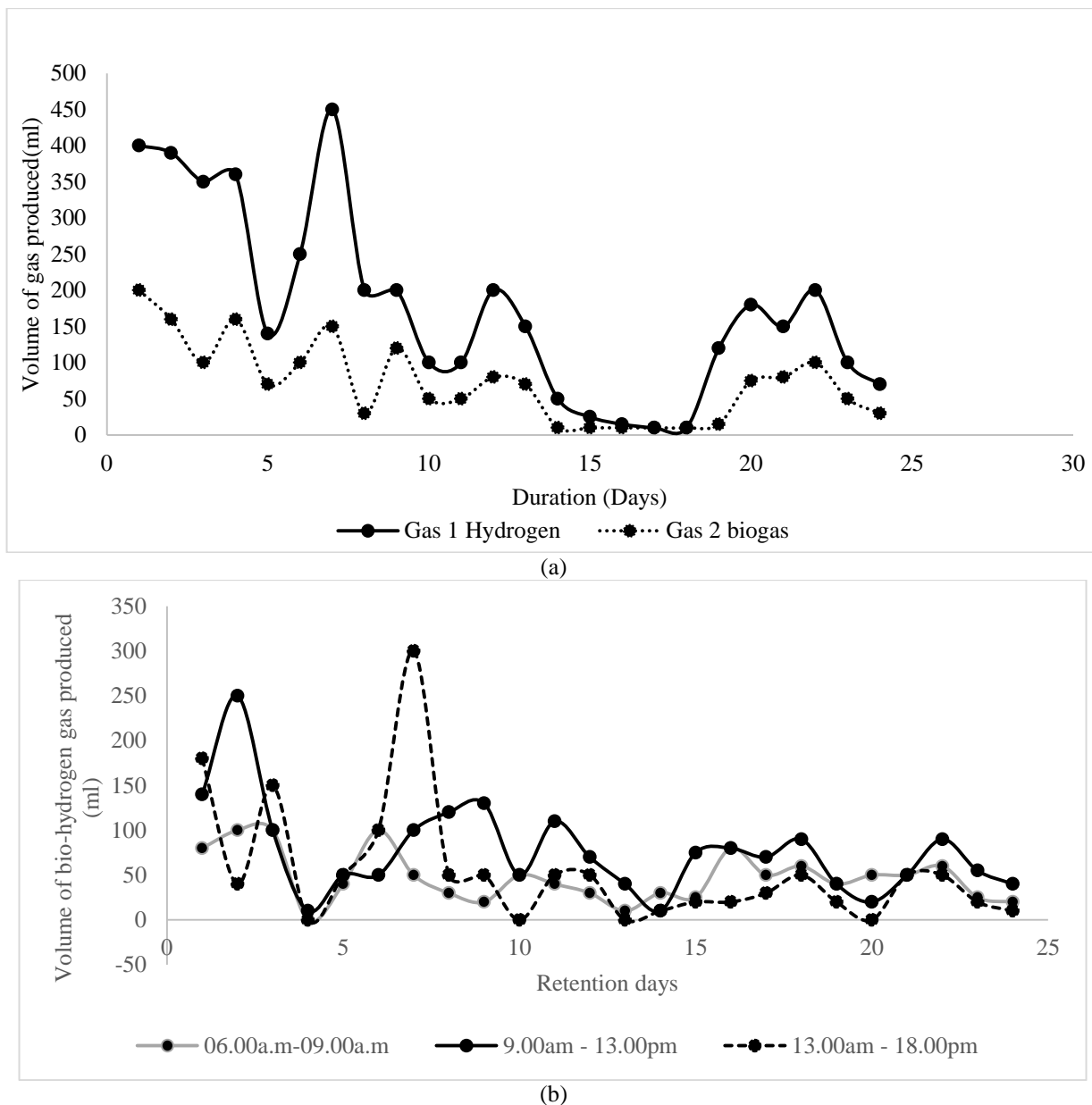


(b)

**Figure-2:** FT-IR spectra of the raw bio-slurry (a) and electrolyzed bio-slurry (b) samples.

**Production of bio-hydrogen and other anodic gases:** The amount of bio-hydrogen gas produced cumulatively during the retention period was significantly higher than the mixture of anodic gases produced (presumably biogas). This is supported by the theoretical molar volumes of hydrogen gas produced in comparison to oxygen gas during electrolysis of water (two folds higher). This is illustrated in equations 1 and 2 above. Experimental conditions such as lack of total homogeneity in the bio-slurry and fluctuations in current densities at the electrodes altered this ratio (bio-hydrogen: anodic gases). The greatest volume of hydrogen gas produced was 450ml (retention day 7) with the least being 0ml (on the 18<sup>th</sup> retention day). The bio-hydrogen yields corresponded to the physico-chemical conditions of the experiment. It was observed that increasing temperature increased the bio-hydrogen gas produced (Figure-3b). Temperature intensity was higher in the afternoons which led to more bio-hydrogen production. Increase in temperature increase the ionization process of ions<sup>19</sup> in the bio-slurry, thus

facilitating easier movement of electrons and thus more products of electrolysis. Temperature also increase the movement of electrons in the conductor from the emf source to the load for faster electrolysis<sup>20</sup>. Bio-hydrogen gas production was quite high at the onset of the experiment due to the effectiveness of the ESBC and abundance of ions in the bio-slurry. With time, the electrodes become poisoned reducing the contact area and therefore the electron density<sup>21</sup> is also abridged. The production patterns were directly influenced by temperature as observed on the 5<sup>th</sup>, 7<sup>th</sup> and 18<sup>th</sup> day (Figure-3a). There was a gradual decrease in gas production until after the 18<sup>th</sup> day, whereby increase in temperature led to the power surge upwards. Electrolysis process interfered with the anaerobic archaea in the system by reducing their population (Table-1). These bacteria, especially hydrogenotrophic methanogenic bacteria are the natural producers of bio-hydrogen<sup>22</sup>. Their reduction with the electrolysis process therefore contributed to the decline in the levels of bio-hydrogen gas over time.



**Figure-3:** The volumes of bio-hydrogen gas produced in the ESBC (a) and at various times of the day (b)

### Conclusion

The solid matter components in the bio-slurry were effectively reduced by 32.15% as the turbidity levels reduced from 18.92 to 6.85NTU within the electrolysis process. The pH of the bio-slurry reduced from 8.5 to 6.5. The pathogenic microbes were also effectively removed with *E. coli* inhibition being more than that of *S. aureus* ( $P > 0.05$ ). There was a change of IR spectra of the raw and electrolyzed bio-slurry samples where the organic acids peaks intensified as other functional groups were quenched with the electrolysis process. The highest bio-hydrogen gas volume recorded was 450.0ml. Gas production was largely influenced by the temperature conditions and decreased over time due to electrode poisoning.

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