






## Effect of Operational Parameters on Anaerobic Digestion of Municipal and Sugar Industry Wastewater



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### ABSTRACT

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Anaerobic digestion (AD) is a versatile process that entails low energy consumption and has low capital costs. Unfortunately, this process is not widely commercialized due to instabilities. The instability in the system is due to variations in the feedstock, operating, and environmental conditions. Therefore, this study aimed to determine the effect of organic loading rate (OLR), temperature, and pH on the AD system. An online pH and temperature monitoring sensor calibration were also studied to adjust AD parameters. The water quality parameters that were monitored were turbidity, chemical oxygen demand (COD), and colour. Wastewater with a low and high organic loading had a 90% and 36% COD reduction respectively after 5 days. Without pH adjustment, the pH of the system was 4.1 to 4.4 and the maximum COD reduction was 56.2%. When the pH was increased to 6.8, the maximum COD reduction was 66.5%. For the unadjusted temperature (room temperature), a maximum COD reduction of 56% was achieved. When the temperature was increased to 40°C, the maximum COD reduction was 66%. The increase in pH and temperature resulted in a 10% increase in COD reduction in the AD system. From the study, online pH, and temperature sensor calibrations errors were found to be 0.5 and 0.05 respectively as compared with the manual analytical technique. One of the limitations of this study was obtaining the apparatus to control temperature and pH at the same time. Future research will involve the automation of the AD system with the determined optimum conditions. This suggests smart monitoring and control sensors of AD operational parameters can repurpose its reliability for commercial activity.

## 1. INTRODUCTION

Wastewater discharged into rivers and dams affect the quality of the water body by introducing contaminants into the water body and this disrupts the natural ecosystem. The streams, in rural and urban areas, are polluted to the point that they cannot return to a pristine condition without the aid of humans [1]. Untreated or poorly treated wastewater that is released into streams gives rise to the growth of bacteria and aquatic plants that reduce the dissolved oxygen in the water. The lack of dissolved oxygen in the water has harmful effects on aquatic life [2]. Current wastewater treatment processes still do not remove all the contaminants from the wastewater and these contaminants enter the ecosystem [3]. Therefore, the wastewater treatment processes need to be improved.

In addition to the wastewater problem, South Africa is also currently facing a clean water and energy crisis [4]. One of the most important resources globally is fresh water. Although 70% of the earth is covered with water, only a small percentage is accessible for human consumption [5]. About 2.1 billion people are deprived of access to fresh water throughout the world. Two of the main reasons for the water crisis are the

rapid increase in human population and the lack of infrastructure to supply fresh water [6]. Pollution and wastewater further reduce the availability of freshwater [7]. In 2017, the Cape Town dam levels dropped to 20% capacity. The city was preparing for day zero, whereby water rationing would have been implemented [8]. Historically, lack of water has led to violent protests and unrest. Therefore, it is imperative to have alternate water sources to ensure the smooth operation of a country [9].

Apart from the water challenges, South Africa is also plagued by power shortages and blackouts. The current power plants, which use coal, a fossil fuel, to generate energy have proven to be unsustainable. Fossil fuels generate vast amounts of waste that have detrimental effects on the environment and contribute to global warming [10, 11]. Biogas is an alternative energy source that can bridge the existing energy gap in the country. Biogas technology can process clean, renewable energy [12].

Anaerobic digestion is an environmentally-friendly method that converts waste material into energy, biogas, as shown in Figure 1. Anaerobic digestion is attractive as a water and wastewater treatment method due to the low energy costs. The

organic matter is broken down by microbes in the absence of oxygen [13]. The four stages of anaerobic digestion: hydrolysis, acidogenesis, acetogenesis, and methanogenesis, take place in the presence of microorganisms. The phases occur sequentially, and the digestion is completed once the production of biogas ceases [14].



Figure 1. Benefits of Anaerobic digestion [15]

Despite the advantages, the process is not widely commercialized due to the instability of the AD process. Instability in the system is caused by changes in feed composition, the accumulation of volatile fatty acids, operating conditions such as pH and temperature, and the total ammonia nitrogen concentration [16, 17]. AD is dependent on the microorganisms and therefore, the conditions to keep them alive and functioning are very crucial [18]. It is crucial to maintain the optimum operating conditions to maintain the microbial community. Some of the key operating parameters that are considered to be monitored include feed composition, biogas composition, pH, and temperature. As the organic loading rate increases, so does the disturbances in the system that lead to instability [19].

It is therefore imperative to design an anaerobic digester that can control the operating conditions that leads to the promotion of the growth of the microorganisms [19]. To do that, the optimum operating conditions need to be determined. Operating at the optimum conditions will stabilize the system. The system can be stabilized by pre-treating the feed, co-digestion, and by ensuring optimum conditions are maintained in the digester. Therefore, this study aimed to determine the effect of OLR, temperature, and pH on an anaerobic digestion system and to calibrate online pH and temperature sensors for its monitoring.

In developing countries, such as South Africa, energy generation is not widely utilized. The optimization of anaerobic digestion systems could help ease the coal-based electricity production process [20].

This study aims to determine the optimum operating conditions for anaerobic digestion in order to automate the system in future work. The process parameters will be varied, in order to determine their effects on the AD system. The parameters that will be varied are organic load, pH and temperature. Online sensors will be installed in the AD system to determine if sensors are compatible with the system. The

optimal AD conditions need to reduce the percentage COD present in the wastewater and produce biogas.

## 2. METHODS AND MATERIALS

### 2.1 Wastewater and inoculum collection

Two sources of wastewater were under investigation. Source 1 (S1) was collected from the influent sample point of a local municipal wastewater treatment plant in KwaZulu-Natal. Source 2 (S2) was collected at the effluent sample point of a local Sugar Refinery in KwaZulu-Natal. The effluent from the sugar industry is the influent to another wastewater treatment plant. The activated sludge that was used as the inoculum was also collected from a local South African wastewater treatment plant in KwaZulu-Natal. The sludge was collected from the anaerobic digester sample point.

### 2.2 Wastewater and activated sludge characterization

Wastewater collected was characterized for the following parameters: COD, pH, temperature, turbidity, and colour.

The parameters were determined using standard methods for the examination of water and wastewater [21]. The water characterization is shown in Table 1. The COD was quantified using COD high-range vials (HACH). For the high range, 0.2 ml of the sample was measured and added to the COD vials. The vials were placed into a digital reactor block (HACH DRB200, Hach Company, CO, USA) at 150°C for 2 hours for digestion. The sample was cooled to 120°C and properly shaken before being allowed to cool to room temperature. A blank was prepared similarly with 0.2 ml of distilled water. At room temperature, the COD was measured using the spectrophotometer (HACH DR3900, Hach Company, Loveland, CO, USA). The pH and temperature were determined using a handheld tester (HACH, Hach Company, Loveland, CO, USA).

Table 1. Wastewater and activated sludge characterization

|                  | Municipal Wastewater (S1) | Sugar Effluent (S2) | Activated Sludge |
|------------------|---------------------------|---------------------|------------------|
| COD (mg/mL)      | 432                       | 1887                | 2841             |
| pH               | 6.91                      | 4.12                | 7.24             |
| Temperature (°C) | 20.9                      | 24.7                | 20.1             |
| Turbidity (NTU)  | 365                       | 762                 | 13200            |
| Colour (PtCo)    | 545                       | 7855                | 72710            |

#### 2.2.1 Effect of organic loading on anaerobic digestion

A 5 L Duran Schott bottle with three-port caps, as shown in Figure 2, was used as an anaerobic digester. The bottle was washed to remove contaminants and then charged with S1 and inoculum in a ratio of 2:3. The volumes of the wastewater and inoculum were 2000 mL and 3000 mL respectively. The pH and temperature were not controlled at this point in the experiment. The system was run for five days, the duration of the trial, and samples were taken daily, and COD analysis was conducted on the sample daily. The same procedure was repeated with S2 and inoculum. Samples were collected from a sample point at the top of the digester.

For all runs, the parameters were determined using standard methods for the examination of water and wastewater [21].

### 2.2.2 Temperature effect on anaerobic digestion

The same set up procedure as in section 2.2.1 is followed: A 5 L Duran Schott bottle with three-port caps, as shown in Figure 2, was used as an Anaerobic digester. The bottle was washed, to ensure zero contamination, and charged with S2 and inoculum in a ratio of 2:3. The volumes of the wastewater and inoculum were 2000 mL and 3000 mL respectively. The trial was conducted for 10 days. The anaerobic digester was operated for 10 days without adjusting the temperature. The temperature of the digester was at room temperature. Samples were taken daily, and the turbidity and COD were analyzed. The trial was run again with the digester bottle placed on a hotplate. The temperature was maintained at 40°C. Samples were taken daily and the study was carried out for 10 days. The pH was not controlled during this trial. Samples were taken from a sample point at the top of the reactor.

### 2.2.3 Effect of pH on anaerobic digestion

S2 and inoculum were charged into a 25 L bioreactor with sensors, as shown in Figure 3 (Biodigester with dissolved oxygen, pH, and temperature sensors with data logger; Glass Chem, Cape Town, South Africa) in a ratio of 2:3. The volumes of the wastewater and inoculum were 10 L and 15 L respectively. The trial was conducted for 10 days. The pH was not corrected, and the initial pH was found to be 4.15. The anaerobic digester was operated for 10 days without adjusting the pH. Samples were taken daily from a sample point at the point of the digester, and the turbidity and COD were analyzed. The trial was run again with the pH maintained at 6.8. Samples were taken daily, and the study was carried out for 10 days.

Acid and base were used as pH correctors. The acid and base were charged into individual bottles connected to the 25 L bioreactor as shown in Figure 2. The acid used was a 1 M solution of hydrochloric acid and the base was a 1 M solution of sodium hydroxide. The 1 M acid solution was prepared by mixing 83 mL of HCl (HCl 37% AR) with 1 L of water. The 1 M base solution was prepared by mixing 20 g of NaOH (NaOH pellets 98.5% AR) with 500 ml of water.

The 25 L bioreactor with sensors (Biodigester with dissolved oxygen, pH, and temperature sensors with data logger; Glass Chem, Cape Town, South Africa) was charged with S2 and inoculum in a ratio of 2:3. A pH set point of 6.8 was inputted into the data logger. The pH sensor obtained the pH value of the substrate in the bioreactor. If the pH value differed from the setup by 0.5 then either acid or base was pumped into the reactor to correct the pH.

### 2.3 pH and temperature sensor calibration

S2 and inoculum were charged into a 25 L bioreactor with sensors, as shown in Figure 3 (Biodigester with dissolved oxygen, pH, and temperature sensors with data logger; Glass Chem, Cape Town, South Africa) in a ratio of 2:3. The pH and temperature readings taken by the online sensor was recorded on the data logger. Samples were taken daily, and the pH and temperature were evaluated using a Hach pH and temperature handheld meter. The study was conducted for 10 days.

The error was calculated according to the following equation:

$$\% \text{ error} = \frac{\text{measured reading} - \text{STD reading}}{\text{True Value}} \times 100 \quad (1)$$

The true value is measured by using a calibrated pH and temperature handheld meter tester (HACH, Hach Company, Loveland, CO, USA).

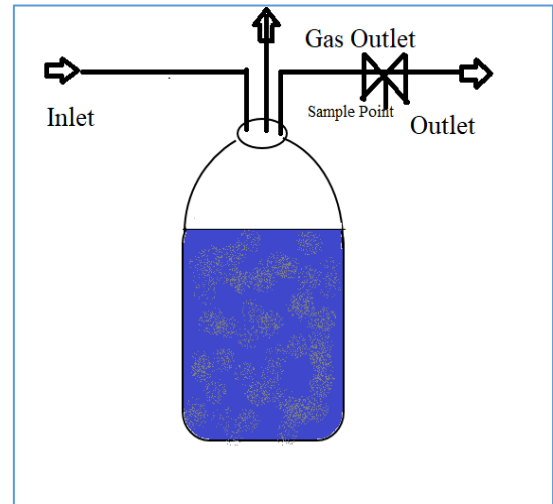


Figure 2. 5 L anaerobic digester

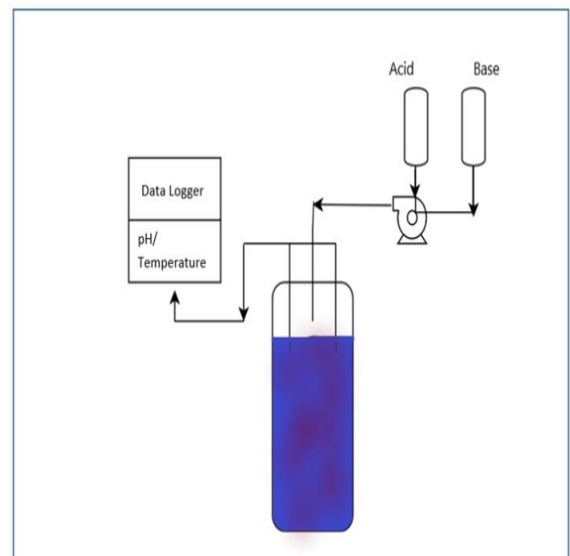


Figure 3. Biodigester set up for sensor calibration

## 3. RESULTS AND DISCUSSION

Monitoring anaerobic digesters are essential for their effective operation. Due to the intricacy of the AD process, which involves different microbial activities (hydrolysis, acidogenesis, acetogenesis, and methanogenesis), many operational parameters are extremely sensitive. To prevent process failure, early detection of process imbalance is important. In this study, the operational impact of the AD process was evaluated by assessing feedstock/substrates conversion or OLR (COD removal) and intermediates accumulation (pH) for wastewater treatment. Even though product generation (biogas production) and microbial diversity are important, they were not considered in this study.

### 3.1 Wastewater and inoculum collection

The two wastewater streams that were collected had a vast difference in organic content. S1 had a COD of 432 mg/L and S2 had a COD of 1887 mg/L.

#### 3.1.1 Effect of organic loading (OLR) on COD removal

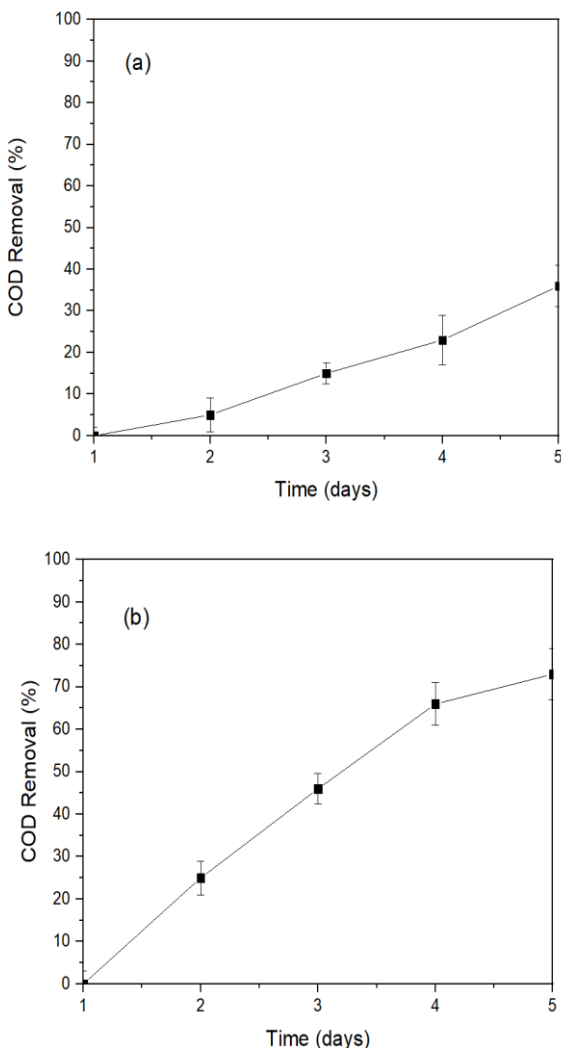
The feedstock type and compositions, which are expressed as a measure of the chemical oxygen demand (COD) of the AD organic load rate (OLR) directly affected the COD removal. In essence, wastewater with high organic fractions degrades with respect to hydraulic retention time (HRT) to produce a high amount of biogas. It is, therefore, important to control the OLR and HRT according to the capacity of the reactor [17]. Moreso, underloading (low OLR) the process becomes uneconomical because the capacity of the reactor is not fully utilized. In contrast, overloading might result in volatile fatty acid accumulation, which becomes more toxic to the methanogens and subsequently system failure. In this study, the initial CODs of S1 and S2 were 432 and 1887 mg COD/L respectively. Therefore, evaluating its impact on the AD process comes in handy.

Figure 4(a) shows that the COD was reduced slowly after a hydraulic retention time of 5 days and the percentage of COD reduction was 36%. This is due to the ratio of microbes present in the sludge vs the organic content in the wastewater. The microbes have to break down more organic content and this increases the hydraulic retention time [21]. Figure 4(b) has low organic loading, after 5 days the COD reduction is 90%. The microbes in the activated sludge are in excess when compared to the organic content present and therefore they break up the organic content quickly. 5 days is insufficient time for the microbes to break down the organic content in S2 and thus negating a major benefit of anaerobic digestion. It is recommended that waste or wastewater with high organic loading is used as feed for anaerobic digestion to obtain biogas. It was found that the highest yield of biogas can be produced via anaerobic digestion between 21 - 25 days [22]. Therefore, from the observation made with the feedwater compositions (S1 and S2), further experiments conducted in this work were continued with S2. The reason has been that a higher retention time (HRT) of 10 days was needed for the other experiments. From Figure 4(a) and 4(b), the HRT was only 5 days, yet at the end of five days, the COD reduction was 90% for S1, it, therefore, indicated that using an HRT of 10 days for S1 was not viable, therefore S2 was chosen to continue the experiments.

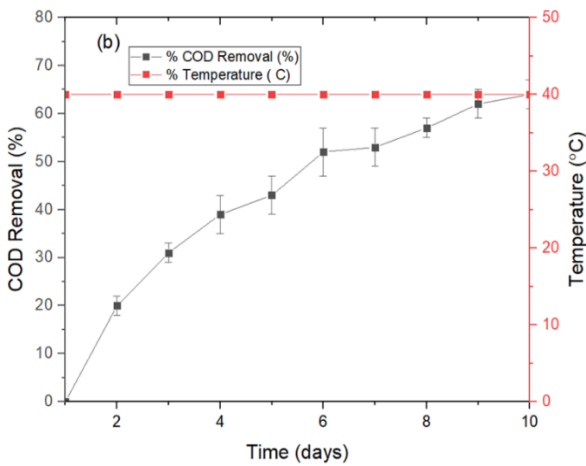
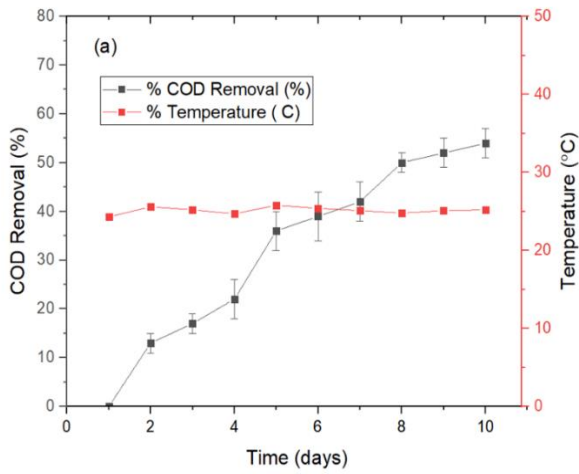
#### 3.1.2 Effect of temperature on the AD performance

Anaerobic digestion is applicable in a wide variety of temperatures, including psychrophilic (20°C), mesophilic (25-40°C), thermophilic (45-60°C), and even hyperthermophilic (>60°C) environments [23, 24]. The temperature has a direct effect on the physio-chemical properties of all components in the digester, as well as the thermodynamic and kinetic activities of the biological entities. Temperature influences the methanogenic favourability of oxidation-reduction and enzymatic reactions. Therefore, monitoring temperature becomes very crucial.

Figure 5 shows the controlled and uncontrolled temperature monitoring for COD removal over an HRT of 10 days. In Figure 5(a), the temperature of the anaerobic digester was not controlled (the experiment was run at room temperature). The system's temperature fluctuated between 24°C and 25°C. Without any influence on the temperature, the temperature range obtained lies on the lower end of mesophilic conditions. The % COD reduction after 10 days was found to be 54%. The second experiment was conducted at a fixed temperature of 40°C, as shown in Figure 5(b). The temperature was selected in the mesophilic phase range because too high temperatures above 60°C, could lead to a decrease in the percentage of COD reduction. Besides that, anaerobic digestion at thermophilic temperatures is also difficult to control, if the temperature of the anaerobic digester changes more than 0.6°C per day then the system is prone to instabilities [23]. At 40°C, the percentage of COD removal was 64% after 10 days. Anaerobic digestion conducted at a higher temperature (40°C) had a greater COD reduction. Higher operating temperatures have proven to be desirable in anaerobic digestion. Although, if the temperature is higher than the optimum operating conditions, then the high temperatures can compromise the stability of the system. A temperature of 40°C is preferred for anaerobic digestion. A jacketed reactor is required in order to control the temperature of the digester.



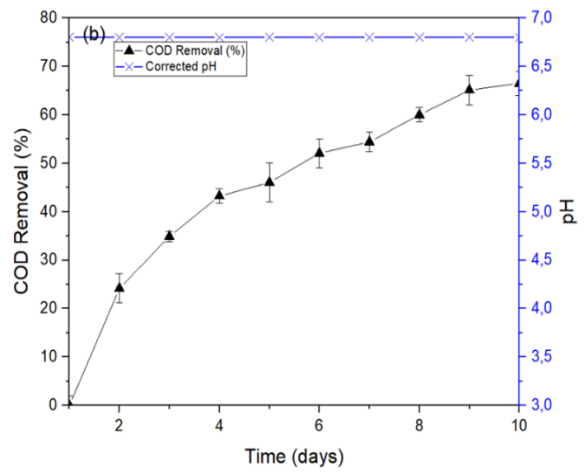
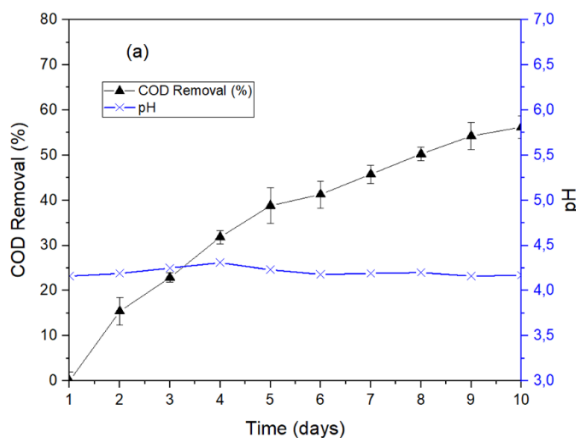
**Figure 4.** (a) Percentage of COD removal with high organic loading (S1) (sugar effluent); (b) Percentage of COD removal with low organic loading (S2) (municipal wastewater)



**Figure 5.** (a) Percentage of COD removal at uncontrolled room temperature; (b) Percentage COD removal with a controlled temperature (40°C)

### 3.1.3 Effect of pH on AD performance

In the AD process, microorganisms' activity depends on ionic strength, since each microbe is active within a certain pH range and ideal pH. For instance, methanogens can function in a narrow pH range from 5.5-8.5, with an optimum range of 6.5-8.0 [17]. This makes monitoring of pH in the AD process become a very crucial operating parameter.



**Figure 6.** (a) Percentage of COD removal with an uncontrolled pH at 4.15 (b) Percentage of COD removal with a controlled pH at 6.8

Figure 6 shows the effect of controlled and uncontrolled pH on COD removal from wastewater. Studies have shown that a pH below 6 could hinder methanogenic activity, which lowers the quality of the biogas [25]. In this context, the wastewater sample (S2) had a low average pH of 4.15 and could disrupt the microbial community. The percentage of COD reduction was monitored without pH control, and this led to a 56% reduction in COD after 10 days, as shown in Figure 6(a). In Figure 6(b) the pH is increased, and the set point is 6.8. After 10 days the percentage of COD removal is 66% removal. With the increase in the pH, there was a 10% increase in COD removal. pH is a limiting variable on anaerobic digestion and the higher pH led to a greater COD reduction. A study was conducted to compare the effects of a controlled and uncontrolled pH dosing system on COD removal. It was found that a controlled pH dosing system has a greater positive impact on organic content degradation and resulted in a high COD reduction, thus confirming the observation made in this study [26].

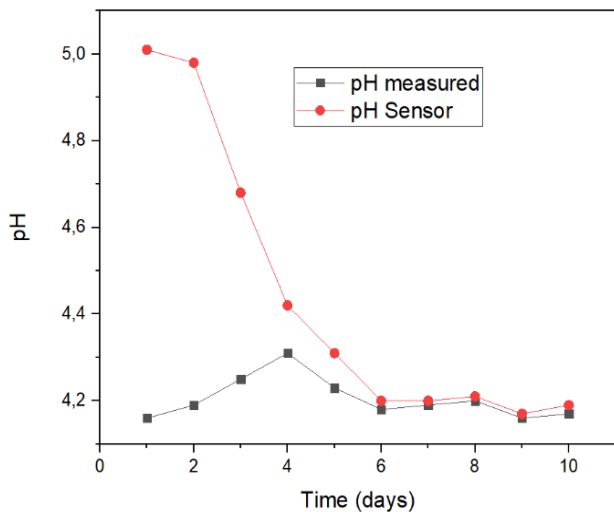
The optimum temperature and pH parameters determined in this study will be viable in the online monitoring and control of the AD system.

Both temperature and pH had a 10% impact on the percentage COD removal which implies that both factors equally impact the performance of AD.

Due to time and equipment constraints, the effect of temperature and pH on the AD system were not investigated at the same time.

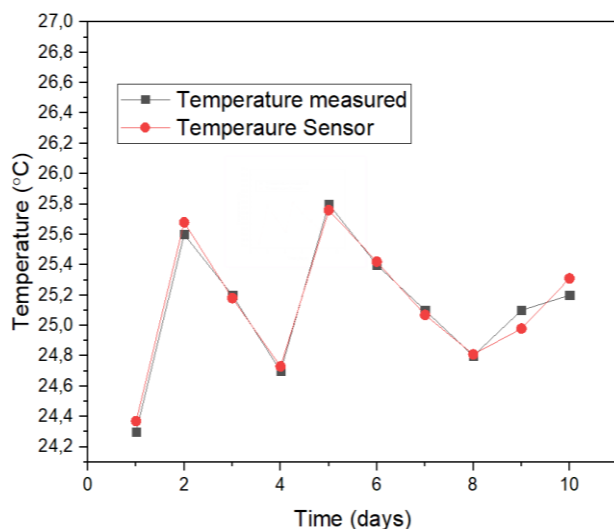
### 3.2 pH and temperature sensors calibration

Process optimization is made possible with online monitoring and control by maximizing capacity utilization and minimizing losses due to process failure. It is interesting to note that AD process microorganisms are directly impacted by operating variables like pH and temperature. For accurate data collection and the possibility of automating the AD process, pH and temperature sensors have to be calibrated.



**Figure 7.** pH sensor readings vs pH manual meter readings

Figure 7 shows the difference between the sensor readings and the readings obtained using a handheld pH meter while 6 shows the temperature measured online and using a handheld device. The usual assumption behind utilizing pH as a process indicator is that a decrease in pH correlates with VFA formation in the AD process. To maintain a pH-balanced reactor for microbial activity, it is necessary to employ pH monitoring and acid or base control. Yet, in a reactor with insufficient buffering capacity and no pH control, VFA accumulation can abruptly drop the pH. To determine the accuracy of the pH, this study compared an online pH sensor reading against a manual daily reading. For the first four days (days 1 - 4), the online sensor had a pH reading that was higher than the manual meter reading. The difference between the readings was calculated to be an average of 13%. This is a considerably high value which could lead to disruptions in the system. Some of the reasons for the discrepancy could be improper mixing or the placement of the sensors, however, by the fifth day, the sensor readings and meter readings were corresponding with now a difference of 0.5% as seen in Figure 7.



**Figure 8.** Temperature sensor readings vs manual readings

In Figure 8, the measured temperatures online and manually corresponded from the first day of the experiment. The difference between the values was only 0.05%. This shows that the sensor is well-placed and accurate. This implies that the temperature online sensor accurately and timeously detects changes in the system which will be essential in an automatic temperature control system. Similarly, for pH, after day 4, the pH readings were stabilized with a small error. This suggests incorporating sensors into the AD process for data collection and control is viable to maintain balance and avoidance process failure.

#### 4. CONCLUSION

This study investigated the effect of process parameters (pH, OLR, and temperature) on an anaerobic system for wastewater treatment. In addition, the efficacy of online pH and temperature data monitoring was compared with manual reading. It was deduced that the organic loading rate (OLR), which is a result of the characterized wastewater influenced the AD treatability efficiency (COD removal). The sugar refinery effluent (S2) had a high organic (1887 mgCOD/L) as compared to municipal wastewater (S1) with 432 mgCOD/L. Regarding the effect of pH on the AD system, the COD removal without pH was 56.2%. while with pH control (pH 6.8), the maximum COD removal was 66.5%. Moreso, the AD system at a mesophilic temperature of 40°C, resulted in 66% COD removal. The calibration of the online pH and temperature sensors had accurate precision with the manual readings with an error margin of 0.5 and 0.05 respectively. The findings of this study suggest AD system coupled with sensors monitoring and process optimization control has industrial prospect treatment of high organic wastewater.

The AD process parameters are optimised to an operational temperature 40°C and a pH of 6.8. Online sensors require efficient placement and will improve the operation of the digester. Due to frequent power outages in South Africa, the sensors went offline at unpredicted intervals. In order to avoid this issue, it is recommended that an uninterrupted power supply is added to the system.

For practical use of AD in the wastewater treatment industry, pH temperature and other AD operational parameters need to be controlled. Environmental benefits and economic value are additional essential aspects of the AD process. Thus, discharging effluent into the environment incurs high transport and energy costs. Therefore, smart-scale biogas technology deployment makes a lot of interest.

Further work includes: determining the biogas quality and quantity when wastewater with different organic loading rates is used, such as industrial wastewater; and repeating the runs investigating other process parameters, such as the total ammonia nitrogen concentration.

It is recommended that a techno economic study is conducted to determine the feasibility of installing and operating sensors in the AD system.

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