Anaerobic Digestion of Liquid Waste from an Attiéké Factory: From the Experimental Scale to the Semi-Industrial Scale

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Abstract
This study focused on the transfer of experimental results of anaerobic digestion of liquid waste from an attiéké (steamed cassava semolina) factory to a 6 m³ pilot digester. The experimental digester and the pilot were powered as follows: Lw + U + C (liquid waste + urine + cow dung). To the results, the experimental digester mesophilic with a progressive elimination of COD. Also, the nitrogen concentrations in the experimental reactor had little removal with alkaline pH. As for the biogas product in this digester, a volume of 3.6 m³ was obtained with a positive flammability test. The transition from the laboratory scale to the semi-industrial scale retains the results of purification and fuel biogas production of the experimental digester.

Keywords
Anaerobic Digestion, Cassava Liquid Waste, Biogas

1. Introduction
Cassava (Manihot esculenta) is the third food product in the tropics after rice and maize (FAO 2008) [1]. Annual production in Côte d’Ivoire, estimated at 2,450,000 tones, ranks second among food crops after yam (FAO, 2010, 2013) [2]. In addition, the ease and control of fresh cassava processing technologies makes it possible to obtain various products such as: gari, tapioca, placali, kokodé, attoupkou, attiééké, etc. (Akoroda, 2007 [3]; Kpata-Konan et al., 2013 [4]).
Very popular in Côte d’Ivoire where it has become a national dish, attiéké (steamed cassava semolina) is the main form of food use for cassava. Indeed, originally from the south, this dish is now produced and consumed throughout Côte d’Ivoire and by all socio-economic groups. Thus, this cassava-based food is now widely adopted across Africa, even the world.

However, does the manufacturing process of attiéké generate toxic liquid waste (Goualo et al., 2007 [4]; Kpata-Konan et al., 2013 [5]; Kpata-Konan et al., 2016 [6]; Kpata-Konan et al., 2018 [7]; Kpata-Konan et al., 2019 [8]), with a very high pollutant load (Ubalua, 2007 [9]; Kpata-Konan et al., 2016 [6]), which is released into the environment without prior treatment. This waste is an important source of pollution for the receiving environment. Indeed, these wastes degrade the living environment, generate olfactory nuisances, promote the spread of pathogens and cause risks to human and animal health (Marache, 2001) [10]. Also, cooking attiéké is done in a traditional way with a large consumption of energy whose main source is firewood or charcoal.

In Côte d’Ivoire, waste treatment trials using anaerobic digestion have been carried out on agricultural waste (Anonyme, 1981) [11], household waste (N’goran, 2006) [12] and cow dung (Kouamé, 2006) [13]. Thus, in order to substitute the current energy source for the production of attiéké by bioenergy, an attempt to produce biogas by digesting liquid waste from the production of attiéké was made in Azito-Village in the Commune of Yopougon (Mahan, 2004 [14]; Kpata, 2005 [15]). But the biogas produced was not combustible. Indeed, liquid cassava waste is biorecalcitrant with an acidic pH (pH < 3) and a nitrogen deficiency (0.6 - 0.8 g/L). To contribute to improving the purification performance of the anaerobic digester in the purification of liquid cassava waste from the attiéké manufacturing process, studies have been initiated on the co-digestion of this waste with human urine and cow dung. This anaerobic digestion can reduce the organic pollutant load by half. In addition, the residues (or digestate) obtained after anaerobic digestion are stable, deodorized, mostly free of pathogens and rich in nitrogen compounds (ammoniacal nitrogen, total nitrogen) (Kalloum et al., 2011 [16]; Kpata-Konan et al., 2016 [6]). They can therefore be valued for the amendment of agricultural soils.

This work aims to transfer the experimental results (experimental scale with a digester of around 200 L) of anaerobic digestion optimization of liquid waste from an attiéké factory to a 6 m³ (semi-industrial) pilot digester.

**2. Material and Methods**

**2.1. Material**

**2.1.1. Co-Substrates**

The substrates used consist of:
- 2.3 m³ of liquid waste from the pressing of cassava paste and washing of chips;
- 1.7 m³ of human urine collected in the village of Azito;
- 323 Kg of cow dung collected at the Azito slaughterhouse.
2.1.2. Reactors Design and Experimental Conditions

An experimental anaerobic reactor has been used (Figure 1(a) and Figure 1(b)). This reactor consisted of two metal drums of 100 L and 186 L, each open on one of the bases. The largest barrel contained digestion substrate and the smallest barrel was used as gasometer to store the produced gas. The experimental reactor used was powered as follows: Cassava liquid waste + human urine + cow dung (Lw + U + C).

For pilot scale, anaerobic digestion was carried out in a 6 m³ capacity bioreactor, closed hermetically (Figure 1(c) and Figure 1(d)). This digester has three parts. The upper part is occupied by the biogas produced. With a volume of 2 m³, the gas holder is equipped with a check valve, a safety relief valve and a manometer. The central part is occupied by the biodigestion cosubstrate (cassava liquid waste, human urine and cow dung). The reaction mixture has a volume of 4 m³. This part of the semi-industrial biodigester comprises a digester mixer powered by a gear motor FIMET, pipe supply, a discharge pipe on which is fixed a valve flap. The mixer is used to homogenize the medium so as to avoid sedimentation. The feed pipe is used for supplying the digester from a motor pump SDMO ST 2.36 H. As to the discharge pipe, situated at the bottom of the digester, it serves to drain the reactor.

2.2. Methods

Temperature, pH, Chemical Oxygen Demand (COD) and Total Nitrogen (estimated by Kjeldahl the method ((TKN)) were determined according to the standard methods (Table 1). Temperature, pH, COD and TKN were determined

![Figure 1](image_url). (a) and (b) 186 L experimental digester, (c) and (d) 6 m³ pilot digesters. Schemas (b) and (d) are respectively from Kpata-Konan et al. (2013) [5] and Kpata-Konan et al. (2015) [17].
Table 1. Synthesis of methods for the analysis of physico-chemical parameters (AFNOR, 1994 [18]; Rodier, 1996 [19]; CEAEQ, 1999 [20].

| Measured parameters             | Analytical Methods                                   | Comments on methods                                           |
|--------------------------------|------------------------------------------------------|---------------------------------------------------------------|
| pH and temperature             | Electrochemical glass electrode (NF T 90-008)        | In situ measurement using HANNA brand pH-meter                |
| Total Kjeldahl Nitrogen (TKN)  | Method after selenium mineralization (NF T 90-010)   | Digestion of 50 ml of sample and determination by titrimetry after distillation. |
| Chemical Oxygen Demand (COD)   | Potassium dichromate oxidizability method (NF T 90-101) | Oxidation by excess dichromate in an acidic medium followed by a dosage of the excess dichromate by the mixed iron and ammonium sulfate. |

twice per week. In this study, Carbon and nitrogen compounds were respectively determined as COD and TKN. The C/N ratio was estimated from the COD/TKN ratio.

Experimental digester: Volume ($V$) of biogas produced was measured daily using this formula:

$$V = H \pi R^2$$

(1); with $H =$ height of rising of the gasometer (small barrel); $R =$ Radius of the gasometer (small barrel).

Pilot: Volume ($V$) of biogas produced was measured daily using this formula:

$$V = \frac{P_i}{P_{\text{atm}}} \times e^{\gamma (V_1 - P_i)}$$

(2); with $P_i =$ initial pressure (bar); $P_{\text{atm}} =$ Atmospheric pressure (bar); $\gamma = 1.42$ (Gama for natural gas); $V_1 =$ Volume of gas holder (m$^3$).

The composition of the produced biogas was determined by gas chromatography. The energy value was obtained using the formula described by Ricard et al. (2010) [21], of the order of: 9.65 kWh/m$^3$ under standardized conditions. Depending on its methane content, the energy value of the biogas produced is obtained as follows:

$$\text{LCV} = 9.65M$$

(3); with: LCV: lower calorific value, expressed in kwh/m$^3$; $M$: percentage of methane in the biogas produced.

3. Results and Discussion

3.1. Characteristics of Substrate at the Inlet of Digesters

The COD of the human urine used is 12.64 g/L for an estimated TKN concentration of 2.64 g/L with a basic pH of 8.99 (Table 2). The liquid waste from the digesters was buffered before feeding. For the experimental digester, the recorded values of COD, TKN and pH are respectively 18.80 g/L, 3.75 g/L and 7.00. In the pilot digester, the COD, TKN and pH values observed are 27.46 g/L, 3.87 g/L and 7.02 respectively (Table 2). The COD/TKN ratio were 4.97 for the experimental digester and 7.08 for the pilot digester.
Table 2. Substrate parameters at the inlet of the digesters.

| Parameters | Human urine | Expérimental Digester | Pilot |
|------------|-------------|------------------------|-------|
| COD (g/L)  | 12.64       | 18.80                  | 27.46 |
| TKN (g/L)  | 2.64        | 3.78                   | 3.87  |
| COD/TKN    | 0.47        | 4.97                   | 7.08  |
| pH         | 8.99        | 7.00                   | 7.02  |

3.2. Physico-Chemical Parameters of the Mixture at the Outlet of the Digesters

The experimental digester and pilot showed pollutant load reductions ranging from 18.78 g/L to 0.29 g/L and 27.46 g/L to 5.01 g/L respectively (Table 3). The corresponding purification efficiencies are 98.43% for the experimental digester and 81.75% for the pilot.

The amount of nitrogen observed in all digesters decreased slightly throughout the experiment. The observed TKN concentrations ranged from 3.78 g/L to 2.49 g/L for the experimental digester and from 3.87 g/L to 2.08 g/L for the pilot. The corresponding purifying yields are 34.07% for the experimental digester and 46.18% for the pilot.

The COD/TKN ratio varied from 4.97 to 0.11 in the experimental reactor and from 0.86 to 0.50 in the pilot (Table 3).

The average temperatures recorded in the experimental digester and the pilot are 28.03°C and 29.0°C respectively. In the digesters, the average pH is 7.45 for the experimental and 7.87 for the pilot.

3.3. Biogas Produced at the Different Digesters

The cumulative biogas production recorded at the experimental reactor after 114 days of operation is 3.60 m³. That of the pilot was 359.18 m³ after 192 days of operation (Figure 2). At the flammability test, it is positive in the experimental digester and the pilot after respectively from the 4th day until the end of the experiment and after 50 days of operation.

3.4. Discussion

The analysis results show a progressive elimination of the pollutant load (COD) of liquid waste. This indicates that the digesters are working well overall. According to Doré (1989) [22], these reductions of the polluting load could be explained by the potential consumption of organic matter by the purifying microflora during its natural evolution in these digesters.

For nitrogen pollution, the concentrations observed in all digesters decreased slightly throughout the experiment. This small decrease in nitrogen could be explained by the low volatilization of ammonia nitrogen observed in anaerobic digestion (Maiga et al., 2008) [23]. Indeed, Barana (2000) [24] reported that anaerobic digestion processes conserve nitrogen.
Table 3. Variation of physico-chemical parameters at the outlet of experimental digesters.

| Parameters                | Experimental digester | Pilot           |
|---------------------------|-----------------------|-----------------|
| COD (g/L)                 | 18.78 - 0.29          | 27.46 - 5.01    |
| Carbonaceous purifying efficiency (%) | 98.43                | 81.75           |
| TKN (g/L)                 | 3.78 - 2.49           | 3.87 - 2.08     |
| Nitrogenous purification efficiency (%) | 34.07                | 46.18           |
| COD/TKN                   | 4.97 - 0.11           | 0.86 - 0.50     |
| Average T°C               | 28.03                 | 29.0            |
| Average pH                | 7.45                  | 7.87            |

Figure 2. Cumulated volume of biogas produced during the monitoring time of the experimental (a) and pilot (b) digesters.

In this study, the treatment of cassava liquid waste with human urine reduced the COD/TKN ratio to below 50 as recommended by Liu et al. (2008) [25] for anaerobic treatment.

Concerning temperature, all digesters operated in the mesophilic fermentation range (24°C and 35°C). This temperature range is favored by the country’s tropical climate characterized by strong sunshine and average annual temperatures above 26°C (Kouamé et al., 2010) [26]. According to De La Farge (1995)
mesophilic systems are the most common and best controlled.
The digesters worked with alkaline pH values. This alkalinity could be explained by the contribution of human urine, which is of a basic nature (Kpata, 2005) [15], to the acid cassava effluent (Ubalua, 2007) [9]. Biogas production and flammability testing are important for controlling and monitoring the anaerobic digestion process. A positive flammability test indicates that the digester is working properly. In the pilot digester, the flammability test was recorded after 50 days of operation, unlike the experimental digester which was observed on the 4th day of operation. This relatively long time before gas combustion could be justified by the lack of cow dung introduced into the reactor, which did not favour the rapid development of microorganisms. Indeed, the work of Kalloum et al. (2007) [28] on the anaerobic digestion of household waste and Igoud et al. (2002) [29] on the anaerobic digestion of bovine waste produced biogas after 25 and 10 days of operation respectively.

4. Conclusions
This work focused on the transfer of the results of anaerobic digestion of liquid waste of the attiéké (steamed cassava semolina) factories from the experimental scale to a pilot. This study was conducted over 114 days for the experimental digester and 192 days for the pilot. The transition from laboratory to semi-industrial scale maintains the results of purification and biogas production despite the production of fuel biogas after 50 days of operation in the pilot. However, it is becoming economically obvious that the production of biogas from the liquid waste from the attiéké (steamed cassava semolina) factories will be an important source of income, which could effectively reduce the use of firewood for cooking the attiéké.

In order to consolidate the results of liquid cassava waste treatment by anaerobic digestion with human urine as co-substrate and to facilitate the popularization of this technology, it would be interesting to accompany it with an engineering effort to automate the feeding of raw wastewater to the biodigester and the evacuation of the digestate.

Conflicts of Interest
The authors declare no conflicts of interest regarding the publication of this paper.

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