

Research Article

# Characterization of *Adansonia Digitata* Seed Cake with a View to Identifying an Energy Recovery Strategy

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## Abstract

Agri-food processing emits huge quantities of waste and is considered one of today's major environmental problems. There are many different options (anaerobic digestion, thermochemistry and feed production) that can be applied to the management and evaluation of waste treatment. The aim of the present study was to explore the appropriate recovery option for *Adansonia digitata* seed cake through its characterization. The physicochemical characteristics of *Adansonia digitata* seed cakes were determined in accordance with international standards. Anaerobic digestion was tested under thermophilic conditions in batch mode over 75 days. Results showed that dry matter content averaged  $87.35 \pm 0.03\%$ , organic matter content averaged  $95.03 \pm 0.41\%$ , carbon to nitrogen ratio averaged 19.75. Digestion of *Adansonia digitata* seed cakes proved highly productive, with a maximum percentage of 68.5% CH<sub>4</sub> for 185.57 liters of biogas, i.e. an average production of 2.47 liters per day for 1720 g DM. The methanogenic potential (BMP) of *Adansonia digitata* seed cake was 331.21 ml/g of organic matter introduced. *Adansonia digitata* oilcake had an average gross calorific value of 18.54MJ/Kg. These results are encouraging and mark the start of any study on the energy recovery of *Adansonia digitata* seed cake in biogas.

## Keywords

*Adansonia digitata*, Biomass, Characterization, Energy

## 1. Introduction

Growing demand for renewable energies to reduce our dependence on traditional fossil fuels is essential in the face of today's environmental and energy challenges. This transition to more sustainable energy sources also calls for innovative solutions to valorize waste, particularly in the agricultural

sector. Methanization of agricultural biomass, through anaerobic digestion (AD), represents a promising approach offering significant benefits such as the production of bioenergy and organic fertilizers [1-3].

The African baobab (*Adansonia digitata*) is a promising

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plant with almost every part used for nutritional, medicinal or even industrial purposes (the seeds are used as a coagulant). The seeds are a rich source of minerals, amino acids and the fatty acid oleic acid. A great deal of research has been carried out on the nutritional aspects of seeds, but little on the oil. The literature reveals that seeds contain 22-45% oil on a dry matter basis [4, 5].

Demand for African baobab seed oil in European, Asian and North American markets has risen sharply in recent years. According to the Global Market Insights report, the estimated value of baobab products, particularly pulp and oil, was around US\$3.7 billion in 2017. This demand is forecast to reach \$5.6 billion by 2024 [4, 6]. African countries exporting baobab seed oil to specialized markets include South Africa, Sudan, Tanzania and Zimbabwe, as well as other West African states [7]. At the present stage of knowledge, the usefulness of baobab (*Adansonia digitata*) oilcake is not sufficiently documented in the literature.

AD is proving effective in reducing the mass of solid organic waste while converting its organic carbon into biogas, a competitive renewable energy source [8, 9]. As well as helping to reduce pollution, this process is in line with the growing trend towards local products in developing countries, where agri-food industries are expanding rapidly. However, these industries also generate large quantities of waste, such as baobab oilcake derived from the processing of *Adansonia digitata*. Although this oil cake has previously been considered unsuitable for animal feed due to its high cellulose content [10], its potential for biogas production remains largely unexplored. The aim of this study is therefore to assess the possibility of valorizing *Adansonia digitata* oilcake by determining its methane production potential through anaerobic digestion and its energy content. The first part of the manuscript will focus on presenting the materials and methods used, while the second part will look at substrate characterization and digestion processes for efficient biogas production.

## 2. Materials and Methods

### 2.1 Materials

#### 2.1.1. Substrate

*Adansonia digitata* seed cake

*Adansonia digitata* seed cake used in the study was recovered after cold processing of *Adansonia digitata* seeds into vegetable oil at the Miseriocardia-Dei company in Zogbodomey (Benin). Figure 1 shows the texture of the *Adansonia digitata* seed cake used in the study.



Figure 1. Texture of *Adansonia digitata* seed cake.

#### 2.1.2. Equipment Used

##### (i). pH Meters

pH is measured using an ecoTestr pH 2cs meter in accordance with ISO 10523:2008. Equipped with a glass electrode immersed in an appropriate volume of the sample, the digital reading gives the pH value to two decimal places.

##### (ii). Oven

A Memmert oven with a temperature range of up to +300 °C was used to determine the dryness of *Adansonia digitata* seed cake.

##### (iii). Muffle Furnace

Volatile organic matter and total volatile solids were determined using a Nabertherm muffle furnace with a temperature range of up to 1100 °C.

##### (iv). Balance

A Citizen-type balance with a maximum capacity of 210 g and an accuracy of (0.0001 g) was used to weigh the substrates.

##### (v). Calorimeter

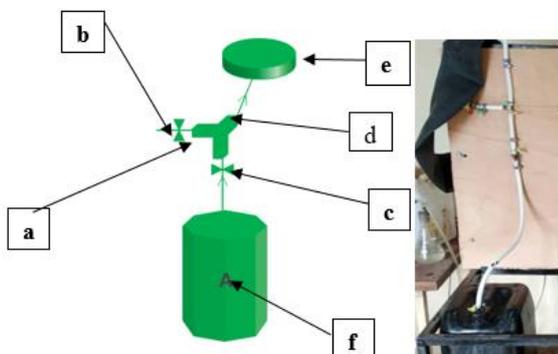
Heat capacity was measured using a Parr model 6050 automatic static chamber calorimeter. It has an accuracy of 0.2%.

##### (vi). Biogas Analyzer

The composition of the biogas produced was analyzed using a portable gas analyzer (BIOGAS 5000). The analyzer samples biogas continuously at a flow rate of 550 ml/min. Infrared sensors are used to determine and quantify methane and carbon dioxide content (accuracy approx. 1.5%). Electrochemical sensors are used to quantify oxygen (accuracy approx. 1.5%) and hydrogen sulfide (accuracy approx. 3%).

### (vii). Digestion Unit

The digestion unit consists of a 20-liter digester, an air chamber serving as a gasometer, and flexible piping. Figure 2 shows the designed digester.



**Figure 2.** The digestion unit: a: 1/4" gas valve; b: biogas outlet; c: copper tee; d: pvc gas piping; e: gasometer and f: digester.

## 2.2. Methodology

### 2.2.1. Substrate Characterization

#### (i). Dry Matter

Dry matter is determined in accordance with NF ISO 11465 AFNOR X 90-029 (1994) by drying in an oven at 105 °C for 24 hours. The difference in weight corresponds to moisture loss, and the residue represents the dry matter content of the sample. The capsule is weighed after cooling in a desiccator. The dry matter content is determined by equation 1.

$$MS = \frac{M_f - M_v}{M_i - M_v} \times 100 \quad (1)$$

Mf: final mass of the crucible and the sample after drying at 105 °C (g); Mv: mass of the vacuum crucible (g); Mi: mass of the crucible and the sample before drying (g); MS: dry matter (g).

From equation 1, we obtain the moisture content (% H) of the substrate which is:

$$\% H = 100 - MS(\%) \quad (2)$$

#### (ii). Organic Matter Content

The organic matter content (total volatile solids for waste water) of the samples is measured in accordance with procedure NF U 44160 (1985). The previously dried samples are subjected to calcination in a furnace at 550 °C for 4 hours in an oxidizing atmosphere, the organic matter burns and the residual matter constitutes the mineral matter. The loss of mass, relative to the amount of dry matter, corresponds to the rate of volatile matter.

$$MO(\%) = \frac{M_f' - M_v}{M_f - M_v} \times 100 \quad (3)$$

Mf': final mass after calcination at 550 °C; Mf: final mass after drying at 105 °C; MO: organic matter.

### (iii). Determination of Total Kjeldahl Nitrogen (N-NTK)

Total Kjeldahl nitrogen was determined in accordance with AFNOR T90-110. The Griess colorimetric method was used. Almost one gram (1 g) of a sample is added to a Kjeldahl tube, followed by 20 mL of concentrated sulfuric acid H<sub>2</sub>SO<sub>4</sub> (96%) and 5 grams of Kjeldahl catalyst (Cu-Se). The sample is then transferred to the digestion block for mineralization, successively at the following temperatures: 180 °C for 2h, 250 °C for 2 h and 340 °C for 2 h. After digestion, the tubes are removed and cooled for approximately 10 min. 30 mL of water are added, and the whole is stirred directly with the vortex mixer to dissolve residual salts. Next, the water is made up to 75 ml and a stopper is placed on the tube and inverted several times. Leave to cool for 24 hours. Total nitrogen is determined using an automated colorimetric analyzer. The color produced by the reaction between ammoniacal nitrogen, salicylate, nitro-ferricyanide and hypochlorite is measured as a spectrum with a wavelength of 660 nm.

### (iv). Determination of Total Carbon

A furnace combined with a Shimadzu TOC-VCSH model carbon analyzer was used to determine soluble total organic carbon. The analysis was carried out according to MA 405-C 1.1, which is a modification of the Walkley and Black method. The method is based on thermal oxidation at 900 °C of all the carbon present in the sample to CO<sub>2</sub> and subsequent detection of CO<sub>2</sub> by infrared spectrophotometry.

### 2.2.2. Methanization

#### (i). Experimental Protocol for Anaerobic Digestion of Substrates

As *Adansonia digitata* seed cake is a solid, woody waste, it is advisable to carry out a pre-treatment prior to any digestion trial. The pre-treatment protocol applied is explained in the following sub-session.

Pre-treatment of *Adansonia digitata* seed cake: As the granulometry of *Adansonia digitata* seed cake is substantial, mechanical pretreatment (grinding with an electric mill) is essential [2, 11]. A first dosage is established using equation 4, with the aim of obtaining a substrate with a dryness of 10% as recommended in the literature for optimal wet digestion [3], and dignifying the lignin contained in the *Adansonia digitata* seed cake.

$$m_{H_2O}ajt(kg) = \frac{(90\% \times \tau_{MS})}{10\%} \quad (4)$$

From equation 5 .1kgMS of *Adansonia digitata* seed cake requires 7.86 liters of water for dilution. Dilution yielded *Adansonia digitata* cake juice, the characteristics of which are summarized in Table 2. The quantity of TB required for the useful digester volume is determined using equation 5.

$$m_{TB}(kg) = \left( \frac{V_c}{\frac{1}{\rho_{TB}} + \frac{7.86}{\rho_{H_2O}}} \right) \quad (5)$$

Where: mTB: the mass of TB required for the load volume of the digester; ρJTb: the density of TB; 7.86: the dosage required for 1 kg of TB (obtained using equation 4) and Vc: load volume of the digester.

The mixture is left for 3 days to ensure that the nutrients mix and dissolve, in order to initiate pre-degradation prior to digestion proper. The aim is to accelerate hydrolysis, increase pore exchange surface area and reduce the risk of organic overload. *Adansonia digitata* cake juice was introduced at a rate of 10 liters into a 20-liter digester. The useful volume is 15 liters. The cake to inoculum ratio (substrate vs inoculum) is (2/3, 1/3).

Next, the pH is adjusted with sodium bicarbonate to, in order to achieve the optimum growth range for the 7–8 microorganisms [3, 12, 13].

Finally, an inoculum operating in a mesophilic digester (cow sludge juice separated from organic matter using a separator at a digester in operation) was added at 1/3 of the useful volume in the digester.

### (ii). Biogas Analysis

Biogas analysis consists in measuring the different compositions of the biogas produced through interaction between electrochemical cells and infrared sensors, in order to get an idea of the composition of the biogas produced. It also involves estimating the volume of biogas produced during each digestion period. To evaluate the volume of biogas produced, equation 6 is used when the torus has its normal shape.

$$V = 2\pi^2 \times r^2 \times R \quad (6)$$

With *r* the small radius of the torus and *R* the large radius (outer radius), as shown in the figure below.

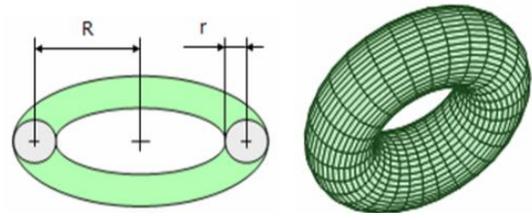


Figure 3. Illustration of the gasometer (torus).

The absolute error is determined on each measurement; the absolute error on the measuring tape is 1 cm. So, for the small radius,  $r=d/2=p/2\pi$  hence  $\Delta r = |\Delta p/2\pi|$ .

For the large radius,  $R=D/2=(P/2\pi)-r=(P/2\pi)-(p/2\pi)$  hence  $\Delta R = |\Delta P/2\pi| + |\Delta p/2\pi|$ .

Since equation 6 is a product, the relative error on volume is calculated from the relative errors of each term according to the following expression:  $|\Delta V/V| = 2|\Delta r/r| + |\Delta R/R|$ .

### (iii). Conversion Efficiency

At the end of the experiment, the conversion efficiency in VS [%] was calculated for all the mixtures in this study using equation (7) [14]:

$$E(\%) = 100 \times \frac{V_{Sb} - V_{Se}}{V_{Sb}} \quad (7)$$

Where  $V_{Sb}$  and  $V_{Se}$  are the quantities of VS in the reactor at the start and end of the batch test respectively. This parameter is used to assess the degree of conversion of volatile solids into biogas.

### 2.2.3. Higher Heating Values (HHV) of *Adansonia Digitata* Seed Cake

The higher heating value (HHV) of *Adansonia digitata* seed cake was determined in accordance with the XP CEN/TS14918 standard protocol using a Parr model 6050 automatic static chamber calorimeter. A crucible containing approximately 1 g of biomass sample was placed in a metal bomb under a high-pressure oxygen atmosphere at ambient temperature (25 °C).

## 3. Results

### 3.1. *Adansonia digitata* cake Oilcake Characterization Results

Table 1 presents the characterization results (dry matter content, volatile organic matter, C/N ratio) for baobab seed cake.

**Table 1.** Physico-chemical characteristics of *Adansonia digitata* cake.

Parameters	Baobab cake	Standard deviation
pH	5.5	-
N (%)	2.74	-
C/N	19.75	-
MO (%)	95.03	0.41
Dryness (%)	87.35	0.03
Total solids (%)	-	-
Total volatile solids (%)	-	-
Total carbon (%)	54.03	-
$\rho$ (kg/l)	1.45	0.29
Higher Heating Value (HHV) (MJ/kg)	18.43	0.37

From the results of the characterization of *Adansonia digitata* oil cake, they present an acid pH (5.5) with a carbon to nitrogen ratio of 19.75. This ratio is within the optimal range for good anaerobic digestion ( $25 \leq C/N \leq 30$ ) [15]. The fairly high dryness content obtained points the study towards a high dosage to obtain a wet substrate. The organic matter levels, which are of the order of  $95.18 \pm 0.44\%$ , lead to the conclusion that *Adansonia digitata* oilcake has a high potential in terms of recoverable biomass for energy purposes.

The higher heating value is around 18.43 MJ/kg. Compared with that of coal, this value shows the high energy potential of baobab oil cake, reinforcing the idea of its promising energy valorization, which means that baobab oil cake can be used as a fuel and, better still, produce ecological coal by carbonization.

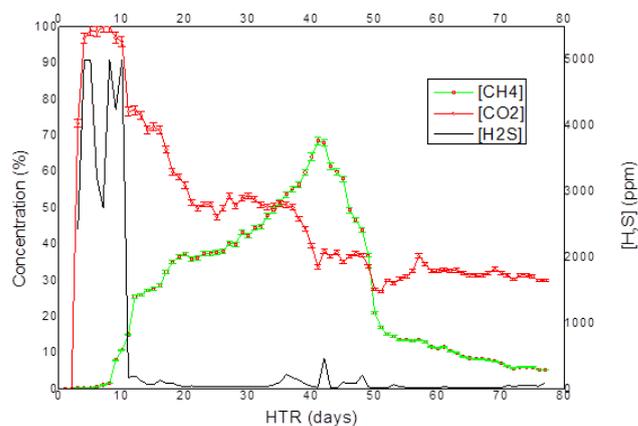
### 3.2. Results of Quantitative Analysis (Molar Composition) of the Biogas Produced

Figure 4 shows the temporal evolution of methane and hydrogen sulfide production in the biogas generated by *Adansonia digitata* seed cake digestion.

Figure 4 shows that from days 1 to 6 of digestion, methane production remains low in all digesters. This is due to the fact that, at the start of metabolism, hydrolysis is the predominant process and the limiting factor in the initial degradation of organic matter. Degradation rates depend on the materials to be degraded. Carbohydrates are degraded first, followed by proteins and lipids, and finally cellulose. Acceleration of this stage requires regular agitation.

From day 8, methane production increases exponentially until day 41, when it reaches its maximum (68.5% methane) for the digester. This can be explained by the stabilization of the microorganisms over time. The environment has been

favorable to their maturation, and they have the quantity of nutrients required for the methanogenic and hydrogenophilic bacteria to initiate the methanogenesis phase. This was supported by [16, 17], who showed that it is not until around a week after the start of the process that the methanogenesis bacteria begin to rapidly consume nutrients to produce biogas.

**Figure 4.** Biogas production kinetics.

After the 41st day of activity, biogas production begins to gradually decline until the end of digestion. This reflects the depletion of organic matter in the environment for the micro-organisms. Authors such as [17] have shown that as soon as the ascending phase of production is reached, the methane content of the biogas becomes progressively lower throughout the rest of the process. The latter justify this low production by the decrease in the bacteria/substrate ratio. They also point out that the bacteria are either transported by the effluent in the digestate (in the case of continuous digesters) or partly die due to their aging rates (in the case of the present study).

Furthermore, hydrogen sulfide concentration at digester level is very high on the first 5 to 8 days of digestion (>5000 ppm) and on the first 11 to 13 days for the others. These high  $H_2S$  concentrations are due to the high sulfur protein and sulfate content of the substrates [18]. This not only destroys the microbial population, but is also corrosive to ferrous materials. The same observation was made by [19]. From day 10 onwards, we observe a drop in hydrogen sulfide concentration in the digester. As a result, some bacteria are likely to survive, others to die. This could explain the slow start to methane production from the digesters.

Based on the above analysis, *Adansonia digitata* seed cake is a candidate for methanization (C/N=19.75 ratio close to the optimum range). The digester has a maximum methane production of 68.5%. It is important to note that the methane content obtained in the digester is within the orders of magnitude of agri-food waste in the literature (68–70%) [3].

### 3.3. Results of Quantitative Analysis (Volume Produced)

Figure 5 shows the cumulative biogas production over 75 days of digestion.

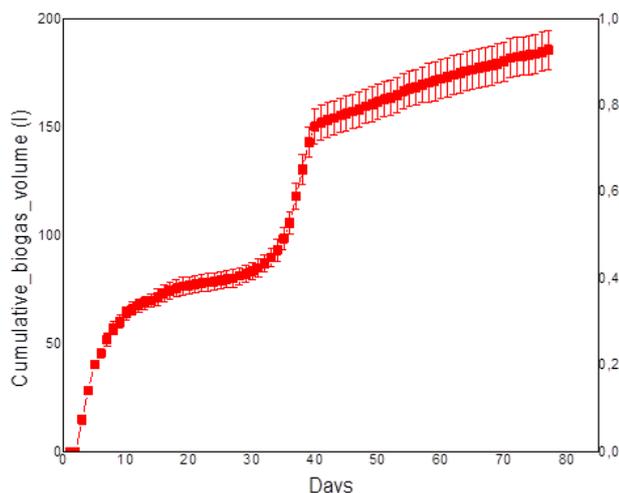


Figure 5. Cumulative biogas production over digestion time.

Figure 5 shows that after the first three days of experimentation, cumulative biogas volumes in the digester follow an exponential trend. This phenomenon can be explained in part by the initial presence of a large number of bacteria in the medium (just after seeding), promoting the breakdown of macronutrients into micronutrients (hydrolysis) to initiate the process. There is also an increase in daily biogas production throughout the digestion period. Cumulative biogas volume in the digester reached 185.57 liters.

### 3.4. Conversion Efficiency Results

The different %MS and %MO ratios proposed were characterized before and after digestion in order to calculate the abatement (efficiency) of digestion. Table 2 shows the results of post-digestion substrate characterization.

Table 2. Digestion efficiency of *Adansonia digitata* cake.

MO (%)		Efficiency (%)
input	output	
88.90	73.03	17.98

Table 2 shows a digestion efficiency of 17.98%. This suggests a relatively low level of nutrient digestion by the microorganisms in the digester. Consequently, efficient effluent

recovery could be envisaged after digestion.

### 3.5. BMP of *Adansonia Digitata* Cake

Figure 6 shows the cumulative volume of methane over the retention time.

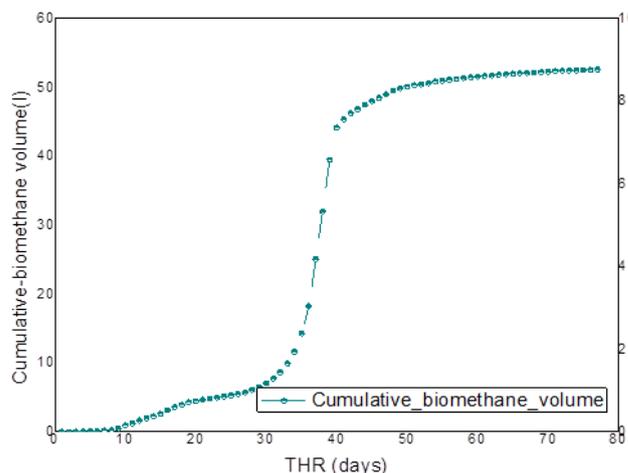


Figure 6. Cumulative methane production for *Adansonia digitata* cake digestion.

Figure 6 shows that the maximum cumulative methane production for *Adansonia digitata* seed cake digestion is 52.56 liters for 158.69 g of organic matter introduced. This represents an average daily production of 0.70 liters. The methanogenic potential (BMP) for *Adansonia digitata* seed cake digestion is 331.21 ml/g of organic matter introduced. It is important to note that the BMP of *Adansonia digitata* oil cake obtained is in the order of magnitude of the BMP of agri-food waste (236 to 1334 NLCH<sub>4</sub>.kgMV<sup>-1</sup>) [20].

## 4. Discussion

Based on analysis of the results obtained from the present study, it emerges that *Adansonia digitata* oilcake is a raw material suitable for anaerobic digestion (C/N ratio=19.75 close to the optimum range). CH<sub>4</sub> content rose to 68.5% v/v. It is important to note that the methane content obtained in the digester is within the orders of magnitude of agri-food waste in the literature (68-70%) [3]. The methanogenic potential (BMP) for the digestion of *Adansonia digitata* meal is 331.21 ml/g of organic matter introduced. It is important to note that the BMP of *Adansonia digitata* cake obtained is of the order of magnitude of the BMP of agri-food waste (236 to 1334 NLCH<sub>4</sub>.kgMV<sup>-1</sup>) [20]. The higher calorific value of baobab cake is of the order of 18.43 MJ/kg. This promising bioconversion performance marks the start of a major study into the conversion of *Adansonia digitata* seed cake into biogas or fuel briquettes. With performances like these, it will also be in-

teresting to investigate in future research the possibility of producing activated carbon as a biodegradation catalyst.

## 5. Conclusions

Characterization of the *Adansonia digitata* oil cake yielded the following results: The organic matter content was  $95.18 \pm 0.44\%$ . The fixed carbon content is  $54.03\%$ . The gross calorific value is  $18.43 \text{ MJ/Kg}$ . These characteristics suggest that *Adansonia digitata* oil cake can be used as a raw material for the formulation of high-energy biofuels.

Anaerobic digestion of *Adansonia digitata* cake (TB) showed remarkable efficiency, reaching its peak methane production on day 41. This AD produced a biogas with a high methane content, peaking at  $68.5\% \text{ CH}_4$  for a total volume of  $185.57 \text{ liters}$ . The methanogenic potential (BMP) of TB was  $331.21 \text{ ml/g}$  of organic matter introduced. The higher heating value of baobab oilcake is around  $18.43 \text{ MJ/kg}$ . These promising results mark the start of a major study into the conversion of *Adansonia digitata* seed meal into biogas.

## Abbreviations

BMP	Biomethane Potential
MO	Organic Matter

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## Author Contributions

**Kayaba Haro:** Conceptualization, Investigation, Writing and editing

**Abdel Anziph Sergel Khalid Nourou:** Investigation, Data curation, Writing original draft

**Sayouba Sandwidi:** Writing – review & editing

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**Guy Christian Tubreoumya:** Writing – review & editing

**Antoine Bere:** Writing – review & editing

**Oumar Sanogo:** Conceptualization, Supervision, Methodology, Validation, Writing – review & editing

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## Data Availability Statement

The data used to support the findings of this study are included in this article.

## Conflicts of Interest

The authors declare no conflicts of interest.

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