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# **Evaluation of Soumbara (***Parkia biglobosa***) Quality During Storage Using Mid-Infrared Spectroscopy and Physicochemical Methods**

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## **Introduction**

*Parkia biglobosa*, known under the name Néré, is a woody food species of multiple-use wooded parks belonging to the Parkia genus and the Mimosaceae family, which is of great dietary importance both regionally and internationally (Mariam et al., 2020). The pod-shaped fruits of *P. biglobosa* are consumed not only for their floury and sweet pulp but also and especially for their seeds used in the preparation of fermented condiments in West Africa. The néré seeds (*Parkia biglobosa*) are traditional fermented products widely consumed in West Africa as condiments (Souley & Diadié, 2020). The fermeted Néré seeds of *Parkia biglobosa*) are known in West Africa under different names: soumbala in Burkina Faso, nététu in Senegal, Soumbara in Guinea Conakry, dawadawa and iru in Nigeria, afitin, iru and sonru in Benin (Ouolouho et al., 2017). They are also known in Ivory Coast as Soumbara (name in Bambara) (Ibrahima et al., 2021). The transformation of fermented seeds of *P. biglobosa* occurs through three essential phases: i) double cooking of the

seeds, ii) fermentation of the cotyledons and iii) drying of the fermented product.

These traditional technologies are characterised by three main stages: a first cooking, shelling of the cooked seeds and a fermentation stage (Mariam et al., 2020). It is one of the popular food seasonings produced from the alkaline fermentation of *Parkia biglobosa* seeds in many West African countries (Akabanda et al., 2018 ; Savadogo et al., 2011). Indeed, Soumbara is an essential source of nutriments. It contains 30 to 47% protein, 20 to 43% lipids and 13 to 17% carbohydrates, and are a great source of energy (464-546 Kcal/100 g), and is a rich source of amino acids (Parkouda et al., 2009). The seeds of this plant are valued for their high polyphenol content, highlighting their therapeutic antioxidant and anti-inflammatory, antimicrobial, antidiabetic and antihypertensive properties (Gilsonda Akweley Kordei Attuquaye; et al., 2023). They constitute a source of protein for low-income families (Ahouansou, 2012). The cheese taste of Soumbara is due to

the presence of glutamic acid, which is very popular and plays an important role as a flavor enhancer in many dishes (Ouoba et al., 2007). In addition, it is also a source of essential fatty acids and vitamins, particularly B group vitamins(thiamine, riboflavin and niacin) (Ndir et al., 2000), as well as minerals such as phosphorus, calcium and iron. The strong flavor and odor characteristic of Soumbara are due to the production of certain chemical constituents such as pyrazines, ammonia, esters, ketones and acids during the fermentation technique (Yérobessor et al., 2020). In addition to these biochemical constituents, it enhances the taste of sauces accompanying cereal-based dishes such as rice, millet, sorghum, corn, etc. (Souley & Diadié, 2020).

In Guinea, the traditional production and fermentation of néré seeds includes: boiling the seeds for 24 to 40 hours, hulling, second parboiling for 1 to 3 hours, fermentation for 48 to 72 hours (25-30  $^{\circ}$ C), drying at in the open air and molding into balls of different sizes (Ouoba, 2017). This technology is not compliant in terms of safety and the quality of the final product (Ouoba, 2017). The uncontrolled process of this technology in terms of good manufacturing and hygiene practices, as well as the non-compliant sales conditions lead to doubts about its physicochemical and health quality (Cheyns & Bricas, 2003; Ouoba, 2017). In addition, the use of rudimentary equipment, the spontaneous fermentation technique as well as sales practices are favorable conditions for the development of certain reactions deteriorating the quality of Soumbara (Glover et al., 2018). Moreover, the organoleptic and nutritional qualities and the safety and stability of soumbala depend largely on the conditions of production and sale. It has also been reported that a lack of knowledge of hygiene rules, lack of training in quality management systems or the concept of good manufacturing practices and non-compliance with processors' practices have led to health risks for consumers (Yérobessor et al., 2020). Despite this problem, to the authors' knowledge, no study has yet focused on monitoring the quality of Soumbara during storage. The work aims to explore the possibility of using mid-infrared spectroscopy and physicochemical methods to assess the quality of soumbara during storage.

## **Material and Methods**

## *Sampling*

The Soumbara samples (5 kg) were taken from the aviation market in Conakry (Guinea). Soumbara was produced by fermentation of néré seeds (*P. biglobosa*) according to the method described by Cissé et al., (2021). All the seeds constituting the samples had the same stage of maturation. Thus, the process of obtaining Soumbara began with soaking the seeds followed by shelling and the first cooking for 13 hours. They were then peeled and washed before being cooked for a second time for 2 hours. After draining and sorting, the seeds were fermented for 72 hours at a temperature of 40°C. Finally, a display in the sun was carried out to obtain the soumbara. The Soumbara samples were carefully sorted, then placed in plastic bags and covered with aluminum foil. Conditions similar to those of sale were created to transport the samples to the laboratory to maintain the quality of the samples as they were taken. The samples were ground using a blender (Moulinex Valentin mini-chopper, France) in the

laboratory (laboratory of the Institut Charles Viollette site Artois (Arras, France)). Subsequently, they were subdivided into seven (7) groups, vacuum-packed, and stored at room temperature. Every 5 days of storage (days 1, 5, 10, 15, 20, 25 and 30), samples were taken for analysis in the laboratory.

## *Physico-chemical Measurements*

The moisture content and dry matter were determined following the AOAC method (1990).

The water activity was measured using equipment AquaLab (Water activity Meter, 4TE). Total protein content was carried out according to the Kjeldahl method described by the AOAC method (2006). The pH measurement of Soumbara powders, was done by pH meter (model 3110 Germany).

The total ash content of the Soumbara samples was determined by dry ashing in a muffle furnace at a temperature of 600°C for 6h (AOAC, 2006). All the measurements were determined in triplicate.

## *Colour Measurements*

The Minolta Chroma colorimeter (Konica Minolta Sensing Europe, Roissy Charles De Gaulle, France) was used to evaluate the color parameters of the Soumbara samples. Before taking colorimeter readings, the colorimeter was calibrated using a black box and a Petri dish. Thus, 10 g of Soumbara powder was put in a Petri dish, everything was placed on the sample holder of the colorimeter. The CIE Lab colour parameters: lightness (*L\**), redness (*a\**) and yellowness (*b\**) have been determined. *L\** is lightness component in the range of 0 to 100 while *a\** (Redness parameter) and *b\** (Yellowness parameter) are two chromatic components without numerical limit. For each sample, the analyzes were carried out in triplicates.

# *Mid-infrared Spectroscopy Measurements*

The spectral region of 4000-700 cm<sup>-1</sup> provides information on molecular bonds with fundamental valence vibrations of functional groups and a high detection level, e.g. compounds present at levels above 0.1% (Karoui et al., 2005). The 4000 and 700 cm-1 spectral range at a resolution of 4 cm-1 was chosen to acquire MIR spectra at a temperature of 20°C. The ATR cell consisted of a horizontal ZnSe crystal with an incidence angle of 45° and a total reflection of 10. The Soumbara powder was placed on the surface of the ATR and light pressure was applied to the handle to allow good contact between the crystal and the samples from Soumbara. Before acquiring the spectra of each sample, the ZnSe crystal was recorded and used as background. The analyses were carried out in triplicate for each sample.

## **Mathematical** *Analysis of Data*

The MIR spectra were normalised by reducing the area under each spectrum to a value of 1. Then, principal component analysis (PCA) was separately applied to the normalized physicochemical and MIR spectra. A factorial discriminant analysis (FDA) was carried out on the first 5 PCs of the PCA applied to physicochemical and colorimetric data and MIR spectra representing more than 99% of the total variance. Thus, seven (7) groups were

created before application to the FDA with hands-off crossvalidation; these groups are namely Soumbara at days 1, 5, 10, 15, 20, 25 and 30. The Partial least squares regression (PLSR) applied on the normalised MIR spectra was applied to determine the ability of the MIR to predict of water activity, moisture, and protein levels. Thus, two groups were formed: the first group designated as the calibration model was composed of 15 spectra and the second containing 6 spectra and representing all the Soumbara groups submitted to the evaluation constituted the validation model. The robustness of the model was assessed by determining the square of the correlation ratio of the standard deviation (SD) to the square root of the squared prediction error (RMSEP), referred to as the prediction deviation ratio (RPD). This ratio must be greater than 2 for good calibration. An RPD ratio less than 1.5 indicates poor predictions and the model cannot be used for further predictions (Karoui et al., 2006). The Xlstat V. 2019 2.2, and Unscrambler X 10.4 software were used to determine the ANOVA and FDA and PCA, respectively.

### **Results and Discussion**

#### *Physico-chemical Measurements*

The physicochemical analyses carried out on the Soumbara seed samples during storage time to the results mentioned in Figures 1 and 2. The moisture contents on days 1, 5, 10, 15, 20, 25 and 30 are respectively  $11.32 \pm$ 0.19.  $10.52 \pm 0.17$ ,  $10.20 \pm 0.38$ ,  $9.79 \pm 0.89$ ,  $9.4 2 \pm 0.19$ ,  $9.57 \pm 0.11$  and  $9.60 \pm 0.49$ %. These results experienced great variability throughout the storage period. For example, when the Fisher test was applied to the moisture level, a significant difference (p>0.05) was observed between the samples throughout the storage time, except for the samples at 5 and 10 days, where no significant difference was observed ( $p$ <0.05). These differences show that the storage duration significantly impacts the moisture content. These results are slightly lower with those obtained by Ibrahima et al., (2021) who found a moisture content of  $13.8 \pm 0.4\%$  in the powder of fermented seeds of *P. biglobosa*. This difference could be attributed on the one hand to the drying techniques on the one hand, and on the other hand to the migration of water throughout the storage period; since in our study, it was found that the moisture content decreased with storage duration from 9.60 to 11.32. Furthermore, our results are similar to previous work by Souley & Diadié, (2020), who found average water contents of Soumbara between  $4.61 \pm 0.21$ and  $13.61 \pm 0.34\%$ .

Regarding the protein levels, the contents increased from 27.54% on day 1 to 29.31% on day 30. These values indicate that storage duration causes a significant variation in protein levels. These variations agree with the moisture levels where significant differences were observed during the 30 days of storage. The results obtained are similar to the work of Ibrahima et al., (2021) who obtained a protein content of  $28.60 \pm 0.15$ % in the powder of fermented seeds of *P. biglobosa*. On the other hand, the protein levels found are lower than those reported by Ouoba, (2017) who indicated that the protein contents of the fermented seeds of *P. biglobosa* are between 30-40%. The authors depicted that Soumbara is an important source of protein.

As shown in Figure 2, the water activity values found remained relatively stable over the first 20 days of storage; which shows that Soumbara powders could be stable to specific alteration reactions. But at 25 and 30 days of storage away from light and at room temperature, significant differences were observed between the samples, indicating that by extending the shelf life of Soumbara, the water activity levels can vary leading to certain deterioration reactions.

The total ash contents on days 1, 5, 10, 15, 20, 25 and 30 are respectively  $3.96 \pm 0.25$ ,  $3.89 \pm 0.43$ ,  $3.97 \pm 0.23$ ,  $3.99 \pm 0.12$ ,  $4.54 \pm 0.14$ ,  $4.57 \pm 0.2$  and  $4.8 \pm 0.21\%$ . During the first 15 days of storage, no significant difference (p<0.05) was observed between the samples. On the other hand, at 20, 25 and 30 days, the samples presented significantly high values  $(p>0.05)$  indicating that the duration of storage could impact the total ash content for approximately 20 to 30 days of storage.



Figure 1. Results of the determination: (a) moisture and protein contents, (b) water activity, and (c) ash content



Figure 2. Raw mid-infrared spectra acquired on Soumbara samples on (a) days 1, 5, 10 and 15, and (b) days 20, 25 and 30 of storage at room temperature





The capital letters (A, B and C) represent the statistical differences observed for each colorimetric parameter between samples depending on storage time; number of samples (n): 7; number of repetitions: 3.

Table 2. Scatter plots of measured versus predicted water activity value; moisture content and protein content of Soumbara with full cross validation after partial least squares regression (PLSR) from mid infrared spectra.

Parameters		Calibration		Validation			
	$R^2$	<b>RPD</b>	<b>RMSEC</b>	$R^2$	<b>RPD</b>	<b>RMSEP</b>	
AW	0.99	0.23	4.00	0.99	1.02	$0.0\,$	
Moisture	0.99	l.02	0.03	0.99	.12 1.12	0.002	
Protein	0.96	1.10	0.13	0.98	1.60	0.17	

AW: Water activity; RMSEC: Root mean square error of calibration; RMSEP: Root mean square error of prediction; RPD: Ratio of prediction deviation

Table 3. Classification table of FDA with leave-one-out cross-validation for physico-chemical and colorimetric and MIR data sets of Soumbara samples during 30 days of storage.

Predited/ Observation	So01	So05	So10	So15	So20	So25	So30	Total	$%$ correct classification				
Physico-chemical and colorimetric measurements													
So01	3	$\Omega$						3	100.00%				
So05									100.00%				
So10									100.00%				
So15									100.00%				
So20									100.00%				
So25									66.67%				
So30									100.00%				
Total	Ć							21	95.24%				
Mid infrared measurements $(4000900 \text{ cm}^{-1})$													
So01	$\mathfrak{D}$							$\mathbf{3}$	66.67%				
So05									$0.00\%$				
So10									100.00%				
So15									100.00%				
So20									100.00%				
So25									100.00%				
So30									100.00%				
Total	4							21	80.95%				

So01: Soumbara on day 1; So05: Soumbara on day 5; So10: Soumbara on day 10; So15: Soumbara on day 15; So20: Soumbara on day 20; So25: Soumbara on day 25, and So30: Soumbara on day 3

Indeed, the results obtained during days 20, 25 and 30 are similar to those obtained by Akabanda et al., (2018)  $(4.36 \pm 0.14\%)$  and by Koura et al.,  $(2014)$ )  $(3.51$  and 4.39%) in the fermented seed of *P. biglobosa*.

## *Color Measurements*

The color measurements results are shown in Table 1. These results showed that the storage duration had a considerable influence the color parameters, since the Fisher and Tukey tests showed no significant difference from the point of view of lightness (*L\**) of the samples from day 15 until  $L^*$  remained relatively stable ( $p$ <0.05) at the first 15 days of storage (Table 1). These values decreased from  $42.16 \pm 0.015$  on day 1 to  $40.02 \pm 0.015$  on day 30; demonstrating variation that could be attributed to low water content and low water activity level. This drop in the value of  $L^*$  could be due on the one hand, to oxidation reactions, and on the other hand, to Maillard reactions due to the interaction between reducing sugars and nitrogen compounds. Furthermore, the levels of redness ( $a^*$ ) from day 1 to day 30 increased from 9.65  $\pm$ 0.005 to  $9.64 \pm 0.005$ . During the 30 days of storage, no significant difference ( $p > 0.05$ ) was observed between the Soumbara samples; which shows that the storage duration did not have a significant influence during the 30 days. Another explanation for this stability would be that the seeds of nere (*P. biglobosa*) underwent drying until reaching stable water levels. Regarding the *b\** value, no significant difference was observed between the samples during storage for 30 days. Considering the results of the colorimetric parameters, it turned out that only the *L\** values.

The PCA was performed jointly on the physicochemical aspects (Figure 1) to extract information from the dataset. Thus, the mapping defined by PC1 and PC2, representing 94 and 2% of the total variance (data not shown) differentiated the Soumbara samples according to their storage duration. The FDA cross-validation with Leave-One-Out was carried out on the 5 PCs of the PCA carried out on the physicochemical and colorimetric data tables to confirm this trend, and allowed to obtain a correct overall classification of 95, 24% % (Table 3). These results show that PCA and FDA can be used in a complementary manner to evaluate the differentiation of Soumbara samples during storage.

#### *Mid Infrared Spectroscopy Measurements*

Information on molecular bonds with fundamental valence vibrations of functional groups was revealed using the spectral range of 4000**–**700 cm-1 . This spectral zone makes it possible to obtain relevant information from the organic molecules present in the samples (for example, chemical constituents present at levels greater than 0.1%) (Karoui et al., 2006). As shown in Figure 2a, the MIR spectra acquired on Soumbara samples on days 1, 5, 10 and 15 are dominated by an absorption band located at approximately  $3008 \text{ cm}^{-1}$  attributable to the presence of O stretching vibrations. **–**H and N**–**H stretching, as described by Pavli et al., (2020). Intense peaks were observed in the area of 2854 and 2924 cm<sup>-1</sup>. These peaks are attributable to saturated aliphatic C**–**H stretching (Pedersen et al., 2003). This corroborates with the results found by Pebriana et al., (2017) who noted that the **–**CH2 functional group exhibits

peaks at 2924 cm-1 and 2854 cm-1 consecutively as results of asymmetric and symmetric vibrations. On the other hand, on days 20, 25 and 30, absorption bands located at  $3212 \text{ cm}^{-1}$ ,  $2920 \text{ cm}^{-1}$  and  $2854 \text{ cm}^{-1}$  were observed. These shifts show that the storage duration impacts on the molecular structure of Soumbara. This trend confirms the physicochemical results where statistically significant differences were observed between samples depending on the storage duration. Furthermore, the peaks observed at 2854 cm-1 could be due to the asymmetric and symmetric stretching of the methylene group  $(-CH<sub>2</sub>–)$  (Sari & Guntarti, 2018, Pebriana et al., 2017). These absorption bands were not affected by storage time. The peaks that appeared at 1743 and 1640 cm-1 could be attributed to carbonyl groups (C**–**O); and cis C=C stretching and stretching (Sari & Guntarti, 2018, Pebriana et al., 2017), H**–**O**–**H of the water and/or amide I group Pavli et al., (2020). These results are similar to those reported by Pebriana et al., 2017) who noted that the carbonyl group (C**=**O) of the triglyceride ester was represented by a peak at 1744/1745 cm-1 . In addition, Pebriana et al., (2017) also noted that the peak at  $1711 \text{ cm}^{-1}$  was attributed to the C=O group of the free fatty acid. Several peaks were observed in the region from 1099 to  $1516 \text{cm}^{-1}$  which may be due to C**–**H deformation of CH2, asymmetric deformation or bending of  $CH_3$ , shear or bending of  $CH_2$ , and or Amide bands III and to amines, free amino acids and C–N stretching (Sari & Guntarti, 2018, Pebriana et al., 2017). The appearance of the MIR spectra corroborates with the physicochemical results indicating that MIR spectroscopy can be used as a rapid and non-invasive method to replace physicochemical methods for evaluating storage effects on the quality of Soumbara during their marketing.

The principal component analysis (PCA) carried out on the MIR normalised spectra allowed made it possible to extract the information and the resulting similarity map of PC1 and CP3 totaling 96% of the variance, allowing to differentiate the samples into three groups depending on the storage duration **(Figure 3)**.

# *Prediction of Some Chemical Parameters from Mid Infrared Spectra*

The  $4000-700$  cm<sup>-1</sup> range was used to evaluate the predictive performance of the developed models on chemical parameters (**Table 2**). Regarding the calibration set, an excellent prediction of water activity levels (R2 =0.99, RPD =0.23 and RMSEC = 4.00 (**Table2; Figure 4a**)), and moisture ( $R^2 = 0.99$ ,  $RPD = 1.02$ , and  $RMSEC =$ 0.03) (**Table 2; Figure 4b**) was obtained. Furthermore, an excellent prediction of these parameters was also observed when considering the validation set. On the other hand, a perfect prediction of protein levels was obtained for the calibration and validation sets. One of the main conclusions of these studies was that the MIR method combined with chemometric tools could be considered a suitable technique to assess water, moisture and protein activity levels. The developed model demonstrated the potential use of MIR as a tool to predict the physicochemical composition of Soumbara during storage. Although it would be ideal to confirm these results on a large number of Soumbara, they indicate the possibility of using MIR as a rapid and excellent method to monitor the physicochemical quality of Soumbara during storage.



Figure 3. Principal component analysis similarity map applied to the spectra from of Soumbara samples during their storage





water activity value, and (b) moisture content of Soumabara samples with full cross validation after partial least squares regression (PLSR) from mid infrared spectra

#### **Conclusion**

This work shows that the Soumbara samples studied are of nutritional interest given their physicochemical composition and structural characteristics. The physicochemical analyses showed that the storage duration (30 days) has an impact on the water activity, moisture, proteins and total ash levels; since the values varied significantly depending on the storage duration. The colorimetric measurements revealed that the storage duration induced a decrease in lightness values (*L\**); the other color parameters (*a\** and *b\**) remained unchanged during storage for 30 days. The principal component analysis (PCA) and factor discriminant analysis (FDA) applied to the physicochemical and colorimetric data allowed to discriminate the Soumbara samples according to the storage time. The shape of the MIR spectra showed a slight difference between the samples depending on the storage time. The results of the application of PCA and FDA on the MIR spectra allowed to discriminate and differentiate the Soumbara samples according to the storage; while partial least squares regression (PLSR) predicted physicochemical parameters excellently/very good. However, other future studies will be carried out on a large number of Soumbara samples in order to provide more robust models. In conclusion, this study reveals that the MIR method combined with chemometrics could be used as a rapid and non-invasive method to assess the quality of Soumbara during storage.

# **Declarations**

#### *Ethical Review*

This study does not involve any human or animal testing

## *Availability of Data and Materials*

The data that support the findings of this study are available on request from the corresponding author [Sangaré M.]. The data are not publicly available because they contain information that could compromise research participant consent.

## *Declaration of Competing Interest*

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper

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#### *Conflict of Interest*

The authors declare that they have no conflict of interest

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