





EXPANDING EXPORT OF SESAME SEED AND SHEANUT/BUTTER THROUGH IMPROVED SPS CAPACITY BUILDING FOR PUBLIC AND PRIVATE SECTOR

PHYSICO – CHEMICAL CHARACTERISATION OF SESAME SEEDS IN NIGERIA Stella Denloye and Gboyega Adegboye, **NAFDAC**, Nigeria



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ABSTRACTS

Sesame seed in Nigeria were characterised at the commencement of SPS Project 172. The aggregate results obtained from the study are as reflected in Tables 1 and 2. There were two streams of sampling: 1st Stream and 2nd Stream, the results from the first stream sampling is reflected in Tables 3; while second stream results are reflected in Tables 4 and 5. However since there were issues of inadequate sampling in respect of first stream not all parameter tests were carried out.

Due to this reason, Table I principally reflects summary of the mean values from the comprehensive results obtained from 2nd Stream (Tables 3 to 4), which were utilised for characterization given in Tables 1 and 2. The brevity of Table 3 notwithstanding, the results were still presented because comparison of some tested parameters with results from 2nd stream presented useful observations highlighted under discussion. The sampling sites for sesame seeds were in Benue, Kogi, Taraba, Nasarawa and Borno states,.

The samples were tested for both quality and safety characteristics. Some of the quality characteristics that were measured include: Saponification Value, Iodine Value, Moisture Content, Ash content, Oil Content, Peroxide Value and Trace Metals and Fatty Acid Profile. The safety parameters analysed were: Total Aflatoxins, pesticides residues and aerobic mesophilic count, mould count, coliform count and *Esherichia coli*.

1.1. METHODS OF ANALYSIS

All proximate analyses for quality determination, chemical, microbiological contaminants and other characterization parameters were performed as described by the Association of Official Analytical Chemist (AOAC) 1990, 2000 and 2005; QuEchERS Method and International Standards Organization (ISO) Method. The summaries are as follows:

1.1.1. Moisture Content:

The percentage (%) moisture content was analysed by oven drying method which involves the measurement of weight loss due to evaporation according to AOAC Official methods of analysis (40.1:04, 2005).

1.1.2. Total Ash:

The total content was determined at 500-550°C according to AOAC (method 40.1.08 2005)

1.1.3. Trace Metals:

Mineral contents were determined by Atomic Absorption Spectrometer (AAS) flame method in accordance to AOAC (method 991.11, 2005).

1.1.4. Oil Content:

Soxhlet extraction method was employed for the extraction of the oil and quantified gravimetrically, according to AOAC (method 945.16, 1990)

1.1.5. Free Fatty Acid:

Titration method was applied in the determination of FFA in accordance to AOAC (method 940.28, 41.1.21, 2005)

1.1.6. Saponification Value:

Titration method was employed in accordance to AOAC (method 920.160, 41.1.18 2005)

1.1.7. Peroxide Value:

Titration method in accordance to AOAC (method 965.33, 41.1.16, 2005) was employed

1.1.8. lodine value:

The lodine value is a measure of the degree of unsaturation in fats and oils. The triglycerides of unsaturated fatty acids present in fats and oils, reacts with a definite amount of halogen (lodine). The Wij's method in accordance to AOAC (method 920.159, 2000) was employed.

1.1.9. Total Aflatoxins:

Total Aflatoxin comprising of Aflatoxin B_1 , B_2 , G_1 and G_2 was analysed according to AOAC (method 999.07, 49.2.29, 2005).

1.1.10. Pesticide Residues:

Organochlorine and organophosphate pesticides residues were analysed according to QuEchERS Method, EN15662.2007.

1.1.11. Microbiological Analysis:

Total aerobic mesophilic bacteria in sesame seed was analysed in accordance to ISO 4833:2003 while Coliform and *Escherichia coli* bacteria were analysed according to ISO 4832:2006 and ISO 16649-2:2001, respectively. Yeast and moulds were analysed according to ISO 21527-2 2008.

1.1.12. Fatty Acid Profile:

The fatty acid composition of the sesame seed samples was determined by conversion of the triglyceride esters to their more volatile methyl esters and quantified with Gas Chromatograph using Flame Ionization Detection (GC-FID) in accordance to AOAC (method 969.33- 2005).

1.2. RESULTS

Table 1:Characterisation of Sesame Seed in Nigeria at the Commencement of SPS Project 172.

s/n	PARAMETERS	EXPECTED		PROJECT COMMODITY
•			(Units)	Sesame Seed
				Range
1	Moisture ¹		(%)	2.64 - 4.61
2	Ash ²		(%)	4.36 - 8.19
3	Zinc ³		(mg/kg)	0.66 - 1.13
4	Copper ³		(mg/kg)	0.17 - 0.29
5	Calcium ³		(mg/kg)	64.52 - 82.19
6	Magnesium ³		(mg/kg)	1.53 - 1.59
7	Iron ³		(mg/kg)	0.53 - 1.64
8	Lead ³		(mg/kg)	ND
9	Cadmium ³		(mg/kg)	ND
10	Total Aflatoxin ⁴		(μ/kg)	0.5 - 4.1
11	Oil Content ⁵		> 45 (%)	51.71 - 72.35
12	FFA (as Oleic) ⁶		8 (%) max	-
13	Sapon. Value ⁷		170-190 (mgKOH/g)	187.94- 196.46
14	PeroxideValue ⁸		10 (mEq/Kg)	0.2 - 9.6
15	Iodine Value ⁹		(WIJ's)	104.25- 112.62
16	Melting Point ¹⁰		30-40 (°C)	-
17	Organochlorine ¹¹		(%)	ND
18	Organophospate ¹¹		(%)	ND
19	Aerobic Count ¹²		10000(cfu)	120-1980
20	Mould Count ¹³		500(cfu)	30-920
21	Coliform Count ¹⁴		100(cfu)	0-18000
22	E. coli ¹⁵		0(cfu)	0-0

ND: No Detection

Table 2: The Profile of Fatty Acids in Sesame Seeds

	FATTY ACIDS PROFILE ¹⁶	
1	Methyl myristate (C14:0) (%)	0.0-0.1
2	Methyl myristoleate (C14:1) (%)	ND
3	Methyl pentadecanoate (C15:0) (%)	ND
4	Methyl palmitate (C16:0) (%)	0.2-0.4
5	Methyl palmitoleate (C16:1) (%)	0.2-0.6
6	Methyl heptadecanoate (C17:0) (%)	0.0-0.1
7	Methyl stearate (C18:0) (%)	
8	Methyl oleate (C18:1) (%)	
9	Methyl linolenate (C18:3) (%)	
10	Methyl linoleate (C18:0) (%)	
11	Methyl arachidate (C20:0) (%)	0.6-0.7
12	Methyl eicosadienoate (C20:2) (%)	ND
13	Methyl eicosatrienoate (C20:3) (%)	0.0-0.1
14	Methyl eicosanoate (C20:1) (%)	0.0-0.3
15	Methyl arachidonate (C20:4) (%)	ND
16	Methyl behenate (C22:0) (%)	0.0-0.1
17	Methyl erucate (C22:1) (%)	ND
18	Methyl lingnocerate (C24:0) (%)	0.0-0.1
19	Methyl mervonate (C24:1) (%)	0.1-0.2

ND: No Detection

Table 3: Means Summary of Characteristics of Sesame by Producing States Stream 1

s/	Parameters	Expected				Sta	tes					
n		(Units)		Benue	enue Kogi		Taraba		Nasarawa		Bornu	
			Av	Range	Av	Range	Av	Range	Av	Range	Av	Range
1	Moisture ¹	(%)	3.18	2.8-3.55	3.16	2.8-3.52	3.13	2.87-3.39	3.26	2.77-3.56	3.41	3.37-3.45
2	Ash	(%)	8.75	5.51-11.99	9.27	6.63-11.9	7.39	5.84-8.94	12.34	8.10-19.34	6.82	5.74-7.90
3	Calcium ³	(mg/kg)	35.10	15.07-55.12	38.70	22.27-55.13	15.56	14.06-17.06	15.49	8.42-22.56	36.7	12.58- 60.82
4	Magnesium ³	(mg/kg)	2.83	1.73-3.93	2.8	1.73-3.87	2.62	1.96-3.27	2.73	1.84-3.61	1.94	1.79-2.08
5	Iron ³	(mg/kg)	2.32	0.31-4.33	2.38	0.42-4.33	0.53	0.31-0.74	0.3	0.26-0.34	4.99	0.28-9.69

 Table 4: Means Summary of Characteristics of Sesame Seed by Producing States Stream 2

s/n	Parameters	Expected	Benue		Kogi Taraba			Nass		Borne	Borno		
			Av	Range	Av	Range	Av	Range		Av	Range	Av	Range
1	Moisture ¹	(%)	3.37	3.05-3.59	3.29	2.81- 3.83	3.74	3.29-4.6	51	3.46	2.64-4.25	3.61	3.17-3.81
2	Ash ²	(%)	5.80	4.55-7.53	5.83	4.62- 6.64	5.58	4.81-6.4	10	6.38	4.36-8.19	6.11	5.96-6.12
3	Zinc ³	(mg/kg)	0.82	0.66-1.13	0.84	0.84- 0.95	0.89	0.71-0.9	9	0.88	0.78-0.98	0.94	0.83-1.04
4	Copper ³	(mg/kg)	0.22	0.18-0.28	0.22	0.17- 0.29	0.21	0.17-0.2	16	0,20	0.17-0.22	0.21	0.17-0.25
5	Calcium ³	(mg/kg)	76.64	70.93-80.80	76.17	67.78- 81.23	72.32	66.07-7	9.63	70.47	64.52-79.4	5 77.74	73.02-82.19
6	Magnesium ³	(mg/kg)	1.56	1.55-1.57	1.55	1.54- 1.57	1.55	1.53-1.5	66	1.55	1.54-1.57	1.57	1.56-1.59
7	Iron ³	(mg/kg)	1.16	0.9-1.68	1.22	0.76- 1.48	0.88	0.53-1.1	.7	1.49	1.20-1.64	1.14	0.94-1.45
8	Lead ³	(mg/kg)	ND		ND		ND			ND		ND	
9	Cadmium ³	(mg/kg)	ND		ND		ND			ND		ND	
10	Total Aflatoxin ⁴	(μ/kg)	1.43	0.80-2.50	1.06	0.9- 1.13	1.0	0.70-1.5	60	8.0	0.5-1.0	2.53	0.80-4.1
11	Oil Content ⁵	> 47.5 (%)	59.30	53.88-72.35	54.61	52.10- 57.03	57.06	54.47-5	9.60	55.47	51.71-60.78	3 56.48	55.59-57.50
12	Sapon. Val ⁷	187-197 (mgKOH/g)	194.8	187.94- 196.35	193.90	187.94- 196.36	191.09	187.94-	191.09	193.08	190.74- 194.95	95.48	193.55- 196.14
13	PeroxideValue ⁸	10 (mEq/Kg)	0%	0.2-4.8	0%	0.8-2.6	0%	1.2-8.6		0%	4.2-6.8	0%	4.2-9.6
14	Iodine Value ⁹	104-120 (WIJ's)	110.51	107.07- 112.62	111.63	110.24- 112.62	104.56	104.51-	104.69	105	104.51- 105.25	105.07	104.25- 106.28
15	Organochlorine ¹¹		ND	ND			ND		ND		ND		
16	Organophosphate ¹¹		ND	ND			ND		ND		ND		
17	Aerobic Count ¹²	10000 cfu		260-	760		120- 840		680- 1930		1200- 1820		580- 1980
18	Mould Count ¹³	500 cfu	83%	80-5	80	100%	190- 780	100%	120- 920	67%	30-820	100%	230- 730
19	Coliform Count ¹⁴	100 cfu	67%	0-35	00	100%	180- 3700	100%	12000- 14000	100%	960- 18000	100%	1570- 5200
20	E coli 15	0 cfu	0	0			0		0		0		

ND: No Detection; %*: Percentage Occurrence

Table 5: Profile of Fatty Acids 16 of Sesame Seeds by State

SESAME	Methyl	Methyl	Methyl	Methyl	Methyl	methyl	Methyl	Methyl	Methyl
	myristate	myristoleat	pentadecano	palmitate	palmitoleate	heptadecano	stearate	oleate	linolenate
	(C14:0)	e (C14:1)	ate (C15:0)	(C16:0)	(C16:1)	ate (C17:0)	(C18:0)	(C18:1) (%)	(C18:3) (%)
	(%)(<0.5)	(%)	(%)	(%)(7.0-12)	(%)(0.5)	(%)	(%)		
STATE									
BENUE	0.1	0.0	0.0	0.2	0.3	0.0			
KOGI	0.1	0.0	0.0	0.2	0.2	0.1			
TARABA	0.1	0.0	0.0	0.4	0.6	0.1			
NASARAWA									
BORNU									
Continuatio	Methyl	Methyl	Methyl	Methyl	Methyl	Methyl	Methyl	Methyl	Methyl
n	arachidate	eicosadien	eicosatrienoa	eicosanoate	arachidonat	behenate	Erucate	lingnocerat	nervonate
	(C20:0)	oate	te (C20:3) (%)	(C20.1)%(<0.	e (C20:4)	(C22:0)	(C22:1)	e (C24:0)	(C24:1) (%)
	(%)(<1.0)	(C20:2) (%)		5)	(%)	(%)(<0.5)	(%)	(%)	
STATE									
BENUE	0.7	0.0	0.1	0.3	0.0	0.1	0.0	0.1	0.2
KOGI	0.6	0.0	0.1	0.2	0.0	0.1	0.0	0.1	0.1
TARABA	0.7	0.0	0.0	0.0	0.0	0.1	0.0	0.1	0.1
NASARAWA									
BORNU									

1.3. DISCUSSION

The aggregate results obtained from the study are as reflected in Tables 1 and 2. There were two streams of sampling: 1st Stream and 2nd Stream, the results from the first stream sampling is reflected in Tables 3; while second stream results are reflected in Table 4, the result of the fatty acid profile by state is reflected in Table 5.

Tables 1 and 2 principally reflect summary of the mean values from the comprehensive results obtained from both streams of sampling (Tables 3 to 5), which were utilized for characterization. The incompleteness of Table 3 notwithstanding, the results were still presented because comparison of some tested parameters with results from 2nd stream presented useful observations.

The samples were tested for both quality and safety characteristics. Some of the quality characteristics that were measured include: saponification value, iodine value, moisture content, ash content, oil content, melting point, free fatty acid, peroxide value and levels of trace metals. The safety parameters analyzed were: total aflatoxins, pesticides residues and microbial contamination.

Results based on Tables 1 and 2 indicate the following:

1.3.1. Moisture Content:

The percentage (%) moisture level of the samples ranged between 2.64 to 4.61 with an average of 3.2 % for sesame seed samples from the five producing States.

1.3.2. Ash content:

The ash content of the sesame seeds ranged from 4.36 to 8.19% between the producing States, giving an average of 5.93%. The average % ash content value within each of the 5 producing states also do not differ significantly indicating that the level of inorganic components of the soil are very similar.

1.3.3. Trace metals:

The levels of trace and heavy metals obtained across the five producing states of sesame seeds were similar. Analysis showed the following results: Zinc (Zn) content ranged from 0.66-1.13 mg/kg with a mean value of 0.87%; Copper (Cu) content ranged from 0.17 to 0.29mg/kg with a mean value of 0.21%.; Calcium (Ca) content ranged from 64.52 to 82.19mg/kg with a mean value of 74.67%; Magnesium (Mg) ranged from 1.53 to 1.59 with a mean value of 1.55%; Iron (Fe) ranged from 0.53 to 1.64mg/kg with a mean value of 1.18%; while Lead (Pb) and Cadmium (Cd) were not detected in any of the sesame seed samples from the producing states.

1.3.4. Oil Content:

The oil content obtained from sesame seed ranged from 51.71 to 72.35%, falling within the expected oil content of more than 45%.

1.3.5. Free Fatty Acid:

The level of FFA is a degradation/spoilage indication of the products. The permissible maximum level of free fatty acid s is 8% (as Oleic), above which the oil or fat is rendered unpalatable. However this is not applicable to oil extracts of the sesame seed samples across the producing states, indicating proper storage practices. (Table 1: 12)

1.3.6. Peroxide Value:

This is also a storage, degradation and rancidity factor. The peroxide value results ranged from 0.2 to 9.6mEq/kg in sesame seed, (the expected peroxide value-10mEq/kg). (Table 1: 14)

1.3.7. Iodine value:

The test value ranged between 104.25 and 112.62 Wij's in the oil extracts from sesame seed across the producing states, falling within the expected values of 104-120Wij's from sesame seeds from other parts of the world, (Table 1:15). Iodine value is a measure of the degree of unsaturation and a useful parameter for identifying oils.

1.3.8. Saponification value:

This value gives an indication of the proportion of lower fatty acid esters present in the oil/fat. The higher the value of alkali needed to neutralize the fatty acid the lower the molecular weight of the fatty acid esters in the oil or fat. Table 1:13indicates the saponification value of the oil extracts of the sesame seeds which ranged from 187.94 to 196.46mgKOH/g of oil extract (expected-170 to 190mgKOH/g.)

1.3.9. Total Aflatoxins:

A 100% occurrence of Total Aflatoxins was observed in all the samples. The total Aflatoxins detected in the sesame seeds from the five producing states ranged from 0.5 to 4.1ug/kg (Table 1:10), the mean values of total aflatoxins were however below the Maximum Residue Limit of 10ug/kg.

1.3.10. Pesticide Residues:

Organochlorine and organophosphate pesticides residues were not detected in any of the products. This confirms natural cultivation of sesame in Nigeria. This scenario may be further explored in establishing natural or organic production claims for the crop. (Table 1: 17&18).

1.3.11. Microbiological Analysis:

The total aerobic mesophilic bacteria count in sesame seed ranged from 120 to 1980 colony forming units (cfu), the expected limit being 10000cfu (Table 1:19). About 67 to 100% of the total samples had a high mould count with the value ranging between 30 to 920cfu. (Table 1:20). The maximum limit for mould count is 500 cfu.

Coliform Count in all the sample sites returned positive coliform count with some in excess of the safe limit (100cfu max.). This is an indication of serious microbial contamination, with the maximum count at 18000 (Table 1:21). *E.coli* which is indicative of feacal contamination, was however not detected in any of the products (Table 1:22).

The relatively high levels of microbial contamination recorded for sesame seeds in some states may be indicative of poor hygienic practices during harvesting, processing and storage conditions which can be improved upon.

1.3.12. Fatty acid profile:

The composition of fatty acids in the sesame seed is outlined in Table 2. The dominant fatty acid in sesame seed are: Methyl arachidate (C20:0) 0.6-0.7% and Methyl palmitoleate (C16:1) 0.2-0.6% (Tables 2 and 5).

1.4. OBSERVATIONS

While the inadequacy of samples did not afford detailed analyses of the First Stream Samples, the results of total aflatoxins tests captured in both Steams 1 and 2 present an interesting result. The expectation is that with sampling procedure given to the Field Officers, lower records of Aflatoxin incidences would be expected in the second stream. The contrary was the case; lower percentages of incidences (20%, 66%, 66% and even ND in one sample) were recorded in Steam 1, while in Stream 2, 100% incidences were recorded throughout. The advent of raining season could be a contributive factor. This is bearing in mind that the first set of samples were taken in April and submitted early May, while the second set of samples were taken in late June at the peak of rain. The probable use of polluted source of water could be a reason.

The microbial results in the second stream samples (Table 4) when compared with first stream (Table 3) showed that season of sampling may play a significant role in characterizing safety parameters of sesame seed. Nigeria has basically two climatic seasons: dry and wet season. First Stream (Table 3), which featured lesser microbial count was sampled during dry season and Second Stream was sampled during wet season. However the inorganic parameters remain fairly constant. This shows the importance of good handling practices and timing in establishing good qualities of sesame Nigeria.

1.5. REFERENCES:

- ¹ AOAC. Official Methods of analysis (method page 1, 40.1:04, 2005)
- ² AOAC. Official Methods of analysis (method 40.1.08 2005)
- ³ AOAC. Official Methods of analysis (method 991.11, 2005)
- ⁴ AOAC. Official Methods of analysis (method 999.07, 49.2.29, 2005)
- ⁵ AOAC. Official Methods of analysis (method 945.16, 1990)
- ⁶ AOAC. Official Methods of analysis (method 940.28, 41.1.21, 2005)
- ⁷ AOAC. Official Methods of analysis (method 920.160, 41.1.18 2005)
- ⁸ AOAC. Official Methods of analysis (method 965.33, 41.1.16, 2005)
- ⁹ AOAC. Official Methods of analysis (method 920.159, 2000)
- ¹⁰ AOAC. Official Methods of analysis (method 920.157, 41.1.09, 2005)

¹¹ QuEchERS Method, EN15662.2007

¹² ISO 4833:2003

¹³ ISO 21527-2 2008

¹⁴ ISO 4832:2006

¹⁵ ISO 16649-2:2001

¹⁶ AOAC. Official Methods of analysis (method 969.33-2005)

APPENDIX

NATIONAL AGENCY FOR FOOD AND DRUG ADMINISTRATION AND CONTROL (NAFDAC)

PROVISIONAL SAMPLING GUIDE FOR CHARACTERISATION OF SESAME SEEDS AND SHEANUTS/BUTTER

(Instructions for taking, preparing and packaging the samples)

Preamble: The purpose for this sampling guide is to assist the field inspectors and other ad hoc staff in the WTO STDF Project 172 on Sesame seed and Sheanut/Butter in Sampling, Packaging, Handling and Safe Delivery of Samples to the testing laboratory(ies) for the characterization study.

Sampling Size: 500g to 1 Kg

Packaging: Preferably wide mouthed glass bottle with screw cap.

Labelling: The sample bottle must be labeled containing the following information:

Name/Type of Sample:

Sampling Location:

Sample No/Code:

Time of collection:

Temperature at collection:

Humidity at collection:

Sampling By: (Name and Signature)

Date of sampling: Sample sent thru: Date of Dispatch

Transportation: By road, air, rail, etc.

Storage Conditions: Dry, Cool, avoid excessive heat.