Oil Content and Fatty Acids Composition of Cookies Produced from Blends of Tigernut and Wheat Flour

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ABSTRACT---- Tigernut is an underutilized crop but only recently its demand has increased tremendously because of its values. Composite flours were formulated from wheat and tigernut flour mixed in the ratios 100:0, 0:100, 80:20, 70:30 and 50:50. Cookies were made from the composite flour samples and analyzed for sensory composition. The fat content of the cookies were extracted and evaluated for iodine value, free fatty acids, saponification value and fatty acid composition. The fatty acid composition was analysed by gas chromatography–spectroscopy. The iodine value, FFA and saponification values varied from 20.3 – 32 gI/g, 0.55 -0.82% and 27.49 - 67.04 mgKOH/g respectively. The FAMEs present were, C17:0, C18:0, C18:1n9, C18:3n6, C20:3n6, C22:1. The total SFAs, MUFAs and PUFAs determined for each samples in the range of 33-49%, 46-59% and 5-8.1% respectively. Both decrease and increase in the amount of SFAs, MUFAs and PUFAs were observed. Sensory evaluation reveals that the taste and texture of the cookies made from wheat flour substituted with 20% tigernut flour was most preferable. The quantity and quality of the lipid fraction of the cookies indicated that all analysed cookies are a good source of SFAs, MUFAs and PUFAs consequently posing no threat to the health of the consumers.

Keywords---- Wheat, tigernut, cookies, fatty acid composition

PRACTICAL APPLICATIONS

Tigernut as an underutilized crop is high in dietary fibre content which could be effective in the treatment and prevention of many diseases including colon cancer, coronary heart diseases, obesity, diabetics and gastrointestinal disorders. Tigernut flour has been demonstrated to be a rich source of quality oil and contains moderate amount of protein. The blend of tiger nut with wheat will provide a traditional nutritious food to more people at lower cost and to utilize indigenous crops to a greater extent and also it will improve the nutritional quality and health benefits of cookies produced from tiger nut.

1. INTRODUCTION

Cookies are popular examples of bakery product of ready-to-eat snack that possess several attractive features including wide consumption, more convenient with long shelf-life and have the ability to serve as vehicles for important nutrient (Ajibola et al., 2015). A cookie is a baked or cooked food that is small, flat and sweet. They are one of the best known quick nutritive snack products produced from unpalatable dough that is transformed into appetizing product through the application of heat in an oven (Farheena et al., 2015; Olaoye et al., 2007).

A cookie usually contains flour, sugar and some type of oil or fat. The flour can be wheat based and part of the wheat can also be substituted with other starchy foods. The idea of substituting part of wheat with other starchy crops is not new. Several institutions, including FAO, have carried out research designed to find ways of partially substituting wheat flour with other sources of flour or replacing wheat altogether (Bokanga, 1995). Different levels of success have been recorded with the use of flours from legume, cereals, roots and tubers in baked goods (Kure et al., 1998; Dhingra and Jood, 2002; Basman et al., 2003). With the progressive increase in the consumption of cookies and other baked products in many countries, composite flour programme, especially in developing countries, has the potential to conserve foreign exchange, provide nutritious food to more people at affordable cost and widen the utilization of indigenous crops in food formulation.

Tigernut (Cyperus esculentus), an underutilized crop, was reported to be high in dietary fibre content, which could be effective in the treatment and prevention of many diseases including colon cancer, coronary heart diseases, obesity, diabetics and gastro intestinal disorders (Anderson et al., 1994). It has been reported to be a rich source of good quality oil (Dubois and et al., 2007; Yeboah et al., 2012) and contains a moderate amount of protein (Oladele and Aina in a 2007). It is also an excellent source of some useful minerals such as iron and calcium which are essential for body growth and development (Oladele and Aina, 2007) as well as vitamins E and C (Belewu and Belewu 2007).
Therefore, tigernut, with its inherent nutritional and therapeutic advantage, could serve as a good alternative in composite flour for the baking industry. Various works on the evaluation and use of tigernut flour as composite flour in the production of baked products such as bread, biscuit, and other snacks (Awolu et al., 2017; Ezeocha et al., 2016; Ahmed et al., 2014ab; Ade-Omonye et al., 2008) had been reported. Information on the evaluation of oil from tigernut baked products is however scanty especially since it’s a good source of quality oil. The quality of fat plays a very important role in food processing technology. The main reason for the deterioration in the quality of foods is fat oxidation and it can directly affect qualities like flavor, color, texture, nutritive value, and safety of food. Thus, this study will evaluate the oil content and fatty acids composition of cookies produced from blends of tigernut and wheat flour.

2. MATERIALS AND METHODS

Dry tigernut, wheat flour, and other principal ingredients like flavor, milk, egg, salt, margarine sugar, and baking powder were obtained from the market. All chemicals used were of analytical grade.

Preparation of tigernut flour

Preparation of tigernut flour was according to the method of Adeyemi (1988). The dry tigernuts were sorted to remove unwanted materials like stones, pebbles, and other foreign seeds before washing with tap water. The cleaned nuts were dried in the cabinet dryer at 60°C for 24hrs to low moisture content. The dried nuts were milled to tigernut flour, sieved through 600µm aperture size and the resultant flour was packed and sealed in polythene bags until it was used.

Preparation of the various flour blends

Flour blends of wheat flour and tigernut flour were prepared by mixing required amount of respective flours in the following ratio - 100:0, 80:20, 70:30, and 50:50.

Table 1: Formulation ratio of wheat and tigernut composite flour

| Sample          | A  | B  | C  | D  | E  |
|-----------------|----|----|----|----|----|
| Wheat flour     | 100| 0  | 80 | 70 | 50 |
| Tigernut flour  | 0  | 100| 20 | 30 | 50 |

Production of cookies with wheat flour/tigernut blend

Production of the cookies was done according to the modified method of Bello et al., 2018. Measured sugar and butter were mixed together until creamy. The egg was then added and continued mixing together until light and fluffy. Flour, baking powder, and vanilla essence were added and mixed in a bowl to form elastic dough. The dough was rolled and flattened into a uniform thickness of about 3.5mm before cutting out to shapes using a hand cutter. The cut out dough was baked in the oven at 180°C for 10-15 minutes in the oven. After baking, the cookies were cooled to room temperature, packed in low density polyethylene (LDPE) bags, and sealed in a plastic transparent container.

Table 2: Ingredients Formulation for Cookies Production

| Ingredients                | Sample code | A  | B  | C  | D  | E  |
|----------------------------|-------------|----|----|----|----|----|
| Flour (g)                  | 200         | 200| 200| 200| 200|    |
| Margarine (g)              | 60          | 60 | 60 | 60 | 60 |    |
| Sugar (g)                  | 50          | 50 | 50 | 50 | 50 |    |
| Baking powder (g)          | 1.8         | 1.8| 1.8| 1.8| 1.8|    |
| Vanilla essence (g)        | 6.25        | 6.25|6.25|6.25|6.25|    |
| Egg (ml)                   | 30          | 30 | 30 | 30 | 30 |    |

Source: Adapted from Bello et al., 2018
**Determination of Physico-chemical Properties of oil from cookies**

Sample Preparation for analysis
Samples were ground into crumbs by a pestle. Total lipids were determined (extracted) by Soxhlet extraction, using petroleumether as described by AOAC (2012) method.

Method for Determination Iodine Value.
Five grams of oil Samples was weighed in 250mL conical flasks and then 25mL of carbon tetra chloride was added to each oil sample and content was mixed well. 25mL of Hanus reagent was added to the solution, swirled for proper mixing, and kept in the flask in dark for half an hour. After standing, 15mL of potassium iodide solution was added and then 100mL of distilled water was added into the mixture and 1mL starch indicators solution was added to the sample solution. Then, liberated iodine was titrated with 0.01N of sodium thiosulphate solution; then, at the end, blue color was formed and then disappeared after thorough shaking. The blank determination was carried in the same manner as test sample but without oil. The iodine value was estimated using the following formula:

\[
\text{Iodine value} = \frac{(b - a) \times N \times 1.269 \times 100}{W}
\]  

(1)

where \(b\) is blank titre value,
\(a\) is sample titre value,
\(N\) is normality of thiosulphate, and
\(W\) is weight of sample (A.O.A.C 2000).

Method for Determination of Free fatty acid.
Three grams of each oil extracted from the samples was weighed in 250mL of conical flasks and 30mL of freshly neutralized ethyl alcohol (ethanol) was added to the samples and then shaken well to dissolve sample. The sample solution was boiled for about five minutes and cooled and then 1mL of phenolphthalein indicator was added to the sample solution. The sample solution was treated with 1N sodium hydroxide solution until permanent pink colour appeared. The free fatty acid value was estimated using the following equation:

\[
\text{Free fatty acid} = \frac{2.82 \times V \times 100}{W \times 1000 \times 4}
\]  

(2)

where \(W\) is weight of oil that equals 3 grams,
\(V\) is titre value of 1N NaOH, and
2.82 is equivalent weight of oleic acid (ISI Hand book of food Analysis, 1984)

Method for Determination of Saponification Value.
Two grams of each oil sample was weighed in 250mL Erlenmeyer flasks; then 25mL of alcoholic potassium hydroxide solution was added into the flasks. The blank determination was conducted along with the sample. The samples flask and the blank flask were connected with air condensers and boiled gently in the water bath, steadily until the saponification was completed, indicated by absence of oily matter and the appearance of clear solution. Clarity was achieved in half hour boiling. After the flask and the condenser cooled, inside of the condensers was washed down with about 10mL of ethanol and then 1mL of phenolphthalein indicators was added to the solution. Excess potassium hydroxide was titrated with 0.5N hydrochloric acid until cloudy solution was formed. The saponification value was estimated using the following equation:

\[
\text{Saponification Value} = \frac{56.1 \times (b - a) \times N}{W}
\]  

(3)

where \(W\) is weight of sample that equals 2 grams,
\(b\) is blank titre value,
\(a\) is sample titre value, and
\(N\) is 0.5 normality of HCl (A.O.A.C, 2000).

**Fatty Acid Profile.**
The fatty acid profile was determined as fatty acid methyl esters by gas chromatography-mass spectrometry. The methyl esters were prepared using the AOCS method (AOCS, 1997). The separation of fatty acid esters was performed on a Buck Scientific Gas Chromatograph GC (model 910) with a Restek RTX-2330 capillary column (30m × 0.25mm × 0.2 mm). The column temperature was programmed at 120°C for 10 min, then increased to 240°C at 5°C/min with a final isothermal period of 60 min. Hydrogen was used as carrier gas with constant linear velocity of 25 cm/sec–1. The injector temperature was set at 250°C, with a split ratio of 1 : 10. The flame ionization detector temperature was 250°C. Fatty acid
methyl esters (FAMEs) were identified by comparison of retention times with authentic standards (Supelco 37 comp. FAME mix 10 mg/mL in CHCl₃), and quantification was performed by the internal normalization method.

**Sensory analysis**
The organoleptic properties of the cookies including: Taste, Colour, Texture, Flavor and Overall acceptability acceptance were assessed by a 25 member panelists screened among students, who were instructed regarding the evaluation procedures in both written and verbal formats prior to the cookies evaluation. Each panelist was given the cookies sample to taste and compare.

3. **STATISTICAL ANALYSIS**
The results of the experiment was subjected to analysis of variance (ANOVA) and the mean was separated with the use of Duncan’s multiple range test to detect significant difference (p<0.05) among the sample values using the statistical package for the social science (SPSS).

4. **RESULTS AND DISCUSSION**

**Physico-chemical properties of oils extracted from the cookies**
Some of selected physico-chemical properties such as iodine value, free fatty acid value and saponification value of oils extracted from the cookie samples analyzed are presented in Table 3. Iodine value is defined as the gram of iodine absorbed per 100g sample and it is a measure of degree of unsaturation (C=C) with respect to the amount of fat or oil. Iodine value therefore, is used to characterize fat and oil and to follow the hydrogenation process in refining. It can also be viewed as a sign of lipid oxidation, this is because there is a decline in unsaturation during oxidation (Pomerenz and Meloan, 1987). Hence, the higher the iodine value the greater the degree of unsaturation. It is equally important due to the fact that it gives the extent to which the lipid sample can be prone to oxidation and thus become rancid. The iodine value varied from 20.3 – 43.2 gI/100g with sample prepared from 100% weight flour (A) having the lowest and samples prepared from 100% tigernut flour (B) having the highest. It was observed that the higher the inclusion of tigernut flour, the higher the iodine value. The higher iodine value of tigernut based cookies indicated a high level of unsaturation. The iodine content in all the samples is lower than those reported in literature. For instance values of 107 was reported by Arshad et al., (2008) for wheat germ oil, value of 87.08 for tigernut oil (El-Naggar, 2016) and reference value for palm oil which is 53.8 (Abdulkadir and Jimoh, 2013). The results showed that the oil present in the cookies pose no significant health risk to consumers. The result also reveals that all samples contain unsaturation within the standard specified in literatures and that possibility of spoilage due to oxidation is minimal.

Free fatty acid (FFA) content is one of the main criteria for checking the quality of oil. This varied from 0.55 - 0.82%, the highest amount of FFA, 0.82% was observed in sample with 100% tigernut and the lowest was observed in sample with 30% tigernut and 70% wheat flour. These levels were comparable to the reported values (0.2-1.4%) for Italian biscuits (Caponioet al., 2006). Values of 27.3%, 0.22% and 2.12 had been reported for wheat germ oil, tigernut oil and palm oil respectively (Arshad et al., 2008; El-Naggar, 2016; Abdulkadir and Jimoh, 2013). Since fats and oils are triglycerides, the free fatty acids should be very low in highly graded lipid sample. Free fatty acid in the oil sample can also be used as an indicator for the age of the oil and as one of important quality attributes measurements (Muhammad et al., 2011 and Belewu and Belewu, 2007). The level of FFA in the samples indicates a moderate level of oil hydrolysis. Free fatty acids (FFA) are produced by the hydrolysis of oils and fats. The level of FFA depends on time, temperature and moisture content because the oils and fats are exposed to various environments such as storage, processing, heating or frying. Since FFA are less stable than neutral oil, they are more prone to oxidation and to turning rancid. Thus, FFA is a key feature linked with the quality and commercial value of oils and fats (Mahesar, 2014). The saponification value of the sample ranges from 27.49-67.04mgKOH/g. The lowest is sample with 30% tigernut and 70% wheat flour with 27.49. Saponification number gives information concerning the character of fatty acids, such as the longer the carbon chain of the fat the less acid is liberated per gram of fat hydrolyzed. Also, it is considered as a measure of the average molecular weight of all the fatty acids present. According to the results, the saponification number is low, which is an indication they contain high amounts of long chain fatty acids and therefore a high molecular weight. Values of 207, 192.88 and 191.3 mgKOH/g had been reported for wheat germ oil, tigernut oil and palm oil respectively (Arshad et al., 2008; El-Naggar, 2016; Abdulkadir and Jimoh, 2013).
Table 3: Physico-chemical properties of oils extracted from the cookies

| SAMPLE | Iodine g (gI/100goil) | Free Fatty Acid | Saponification (mgKOH/g) |
|--------|-----------------------|----------------|-------------------------|
| A      | 20.3±0.31             | 0.64±0.02      | 30.57±0.05              |
| B      | 43.2±0.32             | 0.82±0.02      | 67.04±0.29              |
| C      | 27.9±0.25             | 0.69±0.03      | 49.65±0.02              |
| D      | 29.0±0.25             | 0.55±0.01      | 27.49±0.03              |
| E      | 32.0±0.41             | 0.59±0.01      | 62.27±0.04              |

Note: Values are reported as the average or mean value of the properties

100%W - 0%T = sample A
0%W – 100%T = sample B
80%W – 20%T = sample C
70%W – 30%T = sample D
50%W – 50%T = sample E

Fatty acids composition of oils extracted from the cookies

The chromatograms of the fatty acids methyl esters (FAME) obtained from oil extracted from cookies made from tigernut and wheat flour blends are shown below (Figure 1). Table 4 shows the fatty acids methyl esters (FAMEs) in the five samples. The saturated fatty acids (SFAs) in the analysed samples were margaric acid (C17:0) and stearic acid (C18:0), while the monounsaturated fatty acids (MUFAs) present were oleic acid (C18:1n-9) with a small quantity of erucic acid (C22:1), the polyunsaturated fatty acid (PUFA) present were linolenic acid (C18:3n-6) and small quantity of dihomo-γ-linolenic acid (C20:3n-6). The fatty acids present in sample A (100%W-0%T) changed as part of the wheat was substituted with tigernut flour in different ratios.

Comparing sample A with sample C which was 80%WF-20%TF, C17:0 was constant, C18:0 (SFA) decreased from 48%-42%, C18:1 (MUFA) increased from 46%-51% while C18:3 (PUFA) also increased from 5%-6%. In sample D which was 70%WF-30%TF, C17:0 was constant that is no reduction no increment. C18:0 decreased from 48%-41%, C18:1 increased from 46%-52% and C18:3 increased from 5%-6%. In sample E which was 50%WF-50%TF, C17:0 remained constant (1%), C18:0 decreased from 48%-38%, C18:1 increased from 46%-54%, C18:3 increased from 5%-6%. This showed that as the wheat flour decreases, the saturated fatty acid (C18:0) decreases in all the samples while the unsaturated fatty acids (MUFAs and PUFAs) increases.

From the result, sample B which was 0%WF-100TF has the following fatty acids; C17:0-1%, C18:0-32%, C18:1-58%, C18:3-7.9% with little quantity of C20:3-0.2% and C22:1-1%. In comparing sample B to sample A (100%WT-0%TF), it showed that the C18:0 in A is greater than B, 48%-32% respectively. But C18:1 in B (58%) is greater than that of A (46%) and also for C18:3, 7.9%-5% while B has additional C20:3 in 0.2% and C22:1 in 1%, and these showed that the saturated fatty acid in sample B is lower than the one in A and the unsaturated fatty acids in B is greater than the one in A. Comparing samples C, D and E to sample B, it was detected that as the tigernut flour decreases SFAs increases while (Unsaturated Fatty Acids) UFAs decreases.

Aro et al. (1997) have suggested that fatty acids with a chain length of C12:0-C16:0 are atherogenic. Oleic and poly unsaturated fatty acids on the other hand have a lipid lowering effect. Among the mono unsaturated fatty acid, the major contributor was oleic acid (C18:1n9) in all the cookies samples except in sample A. The maximum amount of oleic acid was determined in sample B and minimum amount in sample A. While comparing sample A with other samples it was indicated that saturated fatty acid (C18:0) decreases, while unsaturated fatty acid majorly MUFAs (C18:1) increases. This suggested that inclusion of tigernut flour pose no threat to the health of the consumers but rather have health benefit.
Figure 1: The chromatograms of the fatty acids methyl esters (FAME) of samples

Table 4: Fatty acid composition of the cookies.

| Fatty acid compounds | A(100%W – 0%T) | B(0%W – 100%T) | C(80%W – 20%T) | D(70%W – 30%T) | E(50%W – 50%T) |
|----------------------|----------------|----------------|----------------|----------------|----------------|
| C17:0                | 1              | 1              | 1              | 1              | 1              |
| C18:0                | 48             | 32             | 42             | 41             | 38             |
| C18:1                | 46             | 58             | 51             | 52             | 54             |
| C18:3                | 5              | 7.9            | 6              | 6              | 6              |
| C20:3                | -              | 0.2            | -              | -              | 1              |
| C22:1                | -              | 1              | -              | -              | -              |
| SFA                  | 49             | 33             | 43             | 42             | 39             |
| MUFAs                | 46             | 59             | 51             | 52             | 54             |
| PUFAs                | 5              | 8.1            | 6              | 6              | 7              |

KEYWORDS
C17:0 → Methylheptadecanoate
C18:0 → Methylstearate
C18:1 → Methyloleate
C18:3 → Methylpalmitoleate
C20:3 → Methylhexacosatrienoate
C22:1 → Methylerucate
SFA → Saturated Fatty Acid
MUFAs → Mono-unsaturated Fatty Acids
PUFAs → Poly-unsaturated Fatty Acids
Sensory Analysis
The result of the sensory evaluation (Table 5) indicated that all the samples had appreciable ratings for colour, flavour, taste, texture, and general acceptability. The taste and texture of the cookies made from wheat flour substituted with 20% tigernut flour was most preferable while the colour, flavour and overall acceptability of cookies made from purely wheat flour was more preferred and was most generally accepted.

| Sample | Colour | Flavor | Taste | Texture | Overall acceptability |
|--------|--------|--------|-------|---------|----------------------|
| A      | 7.58±1.77 | 7.13±1.48 | 6.96±1.68 | 6.83±1.66 | 7.46±1.22          |
| B      | 6.04±2.16 | 6.63±1.74 | 7.17±1.71 | 6.21±1.64 | 7.25±1.45          |
| C      | 6.42±1.35 | 7.04±1.23 | 7.21±1.22 | 6.88±1.30 | 7.13±1.12          |
| D      | 6.71±1.60 | 6.79±1.14 | 6.96±1.63 | 6.83±1.49 | 7.17±1.44          |
| E      | 5.83±1.66 | 6.00±1.62 | 6.17±1.40 | 6.33±1.37 | 6.04±1.20          |

Values are reported as the mean ± standard deviation of the replications
Means followed by the same letter within a column indicate significant difference (p < 0.05)

5. CONCLUSION
This study has shown that nutritious and acceptable cookies with health benefits can be produced from composite flour made from wheat and tigernut flour blends in terms of the quality of oil present in the cookies which was indicated by iodine value, saponification value, free fatty acids value and fatty acid composition of the cookies. This study suggests the use of tigernut flour inclusion in wheat flour provides the required fatty acids at appropriate level. Also, the use of such composite flour could reduce dependency on wheat flour which is usually imported into Nigeria.

6. REFERENCES
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