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Rheological and Physicochemical Characteristics of Table-Grade Margarine Produced from Interesterified Blend of Allanblackia (*Allanblackia floribunda*), African Walnut (*Tetracarpidium conophorum*) and Fluted Pumpkin (*Telfairia occidentalis*) Seed Oils

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Abstract

The objective of this work was to evaluate the rheological and physicochemical properties of table-margarine produced from interesterified oil blend. ***.*** *Oils were extracted using hot water floatation methods, blended in the ratio of 60:20:20 (Allanblackia: African walnut: fluted pumpkin seed oil) and interesterified at optimum temperature, time and catalyst concentration of 74.6°C, 46min and 0.28%, respectively. The consistency of non-interesterified table margarine with 60 % fat base was significantly (p<0.05) higher (0.14 cm/s). Commercial table margarine (control) showed significantly(p<0.05) higher spreadability (33.00 g.cm/s) and viscosity (29.86 cSt). Table-margarine produced with the interesterified oils and non-interesterified oils gave melting point ranging from 32.00 to 33.00 °C. Melting point of the commercial table-margarine was significantly (p<0.05) higher (37.00 °C). Interesterified and non-interesterified margarine containing 60 – 70 % fat base received same overall acceptability with those of the commercial table margarine. Use of Allanblackia, African walnut and fluted pumpkin seed oils for table-based margarine production is highly recommended, sequel to their high content of polyunsaturated fatty acids (omega-6 and omega-3) which have cholesterol lowering properties.*

Keywords: *Table-Margarine, Rheological Properties, Vegetable oils, Acceptability.*

1.0 Introduction

Margarine is a flavored food product containing 80 % fat (O'Brien, 2009). It is made by blending selected fats and oils with other ingredients and is fortified with vitamin A to produce a table, cooking, or baking fat product that serves the purpose of dairy butter but is different in composition and can be varied for different applications (O'Brien, 2009). Margarine was developed to fill both economic and nutritional need when it was first made as a butter substitute. Margarine and spreads are considered water-in-oil emulsions consisting of a fat phase that usually includes oils, emulsifiers, oil-soluble flavors, and nutrient such as vitamins. The water phase consists of water, salt, proteins, starches and/or gums, and acid (Strayer *et al*., 2006). The basic steps involve in table margarine and spreads processing include; mixing, emulsifying, chilling, working, and resting

(Morlok, 2010). Margarine and spreads manufacturing processes can be summed up as preparing the fat-phase, aqueous-phase, addition of emulsifiers, blending, chilling, and tempering (when necessary) (Morlok, 2010). When blending, it is important to add the aqueous phase to the fat-phase in order to promote water-in-oil emulsion (O'Brien 2009). The push to remove trans fats from diet has brought about several changes to margarine and spreads production. This includes use of new oils, interesterified oil, and other oil blends. Processing considerations have also changed to suit the new oils, many of which require stringent processing conditions (O'Brien 2009). Margarine and spreads processing must be a continuously evolving science in order to meet changing consumer demands (Morlok, 2010).

In Nigeria, majority of the commercial margarine are produced from partially hydrogenated palm stearin, with relatively high trans-fat content. Interesterification of a blend of solid fat and liquid oil, rich in poly-unsaturated fatty acids will be useful for production of low trans-fat table-margarine, which can be declared heart friendly. Interesterification causes fatty acid redistribution within and among triacylglycerol molecules which can lead to substantial changes in Iipid functionality (Marangoni and Ghazani, 2012). Interesterification also reduce the melting point and solid fat content of the product to a desired range (Chibor *et al*., 2018). Percentage free fatty acid and peroxide value of the product will also reduce significantly during interesterification, thus enhancing its oxidative stability without hindering the iodine value of fat blends (Chibor *et al*., 2018).

High melting point of Allanblackia seed oil (35.43 °C) at room temperature (28±2 °C) makes it suitable for use as a solid base in margarine production (Chibor *et al*., 2023). High iodine value of 128.69 g/100g and 121.67 g/100g was reported earlier for African walnut and fluted pumpkin seed oil, respectively (Chibor *et al*., 2023). This indicates that the oil is rich in polyunsaturated fatty acids, which will enhance the nutritional value of the table-margarine. Melting points of 35.43, 17.13 and 18.33 ^{o}C, respectively for *Allanblackia*, African walnut and fluted pumpkin seed oil, as reported by earlier researchers (Chibor *et al*., 2023) make the oils suitable in blending for table margarine production. There is need to produce table-margarine that has good nutritional advantage, that is free of trans fatty acid and its associated health implications, such as increase serum cholesterol. Thus, the objective of this work was to produce table-margarine from interesterified blend of Allanblackia, African walnut and fluted pumpkin seed oils and access its rheological, physicochemical and sensory properties.

2.0 Material and Methods

2.1 Materials

Mature fruits of *Allanblackia floribunda* were obtained from the Rivers State *Allanblackia* Project farm, Mgbu-Azuogu in Oyigbo Local Government Area of Rivers State, Nigeria. Fresh fruits were stored in a covered bag for five days to allow the fruit pulp to disintegrate and enhance seed extraction. Seeds were cleaned and, sundried to 10% moisture content before storing in an airtight polythene bag for further analysis. Freshly harvested fluted pumpkin fruits were procured from Bori market in Rivers State, Nigeria. Mature and good quality African Walnut seeds were procured from Oil mill market in Port Harcourt, Rivers State, Nigeria. Commercial bakery shortenings (local brand and imported brand) were purchased from Spar supper store in Port Harcourt, Nigeria and used as product control.

2.2 Oil extraction

Allanblackia, African walnut and fluted pumpkin seeds were dehulled and cleaned separately, then oven dried at 60^{o}C for 24h (Giami *et al.*,1999) in a hot air oven (model QUB 305010G, Gallenkamp, UK), milled using a laboratory mill (model MXAC2105, Panasonic, Japan). Oil extraction was done using hot water floatation method as described by Rosenthal *et al*. (1996), with slight modification. Here, the milled seeds were made into paste by adding warm and cold water intermittently. The paste was placed in boiling water and allowed to boil for 6 hours. Oil floated to the surface and kept standing overnight in the refrigerator. This allowed the oil to crystallize out, making it easier to be skimmed from the mixture and washed after which the oil was heated to 100 ^{0}C to remove any trace of moisture before storing in airtight plastic containers.

2.3 Oil blending and chemical interesterification

The different based stocks of Allanblackia seed oil, African walnut seed oil and fluted pumpkin seed oil were blended in the following ratio: 60: 20: 20, in accordance with recommended blend of solid and liquid fats for bakery and all-purpose shortening and margarine formulation (Chibor *et al*., 2017). The fat blends were chemically interesterified using the method described by Marangoni and Ghazani (2012). Sodium Methoxide (CH_3ONa) powder was used as catalyst. To each 100g of fat was added 0.28 g of CH_3ONa powder, stirred at 74.6 ^{0}C for 46min. The reaction was stopped with acidic water (4% citric acid), then the blend was washed with dilute basic water (0.1N NaOH) (1:8), three grams (3g) of bleaching earth was added, stirred thoroughly then filtered through Whatman (no 4) filter paper. The product was washed with warm water and dried at 100 ^{o}C.

2.4 Table Margarine production

Table Margarine production was based on a modified version of the method of Kaylegian and Lindsay (1995). For each lot (100 g), 80, 70 and 60 % of the lipid phase were homogenized with 20, 30 and 40 %, respectively of the aqueous phase, to form the margarine emulsion. Lipid phase consisted of interesterified blend of *Allanblackia,* African walnut and fluted pumpkin seed oils, while the aqueous phase consisted of skim milk seasoned with 1.5 g table salt. The lipid phase was melted at a temperature of 70 ^{o}C to destroy crystal history, then set aside. The aqueous phase ingredients were thoroughly mixed by hand. The lipid phase temperature was adjusted to 45 ^{o}C and both phases placed in a table-top blender and vigorously mixed for 2 minutes to fully emulsify the preparation. The emulsion was transferred to a scraped-surface ice cream maker (Maxx model 4420413, China) maintained at 1 ^{o}C by ice water. Churning was done for 20 minutes for the blends to further promote crystallization at 6 ^{o}C. The spread was tempered at 25 °C for 4 h and vigorously reworked with the mixer to smooth the texture, then tempered for 42 h at 5 °C and packaged in plastic cups for further analysis.

2.5 Rheological Properties

2.5.1 Spreadability of Table Margarine

Spreadability of the table margarine at room temperature (28±2 °C) was measured using a 'Mutimer' Apparatus, set up in the Laboratory. Two sets of glass slides of standard dimensions (7.5 X 2.5 cm) were taken. Margarine sample (5 g) was placed between the two glass slides. For 5 min, 100 g weight was placed upon the upper slides so that the margarine between the two slides was pressed uniformly to form a thin layer. The weight was removed and the excess of margarine adhering to the slides was scrapped off. An 80 g mass (M) was tied to the upper slide, through a pulley. The time in seconds required to move the upper slide across 6.0 cm on the stationary bottom slide was taken as a measure of spreadability (Djiobie-Tchienou *et al*., 2018; Modi *et al*., 2012). Spreadability was calculated using equation 1:

S (gm. cm/s) = $\frac{M \times L}{T}$ (1)

Where;
S = spreadability (gm. Cm/s)
M = mass tied to upper slide (gm)
L = distance of slide (cm)
T = time taken (s)

2.5.2 Consistency of Margarine

Consistency of Margarine was measured using a laboratory consistometer, according to the procedure of ASTM F1080-93 (2019). In the instrument reservoir was placed 100 g of table margarine, behind the gate of the consistometer. As the gate is released by pressing the lock release lever, the spring action ensured it opened instantaneously. As the margarine flowed down the instrument, distance covered (cm) after 30 seconds was accurately measured to nearest 0.1 cm, using the graduated scale.

Consistency (cm/sec) = $\frac{d}{s}$ (2)

d =distance covered, s =time taken

2.5.3 Viscosity of margarine

The Kinematic viscosity was measured using the established procedure of the ASTM D445 (2009). It was determined with a calibrated Cannon- Ubbelohde Viscometer: No 2 A149 (Cannon Instrument Co. PA, USA). The viscometer was charged with the sample and suspended vertically in a constant temperature bath. The temperature of the system was maintained at 40 °C.
The efflux time of the oil through the capillary bulb was measured and recorded in seconds.
The Kinematic viscosity was calculated using the following formula (equation 3):

$V = Kc \times t_f$ (3)

V = Kinematic viscosity (mm^2/s)
Kc = Capillary factor/ Viscometer constant (mm^2/s^2)
t_f = Sample efflux time (s)
note: mm^2/s = cSt (centistokes)

2.6 Physicochemical Properties

Physicochemical properties of; pH, melting point, peroxide value, free fatty acid and moisture were determined using standard procedures of AOAC (2012).

2.7 Sensory Evaluation of Table Margarine

Samples of margarine produced were subjected to sensory analysis (Iwe, 2007) on a nine-point Hedonic scale (Ranging from 1 to 9 representing extremely dislike and extremely like, respectively). Twenty-five Semi-trained panellists consisting of students and staff of the Department of Food Science and Technology, Rivers State University, Port Harcourt, Nigeria were involved in the sensory evaluation of the food samples. The following quality attributes were assessed; Taste, flavour, mouthfeel, spreadability, appearance and general acceptability.

2.8 Statistical Analysis

All the analyses were carried out in triplicate. Data obtained were subjected to Analysis of variance (ANOVA), differences between means were evaluated using Tukey's multiple comparison test, and significance accepted at $p \leq 0.05$ level. The statistical package in Minitab 20 computer program was used.

3.0 Results and Discussion

3.1 Rheological Properties

Results for the rheological properties of table margarine produced from interesterified and non-interesterified blends of *Allanblackia*, African walnut and fluted pumpkin seed oils (Table 1), showed spreadability and consistency ranging from 25.76 to 33.40 g-cm/s and 0.03 to 0.14 cm/s, respectively. Spreadability is a texture term relating to the ease with which a product can be spread (IFIS, 2009). It defines the extent a fluid or semi-solid can spread with time under the action of an applied force.

Spreadability of the commercial margarine was significantly ($p<0.05$) higher (33.40 g-cm/s), followed by those of non-interesterified and interesterified margarine containing 60 % fat base. Higher spreadability seen in these samples were probably due to high percentage of moisture presence. There was no significant ($p>0.05$) difference in the spreadability of margarine formulated with interesterified, and non-interesterified fat blend. Spreadability of margarine depends on two dominating factors, including the solid fat content of the oil blend and processing conditions during chilling and crystallization of the emulsion (List *et al*., 1995). The commercial table margarine, though with high spreadability, still showed significantly low consistency (0.05 cm/s), this was probably due to high inter molecular/ binding force within the fat molecules created by presence of emulsifiers and other surface-active components. Consistency of non-interesterified table margarine formulated with 60 % fat base was significantly ($p<0.05$) higher than consistency of other samples indicating that it could flow easier than other samples, under gravity. It measures the extent to which the margarine could flow when no external force except gravity is acting on it (ASTM F1080-93, 2019)**.** The consistency of a plastic fat or oil product is influenced by the proportion of the solid phase; as the solids content increases, the edible fat or oil product becomes firmer (O'Brein, 2009).

Kinematic Viscosity of the table margarine measured at 40 °C ranged from 18.83 to 29.86 cSt. Kinematic Viscosity is a measure of the resistance to flow of fluid under the weight of gravity. Gatenby (2020) defined viscosity as the measure of the oils inherent resistance to flow when no external force except gravity is acting on it**.** Commercial table margarine gave significantly ($p<0.05$) higher viscosity, probably due to its high melting point and presence of emulsifiers and stabilizers. Differences in the viscosity of interesterified (60 % fat base), non-interesterified (80 % fat base and 70 % fat base) were not statistically significant ($p>0.05$) from each other but differed from others.

Table 1. Rheological Properties of Table Margarine Produced from Interesterified and Non-Interesterified Oil Blend.

Margarine Sample	Spreadability (g.cm/s)	Consistency (cm/s)	Viscosity (cSt)
Interesterified (80% fat base)	$25.76^{f}\pm0.03$	$0.03^{e}\pm0.01$	$21.41^{b}\pm0.01$
Interesterified (70% fat base)	$27.87^{e}\pm0.11$	$0.08^{c}\pm0.00$	$21.00^{b}\pm0.00$
Interesterified (60% fat base)	$31.17^{c}\pm0.00$	$0.12^{b}\pm0.00$	$19.81^{c}\pm0.20$
Non-Interesterified (80% fat base)	$25.98^{f}\pm0.01$	$0.03^{e}\pm0.01$	$20.12^{c}\pm0.10$
Non- Interesterified (70% fat base)	$28.21^{d}\pm0.10$	$0.10^{bc}\pm0.00$	$20.00^{c}\pm0.09$
Non- Interesterified (60% fat base)	$32.00^{b}\pm0.00$	$0.14^{a}\pm0.02$	$18.83^{d}\pm0.10$
Commercial margarine	$33.40^{a}\pm0.15$	$0.05^{d}\pm0.00$	$29.86^{a}\pm0.43$

Values are mean ± standard deviation of triplicate samples.
Mean values bearing different superscripts in the same column differ significantly ($p<0.05$).

3.2 Physicochemical Properties

As shown in Table 2, The slip melting point of the commercial margarine was significantly ($p<0.05$) higher (37.00 °C) than others. Slip melting point of the interesterified and non-interesterified table margarine samples were all less than 34 °C, implying the ability to melt perfectly at body temperature, leaving no greasy mouth fill.

The pH of the table margarine ranged from 4.93 to 5.20. The pH was in acid region. pH is one of the main quality characteristics that describes the stability of bioactive compounds in food products (Sanchez-Juanes *et al.,* 2009). William and Dennis (1998) explained that foods at pH 4.40 to 5.00 are medium acid foods and have longer shelf life. pH value ranging from 4.4 to 5.6 had been reported earlier for bowl and packet margarine (Pehlivanoglu *et al*., 2016).

The peroxide value was shown ranging from 0.38 to 0.78 mEq/kg. From Table 4.16, the peroxide value (PV) of non-interesterified and the commercial table margarine were significantly ($p<0.05$) higher than those of the interesterified margarine. Peroxide value of all the table margarine samples were however within the acceptable range of < 3.00 mEq/kg for table margarine and shortenings, much less than 10 mEq/kg recommended minimum for virgin vegetable oil (CODEX, 1999). Pehlivanoglu *et al.* (2016) had earlier reported PV ranging from 0.40 to 13.20 mEq/kg for bowl and packet margarine.

Percentage free fatty acid (FFA) for the table margarine samples ranged from 0.09 to 0.27 %, with non-interesterified margarine and the commercial margarine showing significantly ($p<0.05$) higher value than those of the interesterified margarine. The percentage FFA was lower than values of

0.31 to 0.59 % reported earlier by Pehlivanoglu *et al*. (2016) and also within the recommended range of < 0.3% (CODEX, 1999).
The moisture content of samples $NIEM_3$ and CM were significantly ($p<0.05$) higher, due to lower fat base and high aqueous phase.

Table 2. Physicochemical Properties of Table Margarine Produced from Interesterified and Non-Interesterified Oil Blend.

Margarine Sample	Slip Melting Point (ºC)	pH	Peroxide Value (mEq/kg)	Free Fatty Acid (%)	Moisture (%)
Interesterified (80% fat base)	$32.53^{bc}\pm0.23$	$5.20^{a}\pm0.00$	$0.38^{b}\pm0.02$	$0.09^{b}\pm0.00$	$20.29^{e}\pm0.03$
Interesterified (70% fat base)	$32.10^{cd}\pm0.17$	$5.10^{abc}\pm0.10$	$0.38^{b}\pm0.04$	$0.09^{b}\pm0.02$	$31.80^{c}\pm0.05$
Interesterified (60% fat base)	$32.00^{d}\pm0.20$	$4.90^{bc}\pm0.00$	$0.40^{b}\pm0.00$	$0.10^{b}\pm0.01$	$38.41^{b}\pm0.20$
Non-Inter. (80% fat base)	$33.00^{b}\pm0.00$	$5.10^{abc}\pm0.00$	$0.77^{a}\pm0.11$	$0.28^{a}\pm0.04$	$21.40^{d}\pm0.40$
Non-Inter. (70% fat base)	$32.50^{c}\pm0.30$	$5.17^{ab}\pm0.06$	$0.78^{a}\pm0.01$	$0.30^{a}\pm0.00$	$32.00^{c}\pm0.11$
Non-Inter. (60% fat base)	$32.10^{cd}\pm0.10$	$4.93^{bc}\pm0.12$	$0.76^{a}\pm0.12$	$0.27^{a}\pm0.02$	$39.60^{a}\pm0.30$
Commercial margarine	$37.00^{a}\pm0.00$	$4.93^{bc}\pm0.06$	$0.61^{a}\pm0.02$	$0.26^{a}\pm0.03$	$39.80^{a}\pm0.20$

Values are mean ± standard deviation of triplicate samples.
Mean values bearing different superscripts in the same column differ significantly ($p<0.05$).

3.3 Sensory Properties

Taste, flavour and mouthfeel of the table margarine ranged from 7.64 to 8.00, 6.64 to 7.40 and 6.40 to 8.08, respectively (Table 3). There was no significant ($p>0.05$) difference in the taste and flavour scores for the table margarine samples. The taste fell within the liked moderately and liked very much ranges of 7 to 8, while the flavour fell within the liked slightly and liked moderately ranges of 6 to 7. Sensory evaluation is generally based on texture and flavour related perspectives and appearance that assist to define product acceptability (Bodyfelt *et al*., 2008). Mouth feel for the interesterified and the non-interesterified margarine were significantly ($p<0.05$) higher than that of the Commercial table margarine (Blue band). Mouth feel according O'Brien (2009) is dependent upon temperature, taste sensation, and texture. Mouth feel can apply to either the ability of liquid oils to form an oily film, which is viscosity related, and how well a solid fat melts in the mouth to give a pleasant cooling effect instead of a pasty, waxy feeling that can mask desirable flavours (O'Brien, 2009). Table Margarine produced from Allanblackia, African walnut and fluted pumpkin seed oils (interesterified and non-interesterified) gave a fast dissolving mouth feel, which releases the salt and milk flavour easily. While the Commercial margarine gave a greasy, lingering feel on the tongue. A table-grade margarine is expected to melt rapidly in the mouth for a full

flavour release, to be immediately spreadable and to maintain a solid consistency for prolonged periods on the dinner table (O'Brein, 2009).
Spreadability scores ranged from 5.00 to 8.20, with Blue band given significantly higher ($p<0.05$) score of 8.20. This value correlates significantly with the result presented earlier for physicochemical properties of table margarine (Table 4.16). The commercial margarine spreads easily probably due to high aqueous face and presence of fat stabilizers, which prevented 'draining'. Appearance scores for all the margarine samples were >7.00. Overall acceptability scores for all the table margarine were also >7.00, however, interesterified margarine, non-interesterified margarines with 60 –70% fat base and the Commercial table margarine (Blue band) received higher overall acceptability.

Table 3. **Sensory Properties of Table Margarine Produced from Interesterified and Non-Interesterified Oil Blend.**

Margarine Sample	Taste	Flavour	Mouth Feel	Spreadability	Appearance	Overall Acceptability
Interesterified (80% fat base)	$7.76^{a}\pm0.83$	$6.64^{a}\pm0.99$	$8.08^{a}\pm0.86$	$5.92^{c}\pm1.71$	$7.60^{a}\pm0.50$	$7.16^{bc}\pm0.46$
Interesterified (70% fat base)	$7.64^{a}\pm1.04$	$6.76^{a}\pm0.83$	$7.92^{a}\pm1.12$	$6.72^{bc}\pm0.84$	$7.60^{a}\pm0.50$	$7.22^{abc}\pm0.46$
Interesterified (60% fat base)	$7.64^{a}\pm1.04$	$6.88^{a}\pm0.78$	$7.92^{a}\pm1.12$	$7.36^{ab}\pm0.70$	$7.60^{a}\pm0.50$	$7.36^{abc}\pm0.49$
Non-Interesterified (80% fat base)	$8.00^{a}\pm0.65$	$6.88^{a}\pm1.27$	$7.80^{a}\pm0.76$	$5.00^{d}\pm1.29$	$7.40^{ab}\pm0.50$	$7.06^{c}\pm0.32$
Non-Interesterified (70% fat base)	$8.00^{a}\pm0.65$	$6.88^{a}\pm1.27$	$7.80^{a}\pm0.76$	$6.00^{c}\pm0.71$	$7.08^{ab}\pm0.76$	$7.26^{abc}\pm0.23$
Non-Interesterified (60% fat base)	$8.00^{a}\pm0.65$	$6.88^{a}\pm1.27$	$7.80^{a}\pm0.76$	$6.64^{bc}\pm0.49$	$7.00^{b}\pm0.91$	$7.38^{ab}\pm0.31$
Commercial margarine	$8.00^{a}\pm0.91$	$7.40^{a}\pm0.50$	$6.40^{b}\pm0.50$	$8.20^{a}\pm0.76$	$7.60^{a}\pm0.50$	$7.52^{a}\pm0.28$

Values are mean ± standard deviation of 25 responses.
Mean values bearing different superscripts in the same column differ significantly ($p<0.05$).

4 Conclusion

Taste and flavour scores for the interesterified, non- interesterified and commercial table margarines were not significantly different ($p<0.05$). Margarine formulated with *Allanblackia*, African walnut and fluted pumpkin seed oil blends (interesterified and non-interesterified) were shown to melt easily in the mouth and scored significantly higher than the commercial table margarine. The consistency of non-interesterified table margarine with 60 % fat base was significantly higher, while interesterified margarine containing 60 % fat base compared favourably with the commercial table margarine, in terms of spreadability.

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