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Physico chemical properties of oil obtained from groundnut seeds (*Arachis hypogaea* L.) using different extraction methods

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Abstract
The physicochemical properties of groundnut oils extracted from groundnut seeds (*Arachis hypogaea* L.) by traditional, mechanical and solvent methods were evaluated using standard analytical procedures. The results showed that oil samples are within the range of 0.08-0.060% moisture & matter volatile, 0.13-0.44%m/m insoluble impurities, 0.942-0.951 relative humidity, 1.20-4.14mg/KOH/g acid value, 134.60-168.30mg/KOH/g saponification value, 38.46-47.37g/100g iodine value, 5.20-7.03mEq/kg peroxide value, 2.08-3.60mg/KOH/g free fatty acid, 6.80-8.40g/kg unsaponifiable matter with refractive index of 1.46 for all the oil samples, suggesting significant differences ($p<0.05$) among samples. However, oil extracted by traditional method was the best in terms of storability, resistance to hydrolysis, oxidative deterioration and decomposition likely to be caused by enzymes.

Keywords: physicochemical properties, groundnut seeds, extraction methods, groundnut oils

1. Introduction
Groundnut seed (*Arachis hypogaea* L.) is an important food, feed and principal oil seed crops which is cultivated on large scale throughout the world. It is an annual crop principally for its edible oil and protein rich kernel seeds, borne in pods which develop and mature below the soil surface. (Ayoola and Adeyeye, 2010) [1]. Groundnut seed also known as pea nut is the most common oil nut grown as an annual crop on about 19 million hectares of land in tropical, sub-tropical and warm temperature region of the world. It is grown principally for its edible oil and protein rich seeds. They provide characteristic flavor and texture to food as an integral diet component (Odoemalam, 2005) [2]. Peanut is classified as both a grain legume ("Grain Legumes") and because of its high oil content, an oil crop. As a legume, the peanut belongs to the botanical family *Fabaceae;* this is also known as *leguminosea* and commonly known as the bean or pea family (Royal Botanical Gardens, 2016) [3]. Like most other legumes, peanut harbor symbiotic nitrogen-fixing bacteria in root nodules (Legumes of the work (Royal Botanical Gardens, 2016) [3]. The capacity to fix nitrogen means peanuts require less nitrogen- containing fertilizer and improve soil improve infertility, making them available in crop rotations.
Peanut seeds provides an inexpensive source of high quality dietary protein and oil. It contains palmitic acid, arachidonic acid, lignoic acid and other fatty acids. Groundnut seeds contain 44-56% and 22-30% protein on a dry seed basis and are a rich source of minerals (Phosphorous, Calcium, Magnesium and Potassium) and vitamins E, K and B groups. The vast food incorporating groundnut to improve the protein level have helped in no small way in reducing malnutrition in the developing Countries. The special taste and flavor of foods containing groundnut seeds is important in the acceptance of this food preparation. (Asibuo *et al*., 2008) [4] Groundnut seed oil may be one of the most cardio protective foods readily consumed according to the groundnut institute. The health benefits come from its mono saturated fatty acid contents with regular consumption helping to lower the blood cholesterol level.
Traditionally, groundnut oil extraction involves shelling the groundnut pods, roasting the shelled groundnut seeds, de-skinning/winnowing the roasted seeds, milling and kneading the paste produced (Ewaodo, 2007) [5]. Mechanical method of oil extraction involves the direct application of pressure to the wrapped paste of these oil-bearing materials, forcing a relative reduction in their volumes to initiate the expulsion of oil from the materials (Amoo *et al*.,) [6] Solvent extraction method involves the leaching out of the soluble solid structure of the oil seed by the use of volatile organic solvents like N-Hexane. These compounds enable more oil to be extracted from the primary oil bearing material. The extracted oil could be placed in an oven to evaporate. (Nwabueze, 2007) [7].
Different research work have been carried out on grounds, among which are chemical composition of groundnuts (Asibuo, 2008) [4], chemical analyses of groundnut (*Arachis hypogaea*) oil (Anyasor, 2009) [8], peanut allergy: an overview (Al-Ahamed *et al*., 2008) [9], physic chemical studies on oils from five selected Nigerian plant seeds (Akubugwo and Ugbogu, 2007) [10], effect of heating on the chemical composition and physic chemical properties of groundnut seed flour oil (*Arachis hypogaea* L.) (Ayoola and Adeyeye, 2010) [1] among others. Therefore, the objectives of this present work are to evaluate the chemical properties of oil obtained from groundnut seeds (*Arachis hypogaea*) subjected to three different extraction methods with a view of ascertaining whether the method of extraction impose any changes on the properties of the oil.

Materials and Methods
Source of Materials
Matured groundnuts (*Arachis hypogaea* L.) seeds were purchased from Sayedero, a local market in Ilaro, Ogun

State and transported in a polythene bag to the Laboratories of Department of Food Technology, Federal Polytechnic, Ilaro, Ogun State, Nigeria.

Sample Preparation
The nuts were thoroughly screened to remove stones, sticks, other adhering materials and defected ones. The seed coats were removed manually to recover seed kernels. The seeds were toasted in oven for 3 hours at 105ºC. The toasted seeds were grounded into paste using mortar and pestle.

Method of Extraction of Oils
Traditional Method
The paste obtained from grounded toasted seeds (500g) was suspended in boiling water for 30 minutes using firewood as fuel. Liberated oils floated on the surface and further quantities of water were randomly added after boiling to replace lost by evaporation as well as encouraging more oil to float to the surface. The oil was carefully scooped with stainless spoon from the surface of the water into shallow dish and then heated over a fire to remove residual moisture. The slurry was also placed in a muslin cloth and pressed to remove the remaining oil. The oil was put in a plastic bottle and stored at room temperature prior to analysis.

Mechanical Method
This method was carried out using a hydraulic press extraction. The paste obtained from grounded toasted seeds (500g) was tied in a muslin cloth, placed inside the extraction chamber of the hydraulic press and pressure was applied to expel the oils. The oil sample was collected in a conical flask (AOAC, 2000) [11].

Solvent Extraction Method
The method described by (Faritan *et al*., 2010) [12] was used for solvent extraction of seeds. The already grounded roasted paste (500g) was placed in a paper and fed to a soxhlet apparatus connected with a water bath for 6 hours with n-hexane (b.p 65-68ºC). After the extraction was accomplished, the excess of the solvent was removed under reduced pressure using a rotary vacuum evaporator (EYELA, N.N. Series, Rikakikai Co Ltd, Tokyo, Japan). The oil was stored in plastic bottles prior to analysis at room temperature. All chemicals and reagents used were of analytical grade.

Analytical Procedures.
Estimation of Moisture
The moisture content of groundnut sample was determined by drying in oven at 100-107ºC to constant weight. (AOAC, 2000) [11].

Insoluble Impurities
This refers to extraneous substance such as dirt, debris and fibres. Also defined as substances which remain insoluble and can be filtered off after the oil is dissolved in specific solvent such as petroleum (0.003-0.008)

Relative Density
Pycnometer, i.e. specific gravity bottle was used in measuring the density/ specific gravity. The specific gravity of oil is the ratio of the weight in air of a given volume oil at a defined temperature to that of the same volume of water at the same temperature (AOAC, 2000) [11]. Cleaned, dried pycnometer was weighed and filled with water maintained at 20ºC and weighed again. The bottle was emptied, dried and filled with oil and weighed. The value was calculated using the equation:

$$\text{Specific Gravity} = \frac{\text{Weight Oil}}{\text{Weight of Water at 20 oC}}$$

It is temperature dependent and decreases in value when temperature

Saponification Value
Saponification value was determined according to the titrimetric method of Pearson (Pearson, 1981) [14]. 2g of oil sample was weighed into a conical flask and 25ml of alcoholic potassium hydroxide was added. The solution was heated in boiling water for 1hour. 1ml of 1% phenolphthalein was added and titrated with 0.5N HCL. A blank was prepared alongside the oil samples.

Saponification Value = 56.1N (A-B) W

Where;
N=Normality of HCL used
A=Volume of H2So4 for blank in ml
B=Volume of H2So4 for sample in ml
56.1= Equivalent weight of potassium hydroxide
W= Weight of the oil used.

Acid Value
Acid value was determined by trite metric method of (Pearson, 1970) [15]. 5g of the oil sample was weighed and 75ml of hot neutral alcohol was added with few drops of phenolphthalein. The mixture was shaken until the pink colouration remains permanent. Acid value was calculated using the formula:

$$\textbf{Acid value} = \frac{\text{V x 56.1}}{\text{Weight of Sample (g)}}$$

Where V = titration end point value

Iodine Value
This was determined according to the titrimetric method of (Pearson, 1970) [15]. 2 g of oil weighed into a dry glass stopper bottle of 25ml capacity and 10ml of carbon tetrachloride was added to the oil. About 20ml Wij's solution was then added and allowed to stand in the dark for 30minutes. 15ml of (10%) potassium iodide and 100ml of water were added and then titrated with 0.iM sodium thiosuphate solution using starch as indicator first before the end point. A blank was also prepared for the oil sample. Iodine value was calculated from the formula:

$$\textbf{Iodine Value (Wij's)} = \frac{(V2\text{-}V1) \times 1.29}{\text{Weight of Sample (1g)}}$$

Where;
V2 =Titre value for blank
V1 =Titre value for sample (s)

Peroxide Value
The peroxide values for the oil samples were determined using the method described by (AOAC, 1984) [16]. 2g oil sample was weighed into a tube and 1g of powdered

potassium iodide with 20ml of solvent mixture (glacial acetic acid and chloroform) was added. This was then placed in boiling water for 30 seconds. The content was poured into a flask containing 20ml of 55 iodide solution. The tube was then washed with 25ml of distilled water and titrated with 0.002N sodium thiosulphate solution using starch as indicator. A blank was prepared alongside the oil samples. Peroxide value was calculated as:

$$\textbf{Peroxide value} = \frac{2(V1\text{-}V2)\ mEq/kg}{\text{Weight of sample (g)}}$$

Where; V2 =Blank titre value
V1 =Sample (s) titre value

Refractive Index

The refractive index was determined by hand refractometer (Ermma Hand Refractometer) as described by (Ayo and Agu, 2012) [17]. It has a range of 0-32. A drop of the oil was placed on the surface of the refractometer and the reading was taken directly.

Free Fatty Acid

The free fatty acid value was estimated by titrating the oil against Potassium Hydroxide (KOH) in the presence of phenolphthalein indicator. Free fatty acid content is expressed as oleic acid equivalent (Cox and Pearson, 1962) [18].

Results and Discussion

Results

Table 1: Physico-chemical Properties of Groundnut Oil using Different Extraction Methods

Parameters	Samples		
	TM	MM	SEM
Moisture and Matter Volatile at 105oC (%)	0.08±0.028[a]	0.50±0.07[b]	0.60±0.6[b]
Insoluble Impurities (%m/m	0.13±0.007[b]	0.45±0.007[b]	0.44±0.275[b]
Relative Density at 27oC	0.951±0.001[a]	0.951±0.070[a]	0.942±0.227[a]
Acid Value (mg/KOH/g oil)	1.20±0.028c	2.24±0.282[b]	4.14±0.028[a]
Saponofication Value (mg/KOH/g/oil	168.30±0.028[a]	146.87±0.007[b]	134.60±36.869[c]
Iodine Value (g/100g)	44.20±0.282[b]	39.46±0.000[c]	47.37±94.014[a]
Peroxide Value (mEq/kg	5.20±0.000[c]	6.40±0.282[b]	7.03±1.98[a]
Refractive Index at (40oC)	1.46±0.000[a]	1.46±0.000[a]	1.46±0.000[a]
Free Fatty Acid(mg/KOH/g	3.60±0.282[a]	2.22.70±0.282[a]	2.08.4±0.282[b]
Unsaponifiable Matter (g/kg	7.70±0.141[a]	8.40±0.141[a]	6.80±0.141[a]

Values with different superscript in the same row are significantly different at ($p<0.05$) confidence level.
Key: TM: Traditional method, **MM:** Mechanical method, **SE:** Solvent extraction method

The results of the physicochemical properties of groundnut extracted using different methods are shown in Table 1. The determination of these physicochemical values is often used as a general indicator of the condition and edibility of oil. The moisture and matter volatile ranged from 0.08-0.60 for the three oil samples, indicating significant differences ($p<0.05$) among samples. It was observed that oil extracted traditionally had the lowest value of 0.08% with oil extracted by solvent method having the highest value of 0.60%. (Mandioi *et al*., 2014) [19] Had specified 0.20 maximum level of moisture in oil. However, oils containing higher values can lead to infestation by insects and microbial attack which will allow rancidity to set in. the insoluble impurities for the oils samples were 0.13% (m/m), 0.45% (m/m) and 0.44% (m/m) for the three extraction methods respectively. Oil extracted traditionally has the lowest values of 0.13% (m/m), indicating little extraneous materials while both mechanical and solvent extracted oil had higher values of 0.45% (m/m) and 0.44% (m/m) suggesting high insoluble impurities which can reduce the oil quality. There are no significant differences ($p<0.05$) in the relative density of all the oil samples under consideration. Specification level of 0.912-0.920 had been set by (Mandioi *et al*., 2014) [19]. Relative density of 0.951, 0.951 and 0.942 at 27^{o}C as obtained in this present work revealed higher levels above the tolerable level.

The acid values ranged from 1.20mg/KOH/g-4.14mg/KOH/g for the for the oil samples. The acid value is a relative measure of rancidity as free fatty acids are normally formed during decomposition of oil glycerides. The value can also be expressed as percent of free fatty acids calculated as oleic acid. Acid value measures the extent to which glyceride in the oil has been decomposed by lipase and other actions such as light and heat. (Atasie *et al*., 2009) [20]. Acid value of 4.14mg/KOH/g was close to Arachis (4.0mg/KOH/g) by (Pearson 1981) [14] and soya bean 4.279mg/KOH/g (Eka and Chidi, 2009) [22]. Also low level of acid value of about 4mg/KOH/g oil sample shows that the oil is as good as edible oil (Denniston *et al*., 1991) [24]. Saponification value of 165.30mg/KOH/g, 146.87mg/KOH/g and 134.60mg/KOH/g were obtained for traditional, mechanical and solvent extracted oils, indicating significant differences among samples ($p<0.05$). These value are lower 9187-196mg/KOH/g) reported by (Pearson, 1981) [14] this properly makes the oil useful in soap making. (Denniston *et al*., 1991) [24]. stated that oils with high saponification value contain high proportion of lower fatty acid. However, (Ajayi and Oderinde, 2002) [25] reported that high saponification value indicated the presence of greater number of ester bond, suggesting that the fat molecules were intact. Iodine value is a measure of the amount of unsaturated fatty acids in the oil. Iodine value varied from 39.46-47.37g/100g for all the oil samples extracted. A fatty acid that is missing any hydrogen atoms is classified as being unsaturated and this indicates all mono unsaturated and polyunsaturated fatty acids. High iodine value denotes high degree of unsaturation of the oil caused by the extent of extraction and degree of heat treatment during processing (Popoola and Yangomodou, 2006) [26]. The iodine values (39.46-47.37g/100g) indicates low degree of unsaturation and classified the oil as non-drying oil (80-100g/100g) as recorded for most edible oil ((Pearson 1981 and Atasie *et*

al., 2009) [14, 20]. Similar non-drying oil value have been reported for *dacoyedeedulis* pulp and seeds and *cucurbifa maxima* seed (Eromosele and Pascal, 2003; Amoo *et al*.,) [27, 6]. Peroxide values of 5.20mEq/KG, 6.40mEq/kg and 7.03mEq/kg were obtained for oil extracted traditionally, mechanically and use of solvent respectively, indicating significant differences ($p<0.05$) among samples. According to (Pearson 1981) [14], peroxide value is a measure of the peroxides contained in the oil. During storage, peroxide formation is slow at first during an induction period which may vary from a few weeks to several months according to the particular oil or fat, temperature etc. Fresh oils have peroxide values less than 10mEq/kg and values between 20 and 40mEq/kg results in rancid taste (Akubugwo and Ugbogu, 2007) [11]. The low peroxides values obtained in this present work indicates slow oxidation of these oil, hence can resist lipolytic hydrolysis and oxidative deterioration (Eromosele and Pascal, 2003) [27]. The refractive index were 1.46 at 40Oc, suggesting no significant differences among samples. The refractive index of 1.46 revealed that the oil contained some double bonds in its fatty acid composition as reported by (29, 21), that refractive index increases as the double bond increases. The results of acid value are often expressed as the percentage of free fatty acids (FFA). Acidity is usually accompanied by free fatty acid formation; the determination is often used as a general indication of the condition and edibility of oils (Pearson 1981) [14]. The free fatty acid varied from 2.08mg/KOH/g-3.06mg/KOH/g for all the oil samples. The low value obtained as FFA indicates some percentage of fatty acid present in the oils and that the oils may likely undergo oxidation (Akanni 2005) [21] Unsaponifiable matters ranged from 6.80g/kg-8.40g/kg for the oil samples. Unsaponifiable matter is defined as the material present in oils and fats which after saponification of the oil or fat by caustic alkali and extraction by a suitable organic solvent remains non-volatile on drying at 80ºC. These include hydrocarbons, higher alcohols and sterols. Most oils and fats of normal purity contain less than 2% of unsaponifiable matter. (Pearson 1981) [14]. Adulteration of oils and fats with paraffin hydrocarbons will appear in the unsaponifiable matter (Pearson 1981) [14].

Conclusion

The physicochemical properties of oil extracted using traditional, mechanical and solvent methods have shown that oils obtained by traditional method are better in terms of most indices evaluated.

The moisture and matter volatile 90.08% that confers storability on oils, peroxide values indicating resistance to hydrolysis and oxidative deterioration as well as acid value which shows the extent to which glyceride in the oil has been decomposed by lipase and other actions such as heat and light were extremely low in oil obtained by traditional method.

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