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Full Length Research Paper

Extraction performances of polar and non-polar solvents on the physical and chemical indices of African breadfruit (*Treculia africana*) seed oil

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The physical and chemical indices of African breadfruit (*Treculia Africana*) seed oil extracted with polar (isopropanol, Hexane and butanol) and non polar (Acetone) solvents were investigated. The oil (19.85%) and energy contents 452.35 (Kcal) suggest that African breadfruit seeds are a high -energy food. The yields were significantly (*P* 0.05) different with hexane extracted (non-polar solvent) oil having 19.85 % whilst oil extracted with polar solvents ranged from 15.58 -19.30 %. Melting points were 34, 27, 26, 21 ^{0}C for oil extracted with hexane, isopropanol, butanol and acetone respectively. Smoke points were within the limits of 170 – 255 ^{0}C for the four oil samples. Iodine values ranged from 14.50 (hexane extracted) t0 25.17 (acetone extracted) . Saponification values ranged from 125.89-267.85 while peroxide values were 3.20 mg/kg (hexane) and 3.60-3.83 mg/kg for polar solvents. Free fatty (oleic) acid of oil extracted with hexane was 1.71% and polar solvent extraction ranged from 1.65- 1.78%. Hexane had the least peroxide value (8.74 mg/kg) compared to higher values for oil extracted with the polar solvents (9.10 - 9.83 mg/kg).

Keywords: African breadfruit, *Treculia Africana*, oil extraction, saponification, fatty acids, peroxide value, thiobarbituric acid

INTRODUCTION

African breadfruit (*Treculia africana*) constitutes a strategic reserve of essential food nutrients that are available at certain critical periods of the year when reliable sources of these nutrients are under cultivation and are very scarce. Diverse food forms could be produced from the seeds on the basis of custom, tradition, ethnic background. It is boiled and consumed as white porridge and sauce with or without fresh corn, roasted and consumed with coconut or palm kernel as snack, made into refreshing milk drink, prepared into flour as soup condiment or thickener and for bakery and confectionaries. In the past the consumption was limited to poor village dwellers for whom it supplemented their diets during times of food scarcity and substituted the more expensive rice during festivals and other ceremonies on the basis of tradition and cost (Nwabueze and Nwokenna, 2006). But today, African breadfruit has become a delicacy and a specialized meal not only for the rich and the urban dwellers in Nigeria but has also become a foreign exchange earner. Dehulled kernels are sun-dried and exported to cater for the African consumer interests overseas.

African breadfruit seed is of high nutritional value. Each seed contains about 14-17% crude protein, 2.5 % crude fibre, 35-60 % carbohydrate and a good supply of vitamins and minerals (Akubor, 2000). The amino acid composition has also been highlighted to further buttress its nutritional potentials (Nwabueze, 2007) . The oil potential of the seed has been reported (Ajiwe et al., 1995). Generally, plant oil serves as rich source of essential fatty acids and energy yielding approximately 9 kilocalories per gram as well as being a carrier of fat-soluble vitamins (A, D, E, and K) which help the body to absorb most of its nutrients (Ajiwe et al., 1995). Plant oils play other distinctive role in the natural flavour and palatability of a wide of food commodities (Ihekoronye and Ngoddy, 1985)

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while certain of its components (Linoleic, Linolenic and arachidonic acid) are essential in all diets for body growth and normal skin condition (Ebuehi et al., 2006).

It has been reported (Bjorck and Asp, 1983) that only 45-55 % of the oil present in raw materials could be extracted with ethyl ether after extrusion. But Nierle et al. (1980) report an average oil recovery in extruded wheat to be 40 and 20 % for maize. A number of reasons were advanced for this reduction of oil extraction in extrudates. Monoglycerides and free fatty acids have been reported to form complexes with amylose during extrusion processing (Mercier, 1980; Badrie and Mellowes, 1992). Such complexes are likely to cause difficulty in plant oil extraction with petroleum ether.

A literature search did not reveal much information deal ing with selection of organic solvents for plants and in particular African breadfruit seed oil extraction. The objective of this research was to evaluate the effectiveness of polar and non polar solvents in African breadfruit seed oil extraction and to study their effects on the physical and chemical properties of the oil. This study is necessary to assess African breadfruit seed oil quality indices for proper application in food and industrial system.

MATERIALS AND METHODS

Raw Materials and Preparation

About 10 kg of African breadfruit (*Treculia africana*) seeds were purchased from Umuahia main market, Abia State, Nigeria. They were cleaned by hand picking to rid them of any contaminating stones or extraneous and organic materials before being parboiled at 100^{O}C for 15min. Parboiled and drained seeds were then threshed in a commercial attrition mill and manually dehulled to recover the kernels. The kernels were sun dried for about 17 h and then milled in a blender (Moulinex 276, France, speed 1) to fine flour (2 mm particle size). The flour was preserved in a tight polyethylene bag at room temperature (28± 2^{O}C) from which samples were collected for different analyses.

Extraction and yield of African breadfruit seed oil

African breadfruit seed oil was extracted from the resulting flour using four different food grade organic oil extraction solvents (boiling point of 60 - 80^{O}C). These included iso-propanol, butanol, and acetone (polar solvents) and hexane (non polar solvent). The extraction involved the use of Soxhlet extraction method (AOAC, 1995). About 100g of the flour were weighed into a thimble in the Soxhlet extractor fitted to conical flask. African breadfruit seed oil was extracted with 250 ml of each solvent. The solvent was boiled under reflux for about 6 h. African breadfruit seed oil yield was calculated for each extraction solvent by weight difference of the sample before and after extraction and reported as mean of duplicate determinations.

Proximate composition of dehulled full fat African breadfruit seed flour

Proximate composition of dehulled-sun dried African breadfruit seed was determined in triplicate for moisture, crude protein (micro Kjeldhal method), fat (Soxhlet method), crude fibre, and ash according to AOAC (1995) methods. Total carbohydrate was determined by difference. Energy was calculated using the At Water factors of 4 x protein, 4 x carbohydrate and 9 x fat (Nwabueze, 2006).

Determination of Vitamins in African breadfruit seed oil

Vitamin A was determined by the method described by Delia and Meiko (2003) while the spectrophotometeric method described by Pearson (1976) was used for the determination of vitamin E.

Physical properties of African breadfruit seed oil

The physical characteristics of African breadfruit seed oil determined included yield, colour (photometric system), specific gravity, melting and smoke points were determined by the standard methods as described by AOAC (1995). Specific gravity was determined by use of specific gravity bottles at a temperature of 28±2 ^{O}C. Photometric colour index (pci) of African breadfruit seed oil was determined on 1g sample according to the method described by Pike (2003). The sample was weighed and dissolved in 20 ml water/ethanol mixture. The mixture was filtered after standing for 30 min. The absorbance of the filtrate was measured at 400, 550, 620 and 670 nm using spectrophotometer (Unican He 105Y, England). The solvent was used as blank. Photometric colour index was calculated as

$$pci = 1.29\ (A_{400}) + 69.70\ (A_{500}) + 41.20\ (A_{620}) - 56.41\ (A_{670}) \quad (2)$$

Where A = absorbance**.**

Chemical properties and acid concentrations

The acid values, iodine value, saponification value, peroxide and thiobarbituric acid values were determined by the standard methods of AOAC (1995). Fatty acid composition of the oil was determined by the method described by Christie (1980) using a flow-Mac flame ionization gas liquid chromatography. The methylation of the fatty acids was prepared according to standard methods of AOAC (1995) . The equipment was set at oven temperature of 200^{O}C with a 1.8 m by 0.32 cm stainless column packed with 10 % DEGS on material support of silicon mesh size. Nitrogen was used as a carrier gas at a flow rate of 25 ml/min and the quantification of the fatty acid was obtained by automatic integration with linear response attached to the system.

Effect of ambient storage on African breadfruit seed oil

Extracted oil samples from different extraction solvents were stored in plastic bottles tightly corked to practically prevent entrance of air at (ambient) room temperature (28± 2 ^{O}C) for 25 days. During this period the thiobarbituric acid and peroxide values were determined at 0, 8, 19 and 25th days for each extract and reported as mean duplicate determinations.

Statistical analysis

Differences between means were assessed by Students t – test, while the levels of significance of the data were calculated by

Table 1. Proximate composition and energy values of dehulled full fat African breadfruit seed flour.

Moisture (%)	Protein (%)	Fat (%)	Ash (%)	Carbohydrate (%)	Energy (kcal)
9.425±0.002	8.76±0.002	19.85 ±0.025	2.30±0.001	59.67 ±0.006	452.35±0.041

Table 2. Oil Yield and vitamin content of African breadfruit seed oil extracted with different solvents.

Extraction solvent	Yield (%)	Vitamin A (g/g)	Vitamin E (mg/100g)
Isopropanol	18.10^{c} ± 0.01	21.32^{b} ± 0.01	3.11^{b} ± 0.01
Butanol	19.30^{b} ± 0.01	16.09^{c} ± 0.03	2.05^{d} ± 0.01
Hexane	19.85^{a} ± 0.02	27.39^{b} ± 0.01	5.67^{a} ± 0.04
Acetone	15.58^{d} ± 0.06	12.78^{d} ± 0.03	2.56^{c} ± 0.03

Means not followed by the same superscripts along the same column are significantly different (*P* 0.05).

analysis of variance at 0.05 % level of significance according to Steels and Torrie (1981).

RESULTS AND DISCUSSION

Proximate composition of dehulled full fat African breadfruit seeds flour

The proximate composition of dehulled sun - dried full fat breadfruit seeds flour is presented in Table 1. With a moisture content of 9.43 %, the African breadfruit seed full fat flour will keep well. Protein content was 8.70 % which was lower than the range (15.76 -17.52 %) reported by Nwabueze (2006) probably due to variety used. Since African breadfruit seeds have not been classified or graded into varieties or breeds or cooking types, they are marketed as mixtures of varieties, a problem that also affects its cooking time (Nwabueze and Nwokenna, 2006).

The ash content of 2.30 % suggested a good source of minerals (Nwokolo, 1987; Akubor, 2000) while the oil (19.85%) and energy contents suggest the seed to be a high-energy food. The carbohydrate content of 59.66 % could pass African breadfruit seed for composite flour in baked and confectionary products.

African breadfruit seed oil yield and Vitamins

Oil Yield and vitamin content (A as carotenoid and E) of African breadfruit seed oil extracted with different solvents are shown in Table 2. Generally, all the African breadfruit seed oil extracts was yellow in colour and remained liquid at room temperature (28±2^{o} C) . The photometric colour index shows significant (*P* 0.05) differrences in their values. This could be attributed to the selective permeability of the different solvents used. The yields of oil extracted with the hexane (non-polar solvent) were significantly (*P* 0.05) different from that of the oil extracted with polar solvents (isopropanol, butanol and acetone), having a yield of 19.85% while the amount of oil extracted by the polar solvents ranged from 15.58% (acetone) to 19.30% (butanol). Various authors (Nierle et al., 1980; Ajiwe, et al., 1995; Ebuehi and Avwobobe, 2006) have reported varying extraction values with different solvents or seeds. Ajiwe, et al. (1995) reported an extraction yield of 20.83% oil from African breadfruit seed by Soxhlet using petroleum ether as extraction solvent, Nierle et al. (1980) reported an average oil reco-very of 20% in extruded maize while Ebuehi and Avwobobe (2006) reported a yield of 17.36% for water melon. In this study, hexane gave highest oil yield but the use of such oil has been questioned in terms of safety particularly if not purified (Bera, et al., 2004). Ethanol, methanol and acetone have been recommended as solvents for extraction of vegetable oils. Thus, oil extraction yield from African breadfruit seed may correlate closely with polarity of the extraction solvent.

Non-polar solvents have both sides charged and are able to penetrate into the matrix of a feed. This is so, because they lack an O-H end which otherwise would interfere with the extraction process. The low extraction yield of African breadfruit seed oil reported in this study and in literature could be attributed partly to extraction solvents used, state of the food material and partly due to complex formation between fatty acids and carbohydrate breakdown components (Mercier, 1980). Such complexes will definitely cause difficulty in plant oil extraction with petroleum ether. Following the minimum fat requirement of 6% in complementary formulation (Obatolu, 2002) , the oil yields by all the solvents meet this requirement. Vitamin A (– carotene) varied significantly (*P* 0.05) with a range of 12.78 g/g in oil extracted with hexane to 37.39 g/g in oil extracted with acetone. Vita-min A deficiency has been recognized as the second

Table 3. Physical properties of African breadfruit seed oil extracted with different solvents.

Extraction solvent	Specific gravity (g/ml)	Melting point (^{0}C)	Smoke point(^{0}C)	Photometric colour index
Isopropanol	$0.8627^{c} \pm 0.001$	$27.15^{b} \pm 0.002$	$149.67^{d} \pm 0.03$	$148.08^{c} \pm 0.03$
Butanol	$0.8756^{b} \pm .0001$	$26.00^{c} \pm 0.004$	$255.00^{a} \pm 0.06$	$367.11^{b} \pm 0.05$
Hexane	$0.8656^{c} \pm .0001$	$34.00^{a} \pm 0.001$	$250.00^{b} \pm 0.02$	$476.55^{a} \pm 0.02$
Acetone	$0.9760^{a} \pm .0003$	$21.01^{d} \pm 0.002$	$180.27^{c} \pm 0.08$	$140.77^{d} \pm 0.03$

Means not followed by the same superscripts along the same column are significantly different (*P* 0.05).

Table 4. Chemical properties of African breadfruit seed oil extracted with different solvents.

Extraction solvent	Iodine value	Saponification value	Peroxide Value (mg/kg)
Isopropanol	$15.72^{c} \pm 0.06$	$125.89^{d} \pm 0.3$	3.83a±0.1
Butanol	$21.51^{b} \pm 0.01$	$238.43^{c} \pm 0.01$	$3.60^{a} \pm 0.01$
Hexane	$14.50^{d} \pm 0.01$	$259.46^{b} \pm 0.01$	$3.20^{b} \pm 0.01$
Acetone	$25.17^{a} \pm 0.1$	$267.85^{a} \pm 0.03$	$3.78^{a} \pm 0.03$

Means not followed by the same superscripts along the same column are significantly different (*P* 0.05).

most common form of malnutrition. The difference in vitamin A content of the oil samples could be attributed to a stability factor since it is readily stable in non polar solvents. Thus the amount extracted could be as a result of the solvent used as well as the amount of oil extracted (yield) and the concentration of the vitamin in the extracted oil.

The vitamin E content varied from 2.05 mg/100g in oil extracted with hexane to 56.69 mg/100g in oil extracted with butanol. All the oil samples extracted with polar solvents (isopropanol, butanol and acetone) had low values (2.05 – 3.11 mg/100g) of vitamin E. These vitamin E values are higher than those reported (Baurnfeind, 1980) for soybean oil (1.2 mg/100g) and corn oil (2.0 mg/100g). With the working solvents in this study, African breadfruit seed oil will make a good source of vitamin E and hence have an antioxidant stability effect on the oil. This is because vitamin E has been recognized as the most biological oxidant in humans.

Physical properties of African breadfruit seed oil

Table 3 shows physical properties of African breadfruit seed oil extracted with different solvents. Specific gravity was significantly (*P* 0.05) different varying from 0.8627 g/ml Isopropanol to 0.970 g/ml (acetone). This range of specific gravity of African breadfruit seed oil compares with the ranges reported for palm oil (0.91) and groundnut oil (0.84) (Ebuehi, et al., 2006) and 0.91 for pumpkin (*Curcubita pepo*) seed oil (Ihediohanma et al., 2006). Higher deviations from these values point to hydrolytic and oxidative changes. Melting and smoke points of the oil samples were significantly (*P* 0.05) different with extraction solvent used. The smoke points showed that African breadfruit seed oil could be used in deep frying food systems as oils with high smoke points resist ignition.

Chemical properties of African breadfruit seed oil

Chemical properties of African breadfruit seed oil extracted with different solvents are presented in Table 4. Iodine values were generally low when compared to other oils of plant origin. It ranged from 14.50 (hexane extracted) t0 25.17 (acetone extracted) which functionally may not qualify the oil to be used as a drying oil. The degree of unsaturation of oil is determined by measuring its iodine value. Iodine value is a measure of fat or oil stability and resistance to oxidation. Oresanya et al. (2000) stated that fat oxidation which occurs during fat extraction or storage and transportation leads to hydrolysis of triacylglycerols to free fatty acids. Lee (1983) strongly suggested that iodine value of oil be determined by the temperature at which the oil seeds are produced, thus the higher the temperature, the lower the iodine value and vice-versa.

African breadfruit seed oil from the four different solvents showed significantly (*P* 0.05) different values with respect to their saponification values. They ranged from 125.89-267.85. Oil extracted with isopropanol had the least value while that of acetone was the highest. High saponification value indicates high molecular weight oil good mainly for soap making and for hair shampoo (Ajiwe, et al., 1995). According to Champe and Harvey (1994), saponification values measure the amount of alkali required to combine with the fatty acids liberated by the hydrolysis of fats and oils from which weights of fatty acids could be determined.

Table 5. Acid concentrations of African breadfruit seed oil extracted with different solvents

Extraction solvent	pH	Acid value (%)	Thiobarbituric Acid (mg/kg)	Oleic Acid (%)
Isopropanol	$3.38^{b} \pm 0.1$	$3.21^{c} \pm 0.06$	$18.42^{a} \pm 0.3$	$1.65^{b} \pm 0.06$
Butanol	$4.45^{c} \pm 0.0001$	$2.48^{d} \pm 0.0001$	$16.63^{b} \pm 0.0001$	$1.25^{c} \pm 0.0001$
Hexane	$5.70^{a} \pm 0.0001$	$3.40^{b} \pm 0.0001$	$15.69^{c} \pm 0.0001$	$1.71^{ab} \pm 0.0001$
Acetone	$5.16^{b} \pm 0.03$	$3.55^{a} \pm 0.03$	$14.83^{d} \pm 0.03$	$1.78^{a} \pm 0.03$

Means not followed by the same superscripts along the same column are significantly different (*P* 0.05).

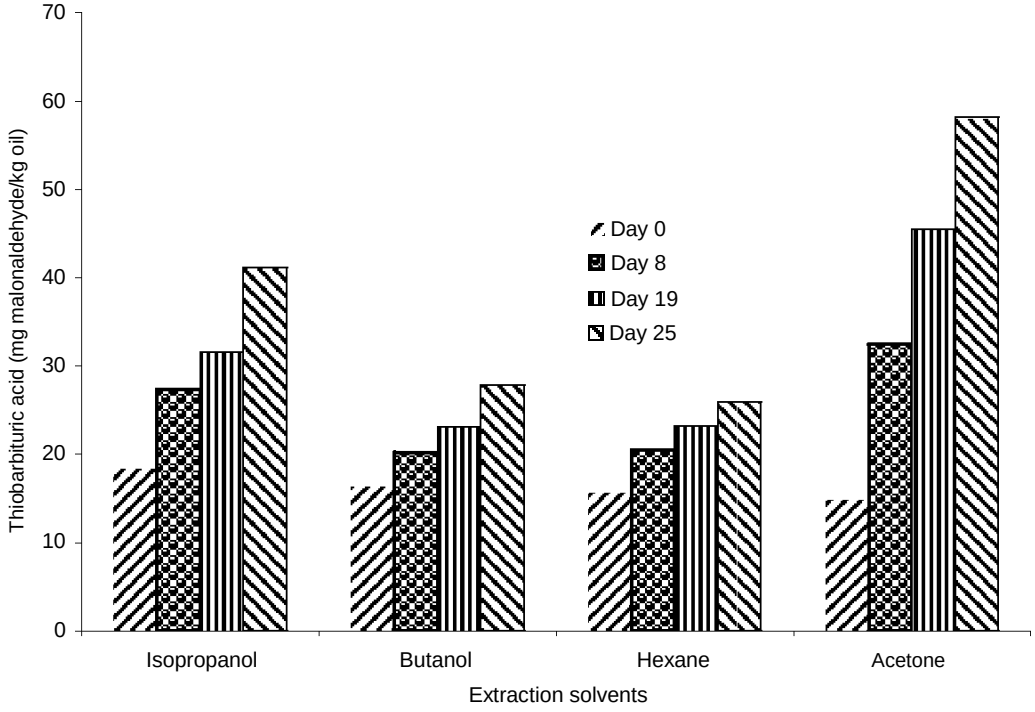

Figure 1. Effect of storage on Thiobarbituric acid of African breadfruit seed oil extracted with different sovents.

Peroxide value of African breadfruit seed oil varied with solvent used. Peroxide value is used as an indicator of deterioration of fats and oils. As oxidation takes place, the double bonds in the unsaturated fatty acids are attacked producing peroxides. This in turn decomposes releasing secondary products which cause rancidity (Champe and Harvey, 1994; Ebuehi, et al., 2006). In this study, peroxide value of oil extracted with hexane (3.20 mg/kg) was significantly (*P* 0.05) lower than oil samples extracted with polar solvents (3.60-3.83 mg/kg).

Acid concentrations of African breadfruit seed oil

Table 5 shows acid concentrations of African breadfruit seed oil extracted with different solvents. Mean pH values of the oils varied significantly (*P* 0.05) with different solvents. It ranged from 3.38 (isopropanol extraction) to 5.70 (hexane extraction). This variation could have affected the values of vitamins observed in this study as pH < 7 has been reported (Ihekoronye and Ngoddy, 1985) to affect the stability of vitamin A and its precursors.

The Table showed that there was no significant (*P* 0.05) difference between the free fatty acid (oleic acid) of oil extracted with hexane (1.71%) and that extracted with either acetone (1.78%) or with isopropanol (1.65%). Oil samples extracted with butanol had the least free fatty acid as oleic acid and so may not be easily prone to oxidation. Champe and Harvey (1994) stated that several cardiovascular diseases have been implicated in human population consuming diets rich in polyunsaturated fatty acids.

The acid value for the four different crude oils were high thus, the oil extracts would need some form of purifi-

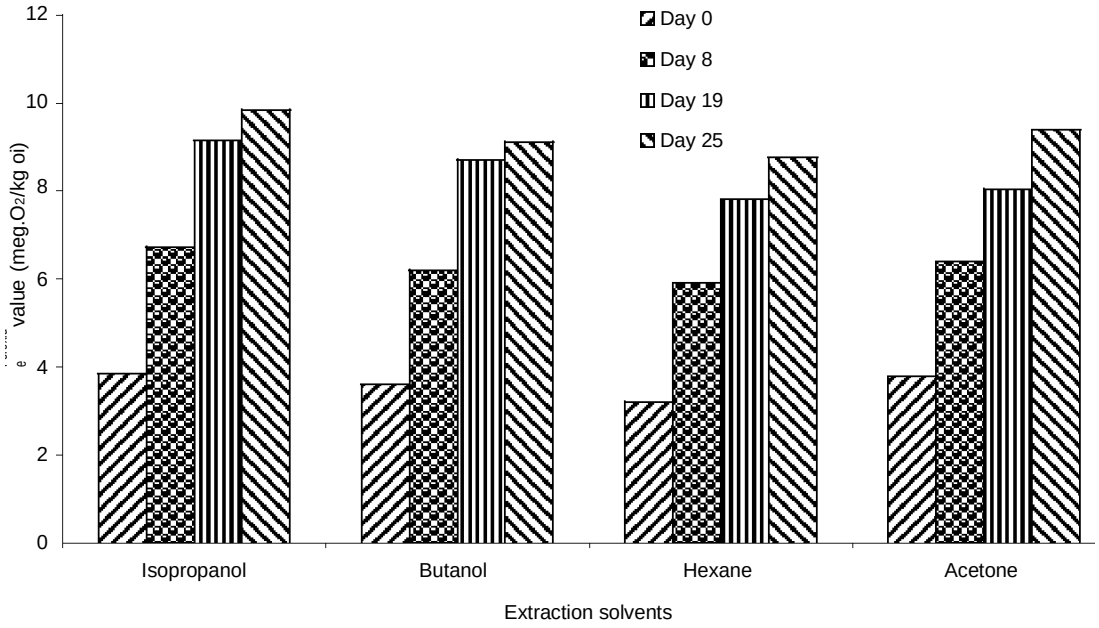

Figure 2. Effect of storage on peroxide valves of African breadfruit seed oil extracted with different sovents.

cation (refining) to enhance stability and storage. Low acid values suggest stability of oil. Oscar (2002) reported that acid value or free fatty acid (FFA) in crude oils, estimates the amount of oil that will be lost during refining. Thus the higher the acid as FFA values the higher the amount of oil that could be lost during processing.

Effect of storage on thiobarbituric acid and peroxide values of African breadfruit seed oil

Effect of storage of African breadfruit seed oil extracted with different solvents on thiobarbituric acid and peroxide value are shown in Figure 1 and 2, respectively. Thiobarbituric acid and peroxide values are two important rancidity indices which were found to vary with storage in this study. At the end of the 25 day storage period at room temperature (28^{o}C), the oil extracted with hexane had the least peroxide value of 8.74 mg/kg compared to higher values for oil extracted with the polar solvents which ranged from 9.10 mg/kg (butanol) to 9.83 mg/kg (isopropanol).

This observation indicates that rate of autocatalytic degradation of oil extracts, measures rancidity, increased with storage. The relatively higher peroxide values of oil samples extracted with polar solvents with storage were still lower than 20 mg/kg allowed for use olive oil (FAO/WHO, 1993). Lower values imply that the oil has lower degree of rancidity.

The rate of increase which was also observed in thiobarbituric acid of oils related to extraction solvents and perhaps to Vitamin E content of the oil as antioxidant. This trend has been attributed to the fact that products formed during the reaction tend to catalyze the rate of autoxidation reaction. Lee (1983) reported that the reaction increases exponentially.

Conclusion

Most of the studies reported in this favoured the use of non polar (hexane) solvent as an extraction solvent. The extraction of oil with hexane gave highest oil yield but the use of such oil has been questioned in terms safety. It is recommended above other physical and chemical properties for use in industries other than Food. However the oil extraction with acetone a polar solvent is recommended for Food use on safety grounds.

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