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EXTRACTION AND PHYSICOCHEMICAL ANALYSIS OF OIL EXTRACTED FROM PINEAPPLE (*ANANAS COMOSUS*) PEELS

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ABSTRACT

The extraction of oil from waste pineapple (Ananas comosus) peels was carried out to determine if the oil extracted from pineapple peels is edible and/or can be utilized in industries by determining it's physicochemical properties. The Pineapple peels were collected from different locations in Amassoma, Bayelsa State. The sample was sun dried for five days and oven dried for 1hr: 30mins, then ground thoroughly. The sample was dipped inside n-hexane for 72 hours and later placed in a water bath for the evaporation of the solvent. Tests on Free Fatty acid, (FFA), Saponification Values, (SV), Iodine Value(IV), Peroxide Value(PV), and Acid Value were carried out in triplicates and the mean results were obtained; FFA = 2.068 mg/KOH/g, SV = 226.27mg/KOH/g, IV = 37.4355g/100g, PV = 7.666meq/kg, AV = 4.11532mg/KOH/g. In comparing these physicochemical parameters with the standards given by the Nigerian Industrial Standards (NIS), it was observed that the content of FFA was above the range, SV and IV were below the range, PV was within the range and AV was above the range. In Conclusion, it can be said that the oil is fit for human consumption because SV, IV, and PV are within the range stated by (NIS), whereas the excess of FFA and AV are good for industrial purposes and can be utilized.

KEYWORD: Pineapple peels, waste, oil, physiochemical parameters, consumption.

1.0 INTRODUCTION

Waste disposal represents a serious problem to many agro industries since it is usually prone to microbial spoilage and causes major environmental problems. The utilization of agro-industrial waste by conversion into value added products is an innovative solution to the environmental waste problem. Agro-industrial wastes in recent times have been the focus of research in drug design and in treating a range of ailments (Omwango *et al.*, 2013). Pineapple is the common name of *Ananas comosus* (*Syns. A. sativus, Ananassa sativa, Bromeliaananas, B. comosa*). Pineapple is the leading edible member of the family Bromeliaceae, grown in several tropical and subtropical countries including Philippines, Thailand, Indonesia, Malaysia, Kenya, India, China and South America. It has been used as a medicinal plant in several native cultures (Mondal *et al.*, 2011) and these medicinal qualities of pineapple are attributed to bromelain, which is a crude extract from pineapple that contains, among other compounds, various closely related proteinases, exhibiting various fibrinolytic, antiedematous, antithrombotic, and anti-inflammatory activities *in vitro* and *in vivo*.

1.1 Pineapple Fruit

Pineapple (*Ananas comosus L.*) known as the fruit of kings, because, despite the fact that the fruit had become familiar to almost the whole world, only the natives of the tropics and the wealthy Europeans, can have access to the fruit. It is still a true exotic, because it is a member of the bromeliad family, in which edible fruits are rare. (Lawal, 2013) reported that the solvent extracts of the various parts of *A. comosus* exhibit antibacterial, antiviral, antifungal, antiparasitic and anti-inflammatory properties.

The flavours in pineapple are due to the presence of small quantity of esters and essential oils distributed throughout the pulp. The presence of organic acids also contributes to the flavours. The nature and quantity of sugar present in fruits depends largely upon the ripeness and the degree of acidity (Yusuf *et al.*, 2012).

When the pineapple fruits are canned or consumed the crown, the outer peel and the central core are usually discarded as pineapple waste which accounts for about 50% of the total pineapple fruit weight corresponding to about ten tons of fresh pineapple or one ton of dry pineapple waste per hectare. Pineapple wastes are recommended as tremendous sources of organic raw materials and are potentially available for conversion into useful products (Martìnez *et al.*, 2012). It contains high amounts of crude fiber and suitable sugars for growth of microorganisms (Hemalatha and Anbuselvi, 2013). Pineapple waste contains mainly peel and pomace, presenting more than one third of the whole fruit mass (Huang *et al.*, 2011) and is only partially utilized as feed or composting for fertilization but mainly discarded, creating environmental issues such as pollution and consequently economic loss. However, these by-products have been reported to contain dietary fibers, sugars, proteins, and minerals (Huang *et al.*, 2011), and availability of these nutrients greatly depends on the recovery processes such as drying techniques employed in the preparation of powder. The drying techniques, namely oven drying and freeze drying have significantly different effects on the functional properties like, solubility, flow behaviours, water- and oil-holding capacity, and foaming capacity of the dried powder (Mirhosseini and Amid 2013).

1.2 Essential Oil and their applications

Essential oils are also used for massages as mixtures with vegetable oil or in baths. In addition, essential oil have a strong psycho-emotional effect and are used for aromatherapy. Some essential oils appear to exhibit particular medicinal properties that have been claimed to cure organ dysfunction or systemic disorder. In nature, essential oils play an important role in the protection of the plants as antibacterial, antivirals, Antifungals, insecticides and also against herbivores by reducing their appetite for such plants. They may also attract some insects to favour the dispersion of pollens and seeds, or repel undesirable others.

Due to their bactericidal and fungicidal properties, they are also used as alternatives to synthetic chemical products to protect the ecological equilibrium without showing the same secondary effects (Hammer et al, 2003). Known for their antiseptic and medicinal properties and for their fragrance, they are used in embalmment, preservation of foods and as antimicrobial, analgesic, sedative, anti-inflammatory, spasmolytic and locally and aesthetic remedies. In those cases, extraction by steam distillation is preferred. On the contrast, for perfume uses, extraction with lipophilic solvents or with supercritical carbon dioxide is favored. Most of the commercialized essential oils are chemo typed by gas chromatography and mass spectrometry analysis.

1.3 Quality of vegetable oil

Vegetable oils constitute a significant part of the human diet as a source of energy, fat-soluble vitamins and essential fatty acids (FAO, 2009).They are extracted from seeds and nuts of various plants and are composed mainly of triacylglycerols. The quality of any oil is indicated by some physicochemical properties such as the Iodine number, Saponification number, Peroxide value and free fatty acid value (Angaye and Maduelosi, 2015). The specific value of some of these properties provides an indication of both the nutritive and physical quality of the oil. For example, oils with low melting point may readily go rancid due to the high level of unsaturations. Edible vegetable oils are water insoluble natural product obtained from the seeds, kernels, nuts and pulps of various plants. They are extracted from plant parts mainly by mechanical pressure and solvent extraction. Generally oils are liquids at room temperature with some exceptions.

Various literatures of the physicochemical properties and qualities of oil exist. Ajaiyeoba and Ekundayo, (1999) gave the Essential oil constituents of *Aframomum melegueta* (Roscoe) K. Schum. Seeds (alligator pepper) from Nigeria. The composition of the essential oil isolated from the seeds of *Aframomum melegueta* (Roscoe) K. Schum, by hydrodistillation was characterized by means of Gas Chromatography (GC) and Gas Chromatography–Mass Spectrometry (GC–MS). A total of 27 compounds constituting 98.6% of the volatile oil were identified. Two sesquiterpene hydrocarbons: humulene and caryophyllene made up 82.6% of the volatile oil, whilst their oxides amounted to a further 9.0%. Seventeen other mono- and sesquiterpenes accounted for only 1% of the volatile oil. Five non-terpenoids were detected in trace amounts only (<0.2%). Of the 27 constituents identified, 12 are reported as components of *A. melegueta* for the first time. Copyright © 1999 John Wiley & Sons, Ltd.

Diomandé *et al.*, (2012) gave the GC/MS Analysis of Essential Oil of Five *Aframomum* Species from Côte D'ivoire. The leaves and rhizomes of five *Aframomum*species, namely *A. elliotii*, *A. strobilaceum*, *A. geocarpum, A. longiscarpum*and*A. Sceptrum* were subjected for hydrodistillation carried out with Clevenger apparatus. Higher yields were found in the leaves varying between 0.28% and 0.42%, while the rhizome oil shown lower yields accounting for 0.13-0.19%. The results of the analysis of volatile oils by GC and GC/MS have showed 52 identified components with the total proportion ranged from 96.3to 97.9%. The chemical composition of the leaves oil was dominated by hydrocarbon compounds such as -pinene, -caryophyllene, -humulene, -selinene, -selinene and germacrene A. Meanwhile, the rhizomes oil characterized with oxygenated components, namely eucalyptol, linalool and caryophyllene oxide accompanied by a few hydrocarbon constituents.

Yusuf *et al.*, (2012) reported the production of Vinegar from Pineapple Peel. Their work was aimed at producing vinegar from fermented pineapple peel. The process was carried out in two-stage fermentation with baker yeast (*Saccharomyces cerevisae*) and other reagents. Pineapple peel was allowed to ferment for 48hrs for conversion of sugar to ethanol. Then a chanced approach was applied for the conversion of ethanol by acetic acid bacteria (Acetobacteraceti) to vinegar with continuous aeration for nine days. The results indicated that vinegar yield increased with an increasing acidity. The results also revealed that pineapple peel produced the desired and optimum yield of vinegar. Hence, the following parameters; pH, density, refractive index, viscosity, % acetic acid and acid value were evaluated and recorded as; 2.80, 1.08 g/ml, 1.390, 0.94cp, 4.77 and 0.0477 respectively, these values compare well with the standard values. The conversion of pineapple peels (waste) to vinegar (useful product) will reduce environmental pollution and in addition yield value added product.

Angaye *et al.*, (2013) studied the effect of heat on the physicochemical properties of Groundnut Oil. Oil was extracted from fresh groundnut seeds (used as control) and groundnut seeds heated at 90 ^{0}C and 120 ^{0}C, and market roasted groundnut seeds (fried and bottled seeds purchased from a local market) using n-hexane. The various oil samples were analyzed for their relative densities, viscosities, refractive indices, iodine values and peroxide values. The authors showed that oxidative rancidity of the oil samples increased with increase in temperature after the physicochemical parameters were evaluated.

Arinola *et al.*, (2013) studied physicochemical characteristics and the effect of packaging materials on the storage stability of selected cucurbits oils. Physicochemical properties and the effect of packaging materials on the quality of oil extracted from *Cucumeropsismanni, Lagenariasicceraria* and *Citrulluslanatus*were studied in this work. After the extraction of the oils, the oil samples were stored in amber glass bottle, transparent glass bottle and transparent plastic bottle at ambient temperature for 12 weeks during which stability of the oil to oxidative and hydrolytic deterioration was assessed by determining peroxide value and free fatty acid value at 2 weeks interval. The authors concluded that the storage stability shows that the oil samples may be relatively stable to oxidation and hydrolysis when properly stored in a good packaging material and that oil samples stored in plastic transparent bottle recorded highest lipid peroxidation values while amber glass bottle gave the maximum protection against lipid peroxidation.

Onyinye *et al.*, (2014) evaluated Tiger nut oil (Cyperusesculentus L.): A review of its composition and physicochemical properties. Despite its high nutritional value, tiger nut oil is hardly used in food industries compared to other vegetable oils such as olive and peanut oil. However, it's benefits are increasingly being recognized including its stability and similarity to olive oil in particular. This review discusses its composition, physicochemical properties and economic potential. Literature reveals that tiger nut oil shares a similar fatty acid profile with olive, avocado and hazelnut oil. Its low content of polyunsaturated fatty acid, tocopherol and phytosterol contributes to its high stability. When compared to soy bean oil, its phospholipids composition (3.1–5.4%) is higher. Its iodine and acid values reflect its quality at 76.60 and0.03 mg KOH/g oil, respectively. Roasted tiger nut oil was found to contain vanillin and to a lesser extent2, 3-dihydro-3, 5-dihydroxy-6-methyl-4H-pyran-4-one and 5-ethylfurfural as key odorants contributing to the overall aroma of the oil. Its low viscosity makes it suitable for use in coating industry and as fuel.In-depth studies on the oils' constituents are on-going but efficient and environmentally friendly extraction techniques are needed. Based on the available data, tiger nut oil has been established as an oil of good nutritional value which may be exploited to the great benefit of growers, processors and dealers of the tuber.

Angaye andMaduelosi, (2015) studied physicochemical Properties of some refined vegetable oil sold in Mile one market and Some Departmental Stores in Port Harcourt, Rivers State, Nigeria. Physiochemical properties of some brands of edible oil (namely:- Turkey, Grand, Gino, tropical, Power, Mammador, local vegetable oil and kuli-kuli (raw/locally extracted groundnut oil) sold in mile 1 market, Everyday supermarket and Next-time supermarket in Port Harcourt, Rivers state were studied to determine their quality and compare the results with some standards. All the oils were characterized for flash point, cloud point, iodine value, saponification value, unsaponifiable matter, acid value and peroxide value using standard method of analysis. The results obtained show that the free fatty acid contents of the local and kuli-kuli oils were higher (0.709 and 1.96 respectively) than the maximum recommended value of 0.3 by SON and NIS while other brands were within the range. The measured moisture content values for Turkey, Grand, Gino and Mammador were higher than the standard range of 0.05 in the samples from Mile 1 market and in some supermarkets. Specific gravity, melting point, and cloud point were satisfactory except for Grand that showed a different cloud point of less than 2. Iodine value for the local oil is 20.25 (Wij's) indicating the predominance of lauric acid while that of kuli-kuli (94.6 Wij's) suggests the predominance of Oleic acid. The other brands showed iodine value of 46-50Wij's which indicates predominance of palmitic acid except the Grand oil that had the highest value of 125-128(Wij's). Saponification values and unsaponifiable matter were within the standard range of saponification value (245- 255) and unsaponifiable matter (0.1-0.15) for all the brands. Acid and Peroxide values of all the brands showed results within the standard. These results of the measured parameters show that the oils sold in the supermarkets are better protected from light induced oxidation (photoxidation) than those sold in open markets.

Fengfeng*et al.*, (2017) studied the electrofluidic pretreatment for enhancing essential oil extraction from citrus fruit peel waste. Pretreatment technologies make pressed fruit peel more valuable both nutritionally and economically. Robust electrofluidic systems, in particular, can facilitate clean and efficient production of valuable compounds from these byproducts. In this study, a multi-series system was designed with a combination of pipeline flow and induced electric field (IEF), then applied to pretreat citrus peel for enhancing the extraction of essential oils. The essential oil yield increased significantly from 0 V to 1 kV. There was a decrease in fluid impedance during the IEF-pretreatment period owing to the dissolution of cell contents, as well as a slight decrease in essential oil yield as frequency increased. Forty components were identified in the essential oils representing over 97% of the total GC peak areas for both procedures. We also observed significant effect on the essential oil yield between the IEF-pretreatments in different winding direction of the sample coils. High throughput was achieved by arbitrarily increasing the reactors within a compact system. In effect, the IEF-pretreatment is an effective, innovative, and ecologically sustainable technique for intensifying essential oil extraction without necessitating metal electrodes, thus avoids electrode surface corrosion, and heavy metal contamination.

3.0 MATERIALS AND METHODS

3.1 Materials

3.1.1 Reagents

Methanol (CH_3OH), sodium thiosulphate ($Na_2S_2O_7$), potassium iodide (KI), iodine crystals, glacial acetic acid (CH_3COOH), hydrochloric acid (HCl), sodium hydroxide (NaOH) and phenolpthalein, Wijs reagent.n-hexane.

3.1.2 Plant used: Pineapple peels were obtained from a nearby fruit store in Amassomma Wilberforce Island, Bayelsa state. Nigeria.

3.2 Method

3.2.1 Oil Extraction Procedure

The hexane extract was obtained by complete extraction using cold extraction technique. 100g of the powdered sample was put into an air tight extraction bottle and 200 cm^3 of n-hexane as extracting solvent was used to soak the sample for 72 hours after which it was decanted. The oil was obtained after evaporation using Water bath at 35°C to remove the excess solvent from the extracted oil. The oil was then stored in refrigerator prior to the analysis proper.

3.2.2 Physicochemical properties

The iodine, acid and peroxide values were determined according to method as prescribed by FAO (2009) and AOAC (1990). The saponification value and free fatty acid value were determined according to the AOAC official procedure. Each experiment was repeated three times and the data obtained was statistically analyzed.

3.2.2.1 Free fatty acids (FFA): Free fatty acids value was determined according to the method describe in AOCS method (AOCS, 1992). 1.0 g oil of the sample was mixed with 25 ml of 95 % neutral ethyl alcohol and swirled. Phenolphthalein was added as indicator. The solution was titrated with 0.1 N sodium hydroxide until pinkish colour was observed at end point. FFA concentration in fats and oils is calculated as percentage oleic acid. The expression as given in

AOCS Official Method by AOCS, (1992) as:

$$\% \text{ FFA as oleic acid} = \frac{\text{ml of alkali X N alkali X 28.2}}{\text{sample wt}}$$

% FFA as oleic acid * 1.99= acid value

3.2.2.2 Saponification Value (SV): The saponification value (SV) of the various oils was determined following procedures described in AOCS method (AOCS, 1992). Oil sample (1 g) was dissolved in 20 ml of 0.5 N ethanolic potassium hydroxide. The mixture was refluxed for 30 minutes until oil droplets disappear and was left to cool to room temperature.

Phenolphthalein indicator was then added and the hot soap solution was titrated with 1 N HCl until the pink colour disappears. A blank titration was also carried out in the same manner except that no oil was added. Saponification value was calculated using the formula:

$$sapvalue = \frac{(S - B) \text{ x N x } 56.1}{\text{sample wt}}$$

Where; a = Volume (ml) of 1 mol/l hydrochloric acid consumed in the blank test,

b = Volume (ml) of 1 mol/l hydrochloric acid consumed in the test, N = Normality of hydrochloric acid, W = Weight of oil sample, g.

3.2.2.3 Iodine Value (IV): About 0.1g sample was delivered to a 300 mL conical flask with ground-in stopper and mixed with 20.0mL carbon tetrachloride and sealed. It was dissolved in an ultrasonic washing machine.

Twenty five millimetres Wij's solution was added and sealed. It was shook for one minute, Kept sealed and left to stand for a specific period. 5.0mL of 15% potassium iodide was added, sealed and Shook for 30 seconds. The mixture was titrated with 0.1mol/L sodium thiosulfate to obtain iodine value. Likewise, blank test was performed to obtain blank level (AOCS, 1992).

$$Iodinevalue = \frac{(B - S)x\ N\ x\ 12.69}{\text{sample wt}}$$

Where; a =Volume (ml) of 0.1 mol/l sodium thiosulfate consumed in the blank test,
b = Volume (ml) of 0.5 mol/l sodium thiosulfate consumed in the test, N = Normality of sodium thiosulfate, W = Weight of sample.

3.2.2.4 Peroxide Value (PV): Peroxide value is determined as follows (AOAC method 965.33; AOCS method cd 8-53; AOCS method 8b-90): Dissolve 5.0g of fat or oil in 30ml of glacial acetic acid-chloroform (3:2, v/v).Add 0.5ml of saturated KI. I_2 is liberated by reaction with peroxides (eq.11).Titrate with standardized sodium thiosulphate using a starch indicator (eq.12). peroxide value is then calculated using the equation below.
H^+, Heat

$$ROOH + K^+I^- \text{ (excess} \longrightarrow ROH + K^+OH^- + I_2$$

$$I_2 + \text{Starch} + 2Na_2\,S_2O_3 \longrightarrow Na_2S_4O_6 \text{ (colorless)} + \text{Starch}$$

$$peroxide\ value = \frac{(B - S)\ x\ N\ x\ 1000}{\text{sample wt}}$$

Where S= sample titration, B=blank titration, N=normality of $Na_2S_2O_3$ solution.

3.2.2.5: ACID VALUE(AV) 5mg of the sample was weighed into a 250mL Erlenmeyer flask followed by addition of 50mL alcohol-ether mixture and 0.1mL phenolphthalein solution. It was then titrated with 0.1M alcoholic KOH until a steady faint pink appeared. Acid Value = mL alcoholicKOH solution X molarity alcoholic KOH X 56.1/g test portion (AOAC).

4.0 RESULTS AND DISCUSSION

4.1 Results

The results obtained from the various analyses are displayed in Table 1 below. The results were arranged in triplicates and the means obtained.

Table 1: Showing the physicochemical parameters of pineapple peels oil.

Parameters	FFA	SV	IV	PV	AV
1ST	2.0304	258.06	44.415	6.000	4.040496
2ND	2.256	207.57	31.725	9.000	4.48944
3RD	1.9176	213.18	36.1665	8.000	3.816024
Mean	2.068	226.27	37.4355	7.666	4.11532
NIS Standard	0.3 max	245-255	45-55	7-10 (depending on the oil)	0.6max

4.2 DISCUSSION

Peroxide value measures a transient product of oxidation. A low value may represent either the beginning of oxidation or advance oxidation. Peroxide value measures the degree of lipid oxidation in fats and oils but not their stability. Peroxide value is defined as the milliequivalent (meq.) of peroxide per kg fat, it is a measure of the formation of peroxide or hydroperoxide groups that are the initial products of lipid oxidation. The peroxide value (meq/kg) of pineapple peel oil determined in triplicates had a mean value of 7.667meq/kg. Similar result was observed for water melon oil by Adejumo *et al.*, (2015), a range of 2-16meq/kg was observed. The peroxide value obtained is within the range for oil as specified by SON (2000) and NIS (1992)which is 7-10 meq/kg. The lower peroxide value of pineapple peel oil may show that it will not easily go rancid which is related to its longer shelf life and stability.

The free fatty acid value measures the extent to which fatty acid hydrolysed. Rancidity is usually accompanied by free fatty acid formation; the determination is usually used as general indication for the condition and the edibility of the oil. The mean of the triplicate value obtained is 2.068 mg/KOH/g which is much higher than the recommended range of 0.3max by SON (2000) and NIS (1992). This is an indication that the extent of hydrolytic rancidity in these oils is appreciable.

The saponification value (mg/KOH/g) of pineapple peel oil was determined in triplicates, and a mean value of 226.27mg/KOH/g was obtained. The value is within the recommended SON (2000) and NIS (1992) standard. Adejumo *et al.*, (2015) in their study of oil extracted from water melon seed found the saponification value to be in the range 196.35-325.38mg/KOH/g. The saponification value gives information about the fatty acid present in the oil and the solubility of the soap derived from it in water. The smaller the saponification number, the longer the average fatty acid chain length. This is an indication that pineapple peel oil could be used for industrial purpose.

The iodine value (g/100g) determined in triplicates had a mean value of 37.4355g/100g. Onyinye *et al.*, (2014) evaluated Tiger nut oil (Cyperus esculentus L) and found the iodine value to be 76.60g/100g.The value is lower than the stipulated SON (2000) and NIS (1992) standard of 45-55g/100g. The iodine value of oil is a measure of the unsaturated acid present; this also indicates its non-drying qualities. Therefore, the test measures the amount of iodine consumed by the acid. The greater the iodine value, the greater the unsaturation and hence the greater the liquidity. The lower value indicates lower degree of unsaturation.

The acid value (mg/KOH/g) of pineapple peel oil was determined by multiplying the %FFA value by 1.99.The mean acid value was observed to be 4.11532mg/KOH/g, which is also higher than the stipulated SON (2000) and NIS (1992) standard value of 0.6maxmg/KOH/g. Onyinye *et al.*, (2014) found the acid value of tiger nut to be 0.03 KOH/g oil. Acid value is an important index of physicochemical property of oil which is used to indicate the quality, age, edibility and suitability of oil for use in industries such as paint (Akubugwo et al 2008)

5.0 CONCLUSION

Physicochemical properties of oils helps to ascertain the quality of the oil we use whether for food or for industrial purpose. The pineapple peel oil as compared with other oil extracted from other plants showed good use both for edible purpose and for industrial usage as the physicochemical properties obtained were found to be in range stipulated by SON and NIS. It is therefore of high quality and should be utilized.

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