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Comparative Analysis of Oil Extraction from Clove and Ginger using Maceration and Soxhlet Methods: Physicochemical Properties and Quality Assessment

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Abstract

This study examined oil extraction from clove and ginger using two methods: Maceration method and Soxhlet extraction method. The materials (clove and ginger) were purchased locally from a credible vendor at Erekesan Market, Akure, Ondo State. The oils' physicochemical properties analyzed included pH, color, specific gravity, impurity content, and moisture content. Additionally, the Free Fatty Acid (FFA) value, acid value, Thiobarbituric acid (TBA) value, saponification value, and FFA profile were assessed. The pH (4.52 - 6.08) and specific gravity (0.85 - 0.97) were well within the accepted range. The oils' hues deviated from those documented in prior studies, likely due to the elevated temperature exposure during extraction. The impurity content varied from acceptable (7.07%) to unacceptable (21.59%) for edible oils. The moisture contents (1.37 - 8.88%) indicate low susceptibility to microbial growth. The commercial ginger oil (control) exhibited the lowest FFA (5.51 g/100ml) and acid value (10.96 g/100ml), indicating better stability and resistance to rancidity while the clove oil (Soxhlet method) had the highest FFA (10.10 g/100ml) and acid value (20.10 g/100ml), therefore it has the least desirable quality. The oils' TBA values (0.0024 - 0.0025 mgMDA/g) indicate enhanced stability and extended shelf life while their saponification value (80.15 - 196.08 mg/g), indicate varying but ideal FFA molecular weights. The oil samples showed diverse FFA profile: the higher ΣSFA composition (73.58%) of the commercial ginger oil (control) implies an undesirable quality as it has a higher risk of heart disease when consumed in large quantity while the ginger and clove samples extracted using maceration exhibited desirable ΣMUFA (22.99%) and ΣPUFA (22.78%) compositions. These affirm the influence of the extraction methods on the quality indices of the oils. Based on the analyses conducted, it is concluded that the maceration extraction method is superior in producing higher-quality oils.

Keywords: *Clove, Ginger, Essential oils, Oleoresins, Extraction, Physicochemical properties*

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1. Introduction

The extraction of oils from herbs and spices, such as clove and ginger, has gained significant attention due to their beneficial properties and ability to impart flavor in various food and pharmaceutical applications. These oils are commonly extracted in the form of oleoresins and essential oils, which contain concentrated amounts of bioactive compounds responsible for their characteristic aroma and taste. The extraction process plays a crucial role in obtaining high-quality oils with optimal yield and desired sensory attributes (Grabowska *et al*., 2022). Several studies have been conducted to optimize the extraction procedures and evaluate the cytotoxic, antioxidant, and antimicrobial potentials of these plant extracts (Chen *et al*., 2017; Suo *et al*., 2018). This background study aims to provide a comprehensive overview of the extraction processes, bioactive compounds, and potential applications of oils derived from clove and ginger.

The extraction of oils from herbs and spices involves the separation of desired compounds from the plant matrix using various techniques. Commonly employed methods include steam distillation, solvent extraction, and supercritical fluid

extraction (Grabowska *et al*., 2022). Steam distillation is widely used for the extraction of essential oils from aromatic plants, including clove and ginger, as it allows for the efficient extraction of volatile compounds without degradation (Chen *et al*., 2017). Solvent extraction techniques, such as maceration and Soxhlet extraction, utilize organic solvents to extract both lipophilic and hydrophilic compounds from plant materials (Suo *et al*., 2018). Supercritical fluid extraction, utilizing carbon dioxide as the solvent, offers advantages such as high selectivity and low environmental impact (Grabowska *et al*., 2022). The composition of oils extracted from clove and ginger is primarily governed by the presence of bioactive compounds, including phenolics, terpenes, and alkaloids. These compounds contribute to the characteristic flavor and aroma of the oils and exhibit various biological activities. Clove oil, for instance, is rich in eugenol, a phenolic compound known for its antioxidant, antimicrobial, and anti-inflammatory properties (Grabowska *et al*., 2022). Ginger oil contains gingerols and shogaols, which possess anti-inflammatory, anti-cancer, and anti-diabetic activities (Chen *et al*., 2017). The extraction process influences the yield and composition of these bioactive compounds, and optimization of extraction parameters can lead to the production of oils with enhanced functional properties (Suo *et al*., 2018). The applications of oils extracted from clove and ginger extend beyond the culinary realm. These oils have been extensively studied for their pharmacological activities and have shown potential in the treatment of various diseases. The antioxidant properties of clove and ginger oils make them valuable in combating oxidative stress and reducing the risk of chronic diseases, such as cardiovascular diseases and cancer (Grabowska *et al*., 2022). The antimicrobial properties of these oils have been explored for their potential in food preservation and as natural alternatives to synthetic antimicrobial agents (Chen *et al*., 2017). Furthermore, the bioactive compounds present in these oils have demonstrated promising effects in the management of diabetes, inflammation, and gastrointestinal disorders (Suo *et al*., 2018). There are different reasons why it is crucial to conduct a study on the extraction of oleoresins and essential oils from herbs and spices. Previous studies have indicated that essential oils are extremely valuable, with a vast growing market worldwide. Their multiple uses make them gold for different types of industries (Mutshinyalo, 2018). Oleoresins and essential oils from herbs and spices are often utilized in the food sector as flavouring agents. Studying their extraction may result in the creation of novel flavours or the enhancement of already-existing ones (Leyva-López *et al*., 2017). Therefore, continued research in this area is crucial. It can lead to the development of more efficient and sustainable extraction methods, enhance our understanding of the factors influencing oil composition and yield, and ultimately contribute to the production of high-quality, flavor-enhancing essential oils and oleoresins. The main objective of this research is to extract oils (oleoresins and essential oils) from clove and ginger using Maceration (cold solvent extraction) and Soxhlet (hot solvent extraction) extraction methods. The specific objectives of this research are to compare extraction methods, analyze the composition of the extracted oils and determine the physical and chemical characteristics of the extracted oils.

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2. Materials and Methods

2.1 Sample collection

The herb (Clove, Syzygium aromaticum), spice (Ginger, Zingiber officinale) and ginger oil (control) were purchased locally from a credible vendor at Erekesan Market, Akure, Ondo State. The extraction solvent, N-hexane, was purchased from Delson Pascals Ventures Ltd., Akure, Ondo State. All other reagents were of analytical grade.

2.2 Ginger and Clove Oil Extraction

2.2.1 Maceration

The maceration extraction of each sample (clove or ginger) was carried out using a modified method of Singh (2021). 300g of the coarsely ground material was placed in a sealed container with the solvent (n-Hexane of about 4:1 v/w of the sample). It was then left to stand at room temperature for 3 days, during which the mixture was frequently agitated until the soluble matter had fully dissolved. The mixture was strained to separate the liquid (solvent + oil) from the marc (the damp solid material) which was then pressed to extract any remaining liquid. The combined liquids were left to stand for a period to settle any remaining solids. The liquid was then carefully decanted and filtered to remove any remaining solids, resulting in a clarified liquid. To further purify the oil and separate it from the solvent, a rotary evaporator was used (Fig, 2). The oil was stored in an amber bottle for analysis.

2.2.2 Soxhlet extraction

The Soxhlet extraction was carried out for each sample according to Aryal (2022). 300g of the ground sample was placed in a thimble-shaped filter cloth and inserted into the Soxhlet extractor thimble. The device was then assembled, and the solvent (n-Hexane of about 3:1 v/w of the sample) was added to the reservoir flask before being placed on a heating mantle. Upon heating, the condensed vapors of the solvent came into contact with the sample powder, mixing the soluble part of the powder with the solvent for extraction. When the solvent surface surpassed the siphon's maximum height, the solvent containing the extract was siphoned back into the flask. The sample was allowed to reflux for 8hrs at a regulated temperature of 55 – 60°C. The heating was stopped when the reflux time was complete. The solvent was evaporated and the extracted oil concentrated in the flask. The oil was stored in an amber bottle for analysis.

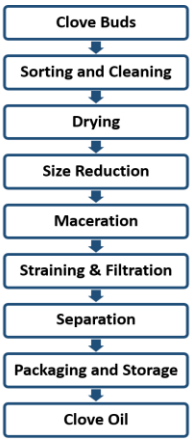

Figure 1: Process flowchart for maceration extraction of clove oil (Mac & Mac, 2022).

Figure 2: Rotary evaporator for separation of oil from solvent.

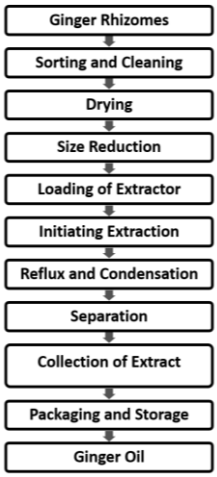

Figure 3: Process flowchart for soxhlet extraction of ginger oil (Yuharmon *et al.*, 2018).

Figure 4: Extracted ginger and clove oils.

2.3 pH determination

The pH values of the oils were determined using an electronic pH meter as described by AOAC (2023). The instrument was switched on, and the electronic components were allowed to warm up and stabilize. The instrument and electrodes were standardized using a commercially prepared standard 4.0 pH buffer. The electrodes were rinsed with water and blotted with soft tissue. The tips of the electrodes were immersed in the buffer solution, and a pH reading was taken, allowing about 1 minute for the meter to stabilize. The standardization control was adjusted so that the meter read exactly 4.0 for the observed temperature. The electrodes were rinsed again with water and blotted with soft tissue. The electrodes were then immersed in the sample, and a pH reading was taken, allowing 1 minute for the meter to stabilize. The electrodes were rinsed and blotted, and this process was repeated on a fresh portion of the sample (2 pH values were determined on the well-mixed sample to ensure accurate and consistent pH readings for the samples).

2.4 Specific gravity

The method of determining the oil's specific gravity, as outlined by Jolayemi and Alagbe (2022), involved comparing its density to that of an equivalent volume of water. A pycnometer, thoroughly cleaned, was filled with distilled water, wiped clean, and then weighed. This process was repeated, but this time, the water was substituted with the oil sample. The specific gravity of the oil was then computed using the formula:

$$\text{Specific gravity} = \frac{\text{Weight of the oil (g)}}{\text{Weight of distilled water (g)}}$$

2.5 Colour determination

This analysis was carried out through the visual assessment of the colour of the oils as described by Pathare *et al*. (2012). The colour of each sample was observed without instruments (with the eyes), under controlled conditions of illumination, and compared with a set of colour standards.

2.6 Impurity content determination

This analysis was carried out by identifying and quantifying foreign substances present in the oils as described by Rawat & Kumar (2017). 2 mL of the oil sample was accurately measured using a calibrated pipette. The solvent, ethanol, was added to the oil sample to dissolve impurities. The mixture was agitated to ensure thorough extraction of impurities from the oil. The solution was filtered to remove any particulate matter or undissolved substances using a fine filter paper or membrane. The solvent from the filtered solution was evaporated using a rotary evaporator, leaving behind a concentrated impurity residue. The residue was re-dissolved in a small amount of a compatible solvent to obtain a concentrated impurity solution. Gas Chromatography (GC) was utilized for the quantitative determination of specific impurities. The instrument was calibrated with standard solutions of known impurity concentrations. The chromatographic data was analyzed to identify and quantify impurities present in the clove or ginger oil. The impurity content was calculated as a percentage of the total oil sample.

2.7 Moisture content determination

The procedure for determining the residual moisture in the oil involved estimating the moisture content of the oil samples using a drying oven as described by Jolayemi and Alagbe (2022). The oil's weight was measured before and after drying it at 105°C until it reached a constant weight. The difference in weight was then calculated and reported as the percentage of moisture removed. This was done using the following formula:

$$\%\ \text{Moisture content} = \frac{\text{Initial weight} - \text{Final dry weight (g)}}{\text{Initial weight (g)}} x100$$

2.8 Thiobarbituric Acid (TBA) analysis

The Thiobarbituric Acid (TBA) analysis was carried out as outlined by Zeb and Ullah (2016). 1 mL of the standard MDA solution was combined with 1 mL of TBA in a 10 mL test tube. This mixture was then heated in a boiling water bath at 95℃ for a duration of 60 minutes. Following the heating process, the test tubes were allowed to cool at room temperature. The absorbance was then measured at 532 nm using a UV-visible spectrophotometer model PharmaSpec 1700 (Shimadzu, Japan). This procedure was repeated 3 times for each standard used in the calibration. A blank sample was also prepared and the procedure was repeated 5 times, substituting the standard or sample with acetic acid or water. The oil samples were collected and two types of sample extracts were prepared; one with 100% glacial acetic acid (AA) and the other with a 50% mixture of glacial acetic acid and water (AW). For each sample, a 1 mL extract was combined with 1 mL of TBA reagent and the aforementioned procedure was repeated five times. The TBARS was then calculated as mgMDA/g of the sample using the following formula:

$$\text{TBARS (mgMDA/g)} = \frac{Ac}{W} xV$$

Where Ac represents the amount determined from the calibration curve, W is the weight of the sample taken, and V is the volume in mL or the dilution factor of the total extract prepared.

2.9 Colour index determination

The colour properties of the samples were evaluated using a colorimeter, following the methodology outlined by Pathare *et al*. (2012). Utilizing a portable tri stimulus reflectance colorimeter, the color parameters, specifically the Hunter L*, a*, and b* values, were determined. In this system, L* represents the lightness of the color, a* denotes the chromaticity ranging from green (-) to red (+), and b* signifies the chromaticity from blue (-) to yellow (+).

2.10 Free Fatty Acids (FFA) determination

The Free Fatty Acid content was determined as described by Francilia *et al*. (2020). Twenty-five milliliter (25ml) of neutral ethanol was heated to boiling and added to 1g of each oil extract to dissolve in a conical flask. The heating was stopped and the solution was titrated with 0.1M potassium hydroxide (KOH) solution using phenolphthalein as indicator. The FFA was calculated as follows:

$$\text{FFA (g/100ml)} = \frac{\text{Vol of KOH used (ml)} \times \text{KOH normality (N)} \times \text{Equiv wt of FA (g/equiv)}}{\text{Wt of sample (g)} \times \text{Vol of sample for titration (ml)}} x100$$

2.11 Acid value

The acid value was determined from the free fatty acid value as described by FSSAI (2016). It was determined from the expression:

$$\text{Acid value (g/100ml)} = \text{free fatty acid value (g/100ml)} \times 1.99$$

2.12 Saponification value

The Saponification Value (SaV) of the oils was calculated as outlined by Jolayemi and Alagbe (2020). In summary, a quantity of 1.5 – 2.0 g of the oil was combined with 25 mL of alcoholic KOH (0.5 N). This mixture was then refluxed and boiled for a duration of 15 – 20 minutes until the oily matter vanished, signifying complete saponification. Alongside the sample, a blank determination was also performed. The resulting clear solution was cooled and then titrated against 0.5 N HCl until the pink color, indicated by phenolphthalein, disappeared. The SaV was then calculated using the following formula:

$$\text{Saponification Value (mg KOH/g oil)} = \frac{56.1 \times \text{N} \times (\text{B} - \text{S})}{W}$$

Where, B and S represent the volumes of HCl required for the blank and the sample, respectively. N stands for the normality of HCl, and W is the weight of the sample (g).

2.13 Fatty acid distribution – Gas Chromatography (GC) analysis

The initial esterification of the fatty acids in the oil samples was performed as outlined by Jolayemi and Alagbe (2022). 0.1 g of the oil sample was dissolved in 10 mL of n-hexane and 100 µL of 2N methanolic potassium hydroxide (2.8 g KOH in 25 mL methanol). The mixture was then vortexed and centrifuged, and the aqueous phase was microfiltered using a 0.45 µm filter, preparing it for Gas Chromatography (GC) analysis. The esterified oil samples were then characterized for their fatty acid methyl esters (FAME) using a HP 6890 Gas Chromatograph, powered by HP ChemStation Rev. A09.01 – 1206, and equipped with a Flame Ionizing Detector (FID). The stationary phase was a capillary column (HP INNOWax, 30 m x 0.25 mm I.D, 0.25- micron dry film), and the mobile phase was nitrogen gas with a flow rate of 1 mL/min.

The oven was initially set to a temperature of 60°C. The first ramping was set at 12°C/min for 20 min and maintained for 2 min, while the second ramping was set at 15°C/min for 3 min and maintained for 8 min. The split injector (with a 20:1 split ratio) and detector were set at 250°C and 320°C, respectively. The individual fatty acids were identified by comparing the sample chromatograms to that of a FAME standard, based on the retention times and peak integrations. A Chromatogram-simulator (NIST, version 1.0.0.0) was used for easier and clearer peak visualization.

2.14 Statistical Analysis

The model's fit quality was assessed using the Analysis of Variance (ANOVA) method. The SPSS statistical software (Version 17.0) was utilized to analyze the results through ANOVA. The Duncan New multiple range test was employed to determine significant differences in the sample means at a p-value less than 0.05. All values were reported as mean standard deviation.

3. Results and Discussion

The physical and chemical attributes of oils play a vital role in their utilization across various fields such as culinary, cosmetics, and in industries. Table 1 shows the several important physicochemical parameters, including pH, Moisture Content, Impurity Content, Specific Gravity, TBA

(Thiobarbituric Acid) Value, and Colour, that were analyzed in the oil samples.

3.1 pH

The pH value is a measure the relative acidity or basicity of an aqueous solution. Ginger oil extracted using Soxhlet extraction had the highest pH value (6.08) and least acidity while the commercial ginger oil (control) had the lowest pH value (4.52) and most acidity. The pH of the oil samples ranged between 4.52 and 6.08, falling well within the accepted range of 4.50 to 6.50 (Sembiring *et al*., 2023). This implies that the oils are of good quality in terms of acidity, ranging from acidic to slightly alkaline.

3.2 Specific gravity

The specific gravity is a measure of the mass of the oils per unit volume (density) relative to that of water (Jolayemi & Alagbe, 2022). The commercial ginger oil (control) showed the highest specific gravity (0.97), while both clove oil samples had the lowest (0.85). There were no significant differences ($p>0.05$) recorded between the ginger oil extracted using maceration and the two clove oil samples. However, the specific gravity of the five samples are within the ideal range of 0.85 to 0.99 (Godwin *et al*., 2019) with an average value of 0.91.

3.3 Colour

Upon visually inspecting the oils, it was observed that both ginger oils extracted using maceration and Soxhlet were amber brown, while both clove oils extracted using maceration and Soxhlet exhibited a dark brown hue. These findings contrast with Uddin *et al*. (2023) who recorded pale yellow to amber for ginger oils, Godwin *et al*. (2019), who reported a pale yellow colour for ginger oils and Adjal *et al*. (2023) who reported pale yellow to green for clove oils. This may be due to the somewhat increased temperature treatment during the Soxhlet extraction and solvent-oil separation using the rotary evaporator.

3.4 Impurity content

Impurity content identifies and quantifies foreign substances present in the oil that are not part of its natural composition (Rawat & Kumar, 2017). These impurities have the potential to affect the characteristics of the oils and their suitability for various applications (Vaisali *et al*., 2015). Clove oil (Soxhlet) demonstrated the highest impurity level (21.59%), whereas ginger oil (maceration) showed the lowest (7.07%). The existence of impurities can affect the flavor, color, and overall quality of these oils. Rehan *et al*. (2017) emphasized the necessity for efficient removal of impurities, recommending the use of natural zeolites to enhance surface area and improve the quality of the tested oil samples.

3.5 Moisture content

Moisture Content refers to the quantity of water in a substance. It's critical in the context of essential oils or oleoresins since excess moisture may cause concerns like microbial growth or changes in consistency (Mohammed *et al*., 2022). Ginger oil (Soxhlet) exhibited the highest moisture content (8.88%), with Commercial ginger oil (control) recording the lowest (1.37%). The drawback of oil with high moisture content is its susceptibility to microbial growth and hydrolytic rancidity, which results from increased acidity and ultimately leads to a decline in quality. Nevertheless, all samples remain within the safe level recommended by the Food and Drug Administration (FDA).

3.6 TBA (Thiobarbituric Acid) analysis

Thiobarbituric Acid TBA is used to determine the degree of lipid oxidation (fat breakdown) in oils. Malondialdehyde (MDA) is released when fats degrade. The buildup of malondialdehyde in oils over time indicates increased lipid oxidation and affects their quality, as it is responsible for the generation of undesirable flavour and a rancid smell (Zeb & Ullah, 2016). There was no significant difference ($p>0.05$) between the clove oil (maceration) and commercial ginger oil (control), both of which exhibited the lowest TBA value (0.0024 mgMDA/g), indicating a better resistance to oxidative processes, while there were significant differences among the two extracted ginger oils (maceration and Soxhlet) and clove oil (Soxhlet), the three of which showed higher TBA values (0.0025 mgMDA/g).

Table 1: Physicochemical Properties of Ginger and Clove Oils under Two Extraction Procedures

Sample	L*	a*	b*	c*	h*
GA1	4.55 ± 0.04^{d}	5.66 ± 0.142^{b}	4.77 ± 0.08^{b}	7.41 ± 0.16^{b}	40.11 ± 0.29^{a}
ZB8	4.79 ± 0.02^{c}	5.07 ± 0.01^{c}	4.13 ± 0.02^{c}	6.54 ± 0.01^{c}	39.13 ± 0.14^{b}
CY5	7.63 ± 0.01^{b}	4.05 ± 0.02^{d}	2.52 ± 0.02^{e}	4.77 ± 0.02^{d}	31.87 ± 0.34^{d}
AD6	2.45 ± 0.01^{e}	3.57 ± 0.00^{e}	2.97 ± 0.02^{d}	4.64 ± 0.01^{d}	39.77 ± 0.18^{a}
E63	9.82 ± 0.01^{a}	7.60 ± 0.05^{a}	5.26 ± 0.05^{a}	9.24 ± 0.06^{a}	34.71 ± 0.11^{c}

$^{a-e}$ Same alphabet within the column are not significantly different ($p< 0.05$). Values are mean ± SME.
GA1= Ginger oil using Maceration, ZB8= Ginger oil using Soxhlet extraction, CY5= Clove oil using Maceration, AD6= Clove oil using Soxhlet extraction, E63= Control (commercial ginger oil).
a* negative and positive values indicate, respectively green and red.
b* negative and positive values indicate, respectively blue and yellow.
L*= Lightness on a scale from 0 (black) to 100 (white)

3.7 Colour index

The colour characteristics of the samples were evaluated using the Hunter Lab colour system. In this system, L* denotes lightness, a* indicates the degree of redness, and b* signifies yellowness. As per the data in Table 2, the L* values for the samples varied between 2.45 and 9.82. The commercial ginger oil (control) exhibited the highest L* value, indicating it is the lightest in colour, while the clove oil (Soxhlet) had the lowest L* value, making it the darkest. The a* values ranged from 3.57 to 7.60, with the commercial ginger oil (control)

displaying the highest a* value, meaning it has the strongest red hue, and the clove oil (Soxhlet) showing the least redness. The b* values, representing yellowness, spanned from 2.52 in the clove oil (maceration) to 5.26 in the commercial ginger oil (control).

Table 2: Colour Index of the Clove and Ginger Oils

Sample	L*	a*	b*	c*	h*
GA1	4.55 ± 0.04^{d}	5.66 ± 0.142^{b}	4.77 ± 0.08^{b}	7.41 ± 0.16^{b}	40.11 ± 0.29^{a}
ZB8	4.79 ± 0.02^{c}	5.07 ± 0.01^{c}	4.13 ± 0.02^{c}	6.54 ± 0.01^{c}	39.13 ± 0.14^{b}
CY5	7.63 ± 0.01^{b}	4.05 ± 0.02^{d}	2.52 ± 0.02^{e}	4.77 ± 0.02^{d}	31.87 ± 0.34^{d}
AD6	2.45 ± 0.01^{e}	3.57 ± 0.00^{e}	2.97 ± 0.02^{d}	4.64 ± 0.01^{d}	39.77 ± 0.18^{a}
E63	9.82 ± 0.01^{a}	7.60 ± 0.05^{a}	5.26 ± 0.05^{a}	9.24 ± 0.06^{a}	34.71 ± 0.11^{c}

$^{a-e}$ Same alphabet within the column are not significantly different ($p< 0.05$). Values are mean ± SME.
GA1= Ginger oil using Maceration, ZB8= Ginger oil using Soxhlet extraction, CY5= Clove oil using Maceration, AD6= Clove oil using Soxhlet extraction, E63= Control (commercial ginger oil).
a* negative and positive values indicate, respectively green and red.
b* negative and positive values indicate, respectively blue and yellow.
L*= Lightness on a scale from 0 (black) to 100 (white)

3.8 Total Free Fatty Acids (FFA) estimation

The assessment of oil quality commonly involves the examination of parameters like Free Fatty Acids (FFA), Acid Value, and Saponification Value. Table 3 presents the Free Fatty Acids (FFA), Acid Value, and Saponification Value for the oil samples.

3.8.1 Free Fatty Acids (FFA)

FFA in plant oils and fats are a quality feature which indicate susceptibility to oxidative aging and, ultimately rancidity (Jolayemi & Alagbe, 2022). Clove oil (Soxhlet) demonstrated the highest FFA value (10.10 g/100ml), signifying an elevated level of free fatty acids and susceptibility to oxidative deterioration and rancidity. The commercial ginger oil (control) exhibited the lowest FFA value (5.51 g/100ml), indicating better stability.

3.8.2 Acid value

The Acid Value serves as a measure of the amount of FFA present in an oil. It's an indicator of the quality and freshness of an oil, with lower values generally indicating better quality (Dudi *et al*., 2021). Clove oil (Soxhlet) displayed the highest Acid Value (20.10 g/100ml), denoting a higher concentration of free fatty acids and the least desirable quality. In contrast, the commercial ginger oil (control) registered the lowest Acid Value (10.96g/100ml), implying a lower content of free fatty acids and enhanced resistance to hydrolysis and rancidity.

3.8.3 Saponification value

The Saponification Value reflects the amount of alkali required to saponify (convert into soap) a certain amount of fat or oil. It can provide information about the average molecular weight and fatty acid composition of the oil. It indicates the number of short-chain fatty acids which are more prone to hydrolysis and oxidation (Dudi *et al*., 2021). Ginger oil (maceration) displayed the highest saponification value (196.08 mg/g), signalling a higher molecular weight of fatty acids. Conversely, clove oil (maceration) showed the lowest Saponification Value (80.15 mg/g), indicating a lower molecular weight of fatty acids, therefore a less likelihood of hydrolysis and oxidation. However, the saponification values of the oil samples are ideal.

Table 3: Total Free Fatty Acids (FFA) estimation of Ginger and Clove Oils under Two Extraction Procedures

Sample	FFA	Saponification value	Acid value
	g/100ml	mg/g	g/100ml
GA1	8.87±.14^{b}	196.08±3.58^{a}	17.65±.28^{b}
ZB8	8.30±.42^{c}	132.71±2.73^{b}	16.52±.84^{c}
CY5	7.13±.20^{d}	80.15±10.20^{d}	14.19±.40^{d}
AD6	10.10±.72^{a}	107.84±2.20^{c}	20.10±1.44^{a}
E63	5.51±.37^{e}	106.43±3.74^{c}	10.96±.74^{e}

$^{a-e}$ Mean values with different lowercase superscript letters in the same column indicate significant difference in oils of different extraction methods ($p< 0.05$).
GA1= Ginger oil using Maceration, ZB8= Ginger oil using Soxhlet extraction, CY5= Clove oil using Maceration, AD6= Clove oil using Soxhlet extraction, E63= Control (Commercial ginger oil).

3.9 Fatty acid distribution

The fatty acid profile by weight of methyl esters was analyzed in the oil samples, and comparisons were conducted among the samples with different extraction techniques. Table 4 outlines the profiles of saturated fatty acids (SFA), monounsaturated fatty acids (MUFA), and polyunsaturated fatty acids (PUFA) for the five oil samples. Analyzing these profiles provides valuable insights into the distinct characteristics of each oil and their potential applications

3.9.1 Saturated Fatty Acids (SFA) composition

The analysis revealed noteworthy variations in the saturated fatty acid composition among the five oil samples. Ginger oil (control) exhibited the highest ΣSFA at 73.58%, underscoring its richness in saturated fatty acids. This implies an undesirable

quality as high intake of SFAs has been associated with increased levels of low-density lipoprotein (LDL), or "bad" cholesterol, which can increase the risk of heart disease (Moll, 2023). In contrast, Ginger oil (Maceration), demonstrated a comparatively lower ΣSFA of 28.64%, indicating its suitability for consumption. Both Clove oil samples had lower values than the control sample, while the Ginger oil (Soxhlet) had the second lowest value, indicating a wide array of SFA composition.

3.9.2 Monounsaturated Fatty Acids (MUFA) composition

The monounsaturated fatty acids (MUFA) composition showed the superiority of clove oil (maceration) in this category, with a ΣMUFA of 22.99%. The substantial contributors were oleic acid methyl esters and erucic acid methyl esters. The higher MUFA value can help reduce bad cholesterol levels in the blood which can lower the risk of heart disease and stroke (Moll, 2023). Ginger oil (control) exhibited the lowest ΣMUFA of 8.90%, primarily due to erucic acid and methyl ester. This value is consistent with the higher ΣSFA composition of the Ginger oil (control). Ginger oil (maceration) had a higher composition than ginger oil (Soxhlet) but a lower composition than clove oil (Soxhlet).

3.9.3 Polyunsaturated Fatty Acids (PUFA) composition

The PUFA analysis revealed little difference in the PUFA compositions of the ginger oil (control) and clove oil (maceration) which had the least value. Ginger oil (maceration) exhibited the highest composition (39.70%), indicating that it has the higher probability to help reduce the levels of LDL cholesterol and improve heart health (Moll, 2023), followed by clove oil (Soxhlet) with 22.78% and ginger oil (Soxhlet) with 10.77%. The highest contributors identified were cis-5,8,11,14,17-eicosapentaenoic acid and arachidonic acid methyl esters.

Table 4: Fatty Acid Profile of Clove and Ginger Oils under Two Extraction Procedures.

Saturated Fatty Acid	**Samples**				
Compound Name	**E63**	**CY5**	**AD6**	**ZB8**	**GA1**
1. Butyric acid methyl esters	0.01	0.00	0.00	0.04	0.00
2. Caproic acid methyl esters	0.35	0.09	0.01	0.62	0.01
3. Caprylic acid, methyl ester	0.00	0.00	0.01	0.01	0.00
4. Capric acid, methyl ester	0.00	0.00	0.01	0.01	0.00
5. Undecanoic acid, methyl ester	4.33	0.96	0.00	3.28	0.61
6. Lauric acid, methyl ester	0.11	0.00	0.00	1.59	0.01
7. Tridecanoic acid, methyl ester	10.90	32.65	21.32	7.59	6.69
8. Myristic acid methyl esters	1.45	0.00	5.16	19.66	0.67
9. Pentadecanoic acid, methyl ester	0.32	0.04	0.05	0.00	0.01
10. Palmitic acid, methyl ester	7.70	0.17	2.14	0.78	0.69
11. Heptadecanoic acid, methyl ester	0.41	0.97	0.38	0.81	1.30
12. Arachidic acid, methyl ester	0.00	0.00	0.00	0.00	0.00
13. Heneicosanoic acid, methyl ester	0.00	0.00	0.00	0.00	0.00
14. Behenic acid, methyl ester	0.00	0.00	0.00	0.00	0.00
15. Tricosanoic acid, methyl ester	36.17	27.09	6.08	18.64	15.85
16. Lignoceric acid, methyl ester	11.83	2.01	4.24	3.46	2.80
ΣSFA	73.58	63.98	39.40	56.49	28.64
Monounsaturated Fatty Acid					
1. Oleic acid methyl esters	6.27	11.35	7.80	7.08	2.66
2. cis-10-Heptadecenoic acid, methyl ester	0.00	0.00	0.00	0.00	0.00
3. cis-11-Eicosenoic acid, methyl ester	0.00	0.00	0.00	0.00	0.00
4. Erucic acid, methyl ester	2.63	11.64	11.12	4.19	15.11
5. Nervonic acid, methyl ester	0.00	0.00	0.00	0.00	0.00
ΣMUFA	8.90	22.99	18.92	11.27	17.77
Polyunsaturated Fatty Acid					
6. Linoleic acid methyl esters	0.17	0.13	0.10	0.23	0.12
7. Arachidonic acid methyl esters	3.49	4.32	0.00	3.19	17.48
8. Cis-5,8,11,14,17-Eicosapentaenoic acid,	3.50	1.92	20.45	4.17	17.48
9. Methyl cis-8,11,14-Eicosatrienoic acid,	1.33	1.81	2.23	3.18	4.62
10. cis-4,7,10,13,16,19-Docosahexaenoic acid	0.00	0.00	0.00	0.00	0.00
ΣPUFA	8.49	8.18	22.78	10.77	39.70

GA1= Ginger oil using Maceration, ZB8= Ginger oil using Soxhlet extraction, CY5= Clove oil using Maceration, AD6= Clove oil using Soxhlet extraction, E63= Control (Commercial ginger oil).

4. Conclusion

To encapsulate, the study's findings underscored the pivotal role of extraction methods in shaping the physicochemical attributes and quality markers of ginger and clove oils. The extraction techniques led to substantial disparities in the oils' chemical and physical traits. The pH (4.52 - 6.08) and specific gravity (0.85 - 0.97) were well within the accepted range. The oils' hues deviated from those documented in prior studies, likely due to the elevated temperature exposure during extraction. The impurity content varied from acceptable (7.07%) to unacceptable (21.59%) for edible oils. The moisture contents (1.37 - 8.88%) indicate low susceptibility to microbial growth. The commercial ginger oil (control) exhibited the lowest FFA (5.51 g/100ml) and acid value (10.96 g/100ml), indicating better stability and resistance to rancidity while the clove oil (Soxhlet method) had the highest FFA (10.10 g/100ml) and acid value (20.10 g/100ml), therefore it has the least desirable quality. The oils' TBA values (0.0024 - 0.0025 mgMDA/g) indicate enhanced stability and extended shelf life while their saponification value (80.15 - 196.08 mg/g), indicate varying but ideal FFA molecular weights. The oil samples showed diverse FFA profile: the higher ΣSFA composition (73.58%) of the commercial ginger oil (control) implies an undesirable quality as it has a higher risk of heart disease when consumed in large quantity while the ginger and clove samples extracted using maceration exhibited desirable ΣMUFA (22.99%) and ΣPUFA (22.78%) compositions. These affirm the influence of the extraction methods on the quality indices of the oils. It is therefore deduced from the analyses conducted that the maceration extraction is the better solvent extraction method.

Recommendations

In light of this study's conclusions, the examined factors could be instrumental in precisely predicting the physicochemical properties of clove and ginger oils, thereby facilitating large-scale production and process optimization. It is also advisable to conduct additional research to evaluate a broader range of physical, chemical, and quality indices for process optimization. This is particularly pertinent given the suboptimal qualities of the clove oil extracted using the Soxhlet method. Ultimately, these efforts could lead to the better identification of the most effective solvent extraction technique.

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Conflict of interest

The authors declare no conflicts of interest.

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