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Citrus Reticulata Pericarp and Pip: Physiocochemical Properties and Chemical Characterization of Methanolic Crude Oil Extracts A Comparison

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Abstract

Citrus reticulata also known as Tangerine, possess high nutraceutical value due to high content of antioxidants and vitamin C. This researched is aimed to evaluated the physicochemical properties, phytochemical, and chemical composition of the pericarp and pips methanolic oil extracts of Citrus reticulata. The pips and pericarps were isolated from mature tangerine, air dried at room temperature and extracted using methanol solvent by soxhlet extractor. The crude oil extract was subjected to physicochemical analysis, phytochemical screening, gas chromatography mass spectrometry (GC-MS) for determination of the active phytoconstituents, Fourier Transform Infrared spectroscopy (FTIR) to determine the functional group present. All analysis were of standard procedure. The pericarp showed higher yield methanolic oil extract of (20.5%) compared to pip (2.6%). Physicochemical properties of the oil extracted were saponification value, 247.60 mg KOH/g of oil; ester value, 232.66 mg/KOH/g; Iodine value 15.47g I_2/100 g oil; and peroxide value 61.65 mEq O2 / kg of oil from the pericarp compared to the pips oil 79.87 mg KOH/g, 60.64 mg KOH/g, 13.59 g I_2/100 g; and 20.75 mEq O2 / kg respectively. Qualitative analysis reavealed the presence of flavonoids, alkaloids, phenols, cardiac glycosides , terpenoids, Anthraquinones, steriods, in both oils but saponin was present in the pericarp only. The results of the GC-MS analysis showed Hexadecanoic acid, 1,2-Cyclohexanediol, Limonene, 9,12-Octadecadienoic acid present in both methanolic crude oil extracts. FTIR analyses showed functional groups such as alcohol, ether, alkyl halids in both the methanolic oil extracts. The oil of Citrus reticulata pericarp and pips contains significant amount of active compounds, which could be useful for industrial purpose, for preservative and functional food, thereby offering a sustainable strategy for citrus waste utilization.

Keywords: *Citrus reticulata,* phytoconstituent, GC-MS, Physicochemical properties, pericarp.

Introduction

Citrus reticulata, commonly known as tangerine, is among the most widely consumed citrus fruits worldwide (Shorbagi et al., 2022). It has a tiny peel with a little bitter taste white mesocarp. The name tangerine originated from Tanger Morocco, where the fruit was first shipped to Europe and the United States. It is one of the citrus fruits that is easy to peel and has a sweet flavor (Barnossi et al., 2020). The edible part is rich in vitamin C, which helps to improve the immune system (Chakraborty et al., 2025). The pericarp and pips, though often discarded, are rich in active compounds including flavonoids, phenolics, and essential oils, which have been linked to various biological activities such as antioxidant, antimicrobial, anti-inflammatory, and anticancer effects (Maqbool et al., 2023).

The tangerine leaves have been used in folk medicine as herbal medicine mainly across Asian cultures in treating sore throats, and reducing inflammation, and is used as a flavoring agent in cooking (Shi et al., 2024). The stem bark of tangerine also contains bioactive compounds such as flavonoids, tannins, and alkaloids, contributing to its antimicrobial and antioxidative properties (Salih et al., 2016). In the food industry, extracted oil from tangerine pips is used as a preservative, due to their antioxidative and antimicrobial properties, extending the shelf life of fresh and processed foods by preventing oxidative deterioration and microbial spoilage (Petcu et al., 2023).

The antioxidant potential of plant extracts reveals their therapeutic potential, which can be linked to oxidative stress. Despite their health benefits, high antioxidant levels can be harmful to human health, thereby causing the restriction of nutrients and increasing the permeability of the intestinal wall. oil extracts from the pips and pericarp of the tangerine fruits help to address the environmental waste management issue as a result of waste from discarded tangerine seeds and pericarp

(Barnossi et al., 2020). Researcher, have reported that oil from both the pericarp and pip of the tangerine fruit addresses global food waste and safety issues (Sabry et al., 2024). Therefore, this study analyzed the physiocochemicals properties, and chemical composition *Citrus reticulata* pericarp and seeds (pips) grown in Nigeria as potential for industrial applications and managing infections and inflammation.

Materials and Methods

Collection of Plant Samples

Ripe fresh tangerines were collected locally in Ogbomoso, South West, Nigeria (8.1227° N, 4.2436° E) in December. The tangerine fruits were identified at the herbarium, Department of Biology, Ladoke Akintola University of Technology, with a voucher number: LHO 879. The tangerine fruits were washed, the pericarp and pips were separated, and air-dried at room temperature for about 7 days. The dried tangerine peel and seeds were then pulverized into coarse powder using an industrial blender.

Extraction

The oil extraction was performed according to AOAC (2000) standard protocols using a Soxhlet apparatus with methanol (BDH, London) solvent. About 200 grams of the powdered tangerine peel and seeds were weighed and placed in a filter paper thimble and then inserted into the main chamber of the Soxhlet extractor, respectively. The methanol extract was concentrated using the rotary evaporator under reduced pressure (200pa) and temperature (40°C) to remove the solvent to obtain a crude syrup extract.

Physicochemical Analysis of *Citrus reticulata* pericarp and pip methanolic crude oil extracts

Extracted oil yield

The percentage yield of the extracted oil was calculated using the formula by AOAC (2000).

$$\text{Percentage oil Yield} = \frac{weight\ of\ oil\ extracted}{weight\ of\ the\ sample} \text{ x } 100\%$$

Sensory Analysis of the Oil

Sensory analysis was carried out according to AOAC (2000) including smell, sense of sight, and touch.

Determination of the solubility of the oil in water

The solubility of the oil extract was determined by according to AOAC (2000). The inability of the oil extract to dissolve in water, forming a separate layer, confirms its insolubility in water.

Determination of Density

The density of the extract was calculated using the following equation;

$$\text{Density} = \frac{weight\ of\ oil\ sample}{volume\ of\ oil}$$

Determination of Specific Gravity

The specific gravity was determined by calculating the ratio of the weight of the extract to that of the water (Abdulrasheed et al., 2015).

$$\text{Specific gravity} = \frac{weight\ of\ volume\ of\ oil\ extracted}{weight\ of\ volume\ of\ water}$$

Determination of Saponification Value

A volume of 0.5 mL of the extract was measured into a 200 mL conical flask unto to which 12.5 mL of 0.5 N of KOH was added. The resulting mixture was refluxed for 30 minutes, followed by addition of 2 drops of phenolphthalein indicator, and was titrated against 0.5 N HCl until coloration disappeared. This procedure was repeated without the extract and the titre value was determined from the blank value (Abdulrasheed et al., 2015). The saponification value was calculated using Equation below;

$$\textbf{Saponification value (SV)} = \frac{(B-S)xNx56.1}{weight\ of\ the\ sample\ (g)}$$

Where:
B = Volume of acid used for the blank (mL)
S = Volume of acid used for the sample (mL)
N = Normality of the acid
56.1 = Molar mass of KOH in g/mol.

Determination of Acid Value

A volume of 0.5 mL of the extract was measured into a conical flask containing 12.5 mL of isopropyl alcohol. 2 drops of phenolphthalein indicator were added to the mixture. The resulting mixture was titrated against 0.1N KOH solution until the endpoint is reached (persistent pink color) (Abdulrasheed et al., 2015). The acid value was calculated using the Equation below;

$$\textbf{Acid value (AV)} = \frac{VxNx56.1}{weight\ of\ the\ sample\ (g)}$$

Where:
V = Volume of alkali used (mL)
N = Normality of the alkali
56.1 = Molar mass of KOH in mg/mol.

Determination of Ester Value

Ester value, which is defined as the number of milligrams of potassium hydroxide required to saponify the fatty acid esters in 1 g of the extract, was also determined for the oil extract. It was obtained as the difference between the saponification value and the acid value of the extract (Abdulrasheed et al., 2015). The saponification value was calculated using the equation.
Ester value = Saponification value − Acid value

Determination of pH Value

The pH value of the extract was determined using an electronic pH meter. The electrode was inserted into the extract, and the reading was recorded. This process was repeated three times, and the mean pH value was calculated (Abdulrasheed et al., 2015).

Determination of Free Fatty Acid

A volume of 0.5 mL of oil extract was measured in a beaker and warmed; 12.5 mL of methanol was added to the sample and stirred thoroughly followed by 2 drops of phenolphthalein indicator and a drop of 0.14 N NaOH solution. The mixture was titrated against NaOH solution until a light pink colour which persisted for about 1 minute was observed. The end-

point was recorded and used to calculate the free fatty acid from the equation below (Abdulrasheed et al., 2015);

$$\text{Free fatty acid} = \frac{titre\ value\ xNx56.1}{weight\ of\ the\ sample\ (g)}$$

N is the normality of the base.

Determination of the iodine value of the oil

Iodine value was determined using Wij,s method according to (A.O.C.S, 2017). A 8.0 g of iodine monochloride was dissolved in 200 cm3 glacial acetic acid. 9.0 g of Iodine crystals was dissolved in 300 cm³ of carbon tetrachloride. The two solutions were mixed and made up to the mark with glacial acetic acid. About 10 g of methanolic oil sample was weighed into a dry 250 cm³ conical flask, and 10 cm³ of carbon tetrachloride was added, followed by 20 cm³ of the prepared Wij's solution. The flask was kept in the dark cupboard for 30 minutes at room temperature; 15 mL of 10 % potassium iodide solution and 100 cm3 of distilled water were added. This was titrated against 0.1 mL Sodium thiosulfate solution using starch as an indicator. A blank titration was also conducted under the same conditions without the sample.

$$\text{Iodine value} = \frac{(A-B)xNx1.26}{weight\ of\ the\ sample\ (g)}$$

Where;
A = volume of 0.1 mL $Na_2S_2O_3.5H_2O$ solution used for the black titration, B = Volume of 0.1 mL of $Na_2S_2O_3.5H_2O$ solution used for the Sample titration, Q = Weight in grams of the oil sample 1.269 = Conversion factor, N = Normality.

Determination of the peroxide value of the oil

The peroxide value was determined according to (Saidu & Garba, 2011). In a conical flask, 30 mL of acetic acid was measured, and 2 mL of the oil sample was added to it. Then, 0.5 mL saturated solution of potassium iodide was also added, followed closely by the addition of 30 mL of distilled water. The flask content was titrated against 0.1 M sodium thiosulfate until the resulting colour disappeared. Thereafter, 0.5 mL starch indicator was added, and the titration continued till an endpoint was observed. A blank titration experiment was also performed. The peroxide value was calculated using the equation.

$$\text{Peroxide value} = \frac{(B-S)xNx56.1}{weight\ of\ the\ sample\ (g)}$$

S is the blank titre value, B is the sample titre value

Phytochemical screening of methanol extract of *Citrus reticulata*

The phytochemicals screening of alkaloids, tannins, anthraquinones, terpenoids, flavonoids, saponins, glycosides, phenol, steroids, and coumarins, were performed using standard procedures described by (Ashraf et al., 2024; Sofowora et al., 1993; Akubugwo & Ugbogu, 2007).

Alkaloids

The oil extracts were screened for alkaloids. 0.1 g of the oil was diluted to 10 mL with acid alcohol, boiled and filtered. About 5 mL of the filtrate was added to 2 mL of dilute ammonia and 5 mL of chloroform was added and shaken gently to extract the alkaloid. The chloroform layer was extracted with 10 mL of acetic acid. Mayer's reagent was added. The formation of a cream colouration, was regarded as positive for the presence of alkaloids.

Flavonoids

The presence of flavonoids in the oil extracts was determined by adding 1 % ammonia solution to the oil extracts in a test tube. A yellow coloration, which disappears when sulphuric acid is added to it, confirms the presence of a flavonoid.

Tannin

Tannin was screened for by adding 0.1 g of oil to 10 mL of distilled water in a test tube and filtered. About 0.1 % ferric chloride was added to the filtered samples and observed. The presence of brownish green or a blue black colouration confirms the presence of tannins.

Saponin

To determine the presence of saponin, 0.1 g of the oil extract was boiled together with 20 mL of distilled water in a water bath and filtered. About 10 mL of the filtered oil extract was mixed with 5 mL of distilled water in a test tube and shaken vigorously to obtain a stable, persistent froth. The frothing was then mixed with 3 drops of olive oil and observed for the formation of an emulsion, which confirms the presence of saponins.

Terpenoid

A weight of 0.1 g of the oil extract was mixed with 2.0 mL of chloroform in a test tube. 3.0 mL of concentrated sulphuric acid was carefully added to the mixture to form a layer. An interface with a reddish brown coloration was formed, confirming the presence of terpenoids.

Steroids

In a test tube, 0.1 g of the oil extract was dissolved in chloroform, shaken and filtered into another test tube. A few drops of acetic anhydride were added, followed by the addition of concentrated sulphuric acid and the mixture was observed for a green colour, confirming the presence of steroids.

Anthraquinones

To determine the presence of anthraquinones, 0.1 g of the oil extract was boiled with 10.0 mL of sulphuric acid and filtered while hot. The filtrate was shaken with 5.0 mL of chloroform. The chloroform layer was pipetted into another test tube and 1.0 mL of dilute ammonia was added. The resulting solution was observed for colour changes.

Cardiac Glycosides

To 0.1g of oil extract was diluted with 5.0 mL of water, then 2.0 mL of glacial acetic acid containing one drop of ferric chloride solution was added. This was underlaid with 1.0 mL of concentrated sulphuric acid. A brown ring at the interface confirmed the presence of a deoxy sugar characteristic of cardenolides. A violet ring may appear below the brown ring, while in the acetic acid layer, a greenish ring forms just above

the brown ring and gradually spreads throughout this layer.

Coumarins

A weight of 0.1 g of the oil extract was put in a dry test tube, and 2 mL of 10 % sodium hydroxide solution was added. The solution was mixed thoroughly and observed for color change. Formation of a yellow color confirmed the presence of coumarins.

Phenols

To screen for phenols, 0.1 g of the oil extract was dissolved in a test tube with ethanol, shaken and filtered into another test tube. A few drops of ferric chloride were added and observed for a blue-black colouration, confirming the presence of phenol.

Quantitative analysis of Phytochemical Constituents of *Citrus Reticulata*

The analysis was conducted according to the method described by Krishnaiah et al. (2009) on both the pericarp (peel) and pips (seed) methanol oil extracts of *Citrus reticulata*.

Alkaloids Determination

The presence of alkaloid was determined by weighing 0.2 g of the oil extract, which was put in a 250 mL beaker and 200 mL of 1.0 % acetic acid in ethanol was added. The mixture was covered and allowed to stand for 4 hours. It was then filtered and the filtrate was concentrated in a water bath until it reached a quarter of its original volume. Concentrated ammonium hydroxide was added until precipitation was complete. The mixture was allowed to settle and the precipitate was collected on a weighed filter paper and washed with dilute concentrated ammonium hydroxide. The precipitate, alkaloid, was dried and weighed. The percentage alkaloid was then calculated by difference.

$$\% \text{ Alkaloids} = \frac{Final\ weight\ of\ sample}{Total\ weight\ of\ sample} x100$$

Determination of Flavonoids

Flavonoid content was measured by the aluminium chloride colorimetric assay. The reaction mixture, consisting of 1 mL of extract and 4ml of distilled water, was taken in a 10 mL volumetric flask. To the flask, 0.30 mL of 5 % sodium nitrite was added and after 5 minutes, 0.3 mL of 10 % aluminium chloride was mixed. After 5 minutes, 2 mL of 1 M Sodium hydroxide was treated and diluted to 10 mL with distilled water. A set of reference standard solutions of quercetin was prepared in the same manner as described earlier. The absorbance for test and standard solutions was determined against their agent blank at 510 nm with a UV/Visible spectrophotometer. The total flavonoid content was expressed as mg of QE/g of extract.

Determination of Saponins

A weight of 0.2 g of the oil was weighed into a 250 mL conical flask. 100 mL of 20 % ethanol was added. The mixture was then heated over a hot water bath for 4 hours with continuous stirring at about 55°C and filtered with a filter paper. The residue was re-extracted with another 200 mL of 20 % ethanol. The combined extract was then reduced to 40 mL over a water bath at about 90°C. The concentrated extract was transferred into a 250 mL separator funnel and 20 mL of ethyl ether was added to the extract and shaken vigorously. The aqueous layer was recovered while the ethyl ether layer was discarded. This purification process was repeated. 60 mL of n-butanol was added and the combined n-butanol extract was washed twice with 10 mL of 5 % Sodium chloride solution. The remaining solution was then heated on a water bath in a pre-weighed 250 mL beaker. After evaporation, the residue was dried in an oven to a constant weight.

$$\% \text{ saponin} = \frac{Final\ weight\ of\ sample}{Total\ weight\ of\ sample} \text{ x } 100$$

Determination of Tannins

The tannins were determined by the Folin-Ciocalteu method (Mahmoud et al., 2021). About 0.1 mL of the sample extract was added to a volumetric flask (10 mL) containing 7.5 mL of distilled water and 0.5 mL of Folin-Ciocalteu phenol reagent, 1 mL of 35 % sodium carbonate solution and diluted to 10 mL with distilled water. The mixture was shaken well and kept at room temperature for 30 minutes. A set of reference standard solutions of gallic acid was prepared in the same manner as described earlier. Absorbance for test and standard solutions was measured against the blank at 725 nm with a UV spectrophotometer. The tannin content was expressed in terms of mg of GAE/g of extract.

Determination of Phenols

Phenolic compound contents were determined by the Folin-Ciocaltea method (Zhang et al., 2018). The extract samples (0.5 mL of different dilutions) were mixed with Folin Ciocalteu reagent (5 mL, 1:10 diluted with distilled water) for 5 min and aqueous sodium carbonate (4 mL, 1 M) was then added. The mixture was allowed to stand for 15 min and the phenols were determined by the colorimetric method at 765 nm. The standard curve was prepared and solutions of Gallic acid in methanol: water (50:50 v/v). Total phenol values are expressed in terms of Gallic acid equivalent (mg/g of dry mass), which is a common reference compound

Gas Chromatography-Mass Spectrometry Analysis

The volatile compounds in the *Citrus reticulata* pericarp and pip methanolic oil extracts were identified and quantified using Gas Chromatography–Mass Spectrometry (GC-MS). The volatile extracts were subjected to chromatographic analysis using a Varian 3800/4000 gas chromatograph mass spectrometer equipped with an Agilent equipped with a splitter split/splitless. With a BP5 (30 m × 0.25 mm × 0.25 microns) capillary column.. Nitrogen was used as a gas carrier. 1.0 µL volumes were injected using a splitless mode at an injector temperature of 2700C. The oven temperature was programmed at 50˚C for 1 min, then a heating rate of 10˚C/min up to 250˚C, and then left at 250˚C for 10 minutes. The operating temperature range had an upper limit of 280˚C. The mass spectrometer conditions were as follows: ionization voltage was above 50 eV; ion source temperature was more than 120˚C; electron ionization mass spectra were acquired over the mass range of m/z = 40 – 800. The ion source temperature was set to 250 °C. The blank was first injected, and was followed by the volatile compound injection.

Organic compounds in the volatile compounds were identified in Wiley's NIST 08 Mass Spectral Library. Contents of individual compounds in the extract were given in percent of the total compound in the oil (Mahmoud et al., 2021).

Fourier Transform Infrared Spectroscopy Analysis

Fourier-Transform Infrared (FTIR) Spectroscopy was employed to determine the characteristic functional groups of the methanolic oil extracts. The analysis was conducted using an FTIR spectrometer. The dried powder of *C. reticulata* pericarp and pip were analyzed directly in the solid state utilizing the Attenuated Total Reflectance (ATR) technique. FT-IR spectra were recorded in the 400–4000 cm^{-1} absorption range (Khan et al., 2018).

Statistical Analysis

The results of this research work were presented as mean ± standard deviation (S.D) of triplicate values. The data generated were analyzed using a two-way analysis of variance (ANOVA), followed by Tukey's Post Hoc multiple comparison test to identify variations within the pericarp and pips methanolic oil extract from *citrus reticulata*. P-value < 0.05 was regarded as statistically significant and denoted by an asterisk.

Results and Discussion

Physicochemical analysis of the methanolic oil extracts of *Citrus reticulata* pericarp and pip

The physicochemical properties of methanolic oil extracts from *Citrus reticulata* pericarp and pips are shown in Table 1. The pericarp and pip of citrus reticulata methanolic oil extracts yields 20.5 % and 2.6 % respectively. The pericarp gave higher yeid as a result of low moisture. The result different from (Wulandari et al., 2024), which ethanolic oil citrus reticulata peel extract yeilded 19.50 %. The difference might be due to solvent used and geographical distribution of the plant. From the sensory analysis tests, both oils had dark brown coloration, pleasant citrus-like smell, light texture, and were insoluble in water. The density value of the pip oil (1.19 g/mL) is slightly denser than the pericarp oil (1.13g/mL). The specific gravity of both the pericarp oil (1.12) and the pip (1.18) is denser than water. The study of Spezzini et al. (2024) reported that the difference in the density may be due to the different fatty acid compositions in the *citrus reticulata* methanolic oil extracts.

The chemical properties of the methanolic crude oil are used to determine the present condition and quality of the oil. Peroxide value and free fatty acid are used to monitor the quality of edible oil for further refining process of crude oil. The degree of edibility of a fat is considered to be inversely proportional to the total amount of free fatty acid. The acid value and free fatty acid content of pips (19.23mg KOH/g and 48.09 mg KOH/g) is higher than pericarp oil (14.94 mg KOH/g and 11.42mg KOH/g), respectively. The lower acidity oils showed that they are edible and could have a long shelf life.This finding were comparable to (Uçar et al., 2024). Low acid and free fatty acid values indicated that the methanolic oil from the pericarp may be stable for a long period of time and protect against rancidity and peroxidation than the pip oil, this finding is supported by (Uçar et al., 2024). The saponification value for cirrus reticulata pericarp and pip oils were comparable to the value reported by (Adeleke et al., 2017). The pericarp showed higher value than the pips which contains shorter chain fatty acids (Adeleke et al., 2017). The ester value indicated that the pericarp oil (232.66 mg KOH/g) contains esterified fatty acids, contributing to the oil stability compared to the pip oil (60.60 mg KOH/g). The peroxide value, an indicator of primary oxidation products, was much higher in pericarp oil (616.45 meq/kg) than in pip oil (207.85 meq/kg). This implied that despite the oil's richness in unsaturated fatty acids, it is more prone to oxidation, but it still remains valuable as a natural preservative when properly managed, as reported by (Alsufiani & Ashour, 2021). The result showed that the pericarp has (15.47 g I_2/100 g) iodine value compared to the pip oil extract with (13.59 g I_2/100 g). These relatively low iodine values for both methanolic oil extracts depicted the presence of saturated lipids, including alkanes and saturated fatty acid esters from the GC-MS analysis (Table 3 and 4). The peroxide value of the pericarp (61.65 mEq/kg) was higher than the pip oil peroxide value (20.25 mEq/kg) The pip methanolic oil is more stable, but having low yield than the pericarp oil extract (Roiaini et al., 2015). reported a much lower value of 29.91±0.027 mEq/kg for blend of olive, palm olein and canola oil. Low peroxide values confirm the stability of an oil sample while higher values between 20 and 40 mEq/kg result to a rancid taste (Tsado et al., 2018).

Table 1: Physicochemical properties of methanolic crude oil extracts of *citrus reticulata*

Parameters	Pericarp	Pip
Oil yields %	20.5	2.6
Specific gravity	1.12	1.18
Acid value (mg KOH/g)	14.94	19.23
Saponification value (mg KOH/g)	247.60	79.87
Iodine value (g I_2/100 g)	15.47	13.59
Peroxide value (mEq O_2/kg)	61.65	20.75
pH	3.88	4.28
Density (g/mL)	1.13	1.19
Fatty acid (mg KOH/g)	11.42	48.09
Ether value (mg KOH/g)	232.66	60.64

Phytochemical Constituents

The qualitative phytochemical analysis of both the *Citrus reticulata* pericarp and pips oil extract revealed the presence of phytochemicals like phenol, alkaloid, steroids, tannins, flavonoids, cardiac glycosides, anthraquinones, and terpenoids, as shown in Table 2. Saponins was absent in the pips oil extract in contrast with the report of (Wulandari et al., 2024) which confirm the presence of saponin. The differences in the results obtained from the phytochemical screening of Citrus reticulata by different researcher might be due to the variations in methods, geographic distribution, the extraction process, handling and storage, plant part, time of collection of the plant material, climatic condition and the solvents used for the extraction. All these can influence the concentrations of phytochemicals (Oladipo et al., 2023).

Table 2: Qualitative phytochemical analysis of the methanolic pericarp and seed extract of *Citrus reticulata*

Phytochemicals	Pericarp	Pip
Saponins	+	-
Tannins	++	+
Flavonoids	++	+
Cardiac glycosides	++	++
Anthraquinones	++	++
Terpenoids	++	++
Steroids	++	++
Phenols	++	++
Alkaloids	++	+

key: + = present; ++ = abundant; - = absent

Quantitative Phytochemical composition

The quantitative analysis of the *Citrus reticulata* pericarp and pip extract also revealed the concentration of phytochemicals expressed as percentage yield (% w/w). Figure 1 showed the level of comparison between the pericarp and the pip oil. There is a significant increase ($p<0.05$) in the methanolic *Citrus reticulata* oil extract from the pericarp than pip oil extract. Alkaloids are commonly used as medicinal agents for their bactericidal, antipasmodic and anti-inflammation effects. The alkaloids contents of pericarp is higher (6.54 ± 0.15 %) than pip (3.45 ± 0.12 %) and is in close range with 3.09 % reported for soursop seed flour (Onyechi et al 2014). The terpinoids contents is higher for pericarp oil (1.85 ± 0.15 %) compared to pips (1.49 ± 0.15 %) which is lower than terpenoid content 3.433 ± 0.208 obtained in Carica Papaya (Papaw) Seed (Adeleke et al., 2023). This is consistent with the research conducted by Santoso et al. (2020), which also showed the presence of these compounds after the qualitative phytochemical screening of 96% ethanol extract of Citrus reticulata peel. The finding aligns with Musara et al. (2020) that the pericarp of *C. reticulata* is a potent source of different secondary metabolites responsible for many bioactivities, including antioxidant, anti-inflammatory, and antimicrobial properties, contributing to their medicinal and nutritional value.

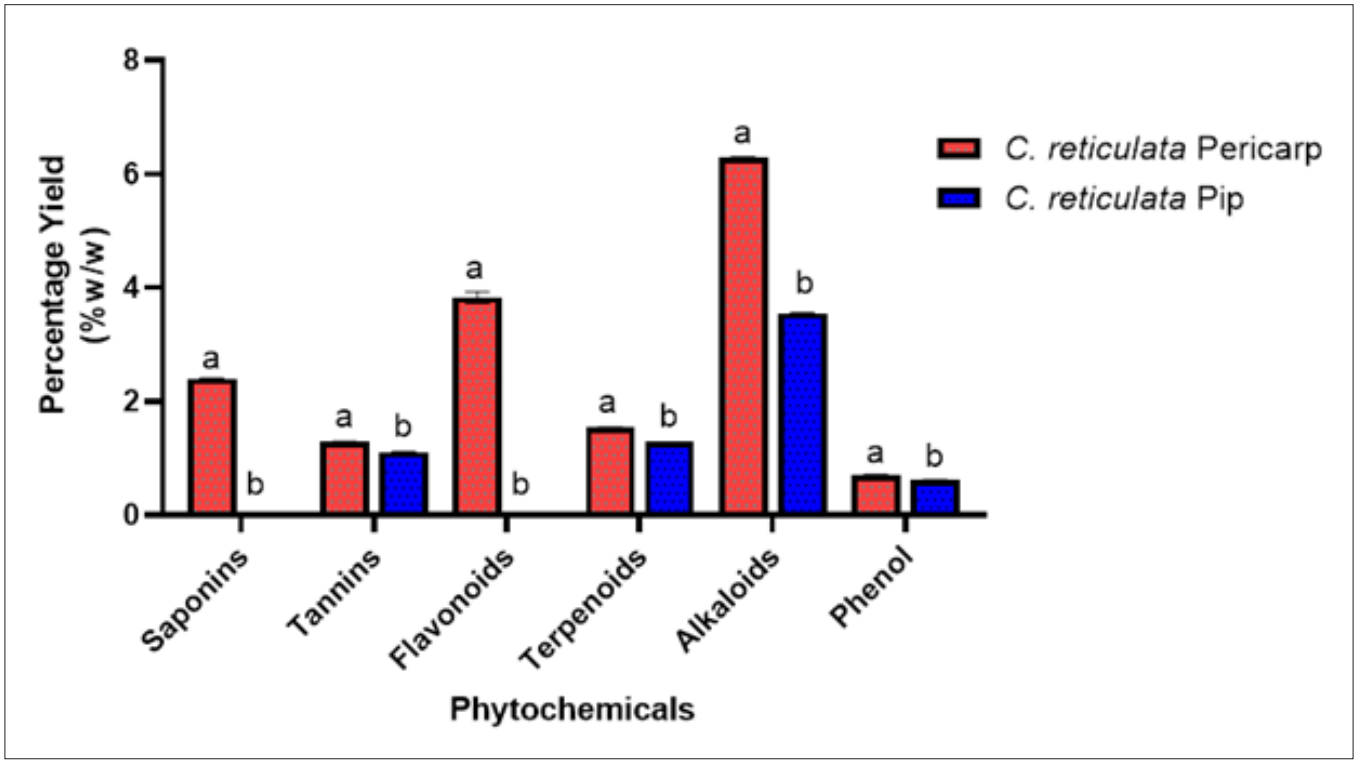

Figure 1: Quantitative analysis results of methanolic oil extract from Citrus reticulata pericarp and pip

Gas Chromatography-Mass Spectrophotometry Analysis

Table 3 and 4 shows the fatty acid composition of crude methanolic oil extract of pericarp and pips. The pericarp oil sample had high amount of D-limonene (15.44 %) as the major constituent, with 3,7-dimethyl-1,6-octadien-3-ol (12.89 %), 1,2-cyclohexanediol derivatives (12.36 %), monoterpenes, and 2-methoxy-4-vinylphenol (6.90 %) while pip oil extract contain higher amount 6-octen-1-ol, 3,7-dimethyl-, propanoate (23.20 %), pentacosane (13.74 %), 6-epi-shyobunol (11.14 %) and lower amount monoterpenes and long-chain esters, alkanes, and oxygenated sesquiterpenes. The methanolic crude oil extract also contain fatty-acid methyl and ethyl esters such as hexadecanoic acid methyl ester and linoleic acid esters which are commonly reported in citrus pericarp oils and are known for their antioxidant, anti-inflammatory, and antimicrobial properties. The presence of these compounds indicated that the pericarp methanolic oil is rich in volatile bioactive components that may account for the strong therapeutic effects often associated with citrus pericarp (Lin et al., 2023). These volatile compounds are linked to different phytochemical constituents present in the oil extracts and serve as potential use in medicine and nutraceutical or cosmetic formulation (Luo et al., 2017).

Table 3: Chemical Composition of *Citrus reticulata* Pericarp Methanolic Oil Extract.

Peak #	Compound	Retention Time	Area (%)
1	D-Limonene	10.730	15.44
2	.gamma.-Terpinene	11.127	0.99
3	1,6-Octadien-3-ol, 3,7-dimethyl-	11.65	12.89
4	Octanoic acid, ethyl ester	12.846	1.10
5	Terpinen-4-ol	13.130	0.65
6	Decanal	13.204	2.85
7	alpha.-Terpineol	13.341	3.06
8	Benzene, 1-methoxy-4-methyl-2-(1-methylethy	13.564	0.79
9	2,4-Cycloheptadien-1-one, 2,6,6-trimethyl-	13.947	0.62
10	Phenol, 2-methyl-5-(1-methylethyl)	14.485	3.60
11	1-Cyclohexene-1-carboxaldehyde, 4-(1-methyl	14.737	0.68
12	2-Methoxy-4-vinylphenol	14.995	6.90
13	1,2-Cyclohexanediol, 1-methyl-4-(1-methyleth)	15.235	1.55
14	1,2-Cyclohexanediol, 1-methyl-4-(1-methyleth	15.394	11.98
15	(3S,4R,5R,6R)-4,5-Bis(hydroxymethyl)r	15.752	3.81
16	1,5-Cyclodecadiene	16.206	2.59
17	1-Cyclooctene, 3,6-epoxy-7-acetyloxy-	16.690	0.90
18	alpha.-Farnesene	16.752	1.32
19	3-Furanacetic acid, 4-hexyl-2,5-dihydro-2	17.102	1.22
20	2-Oxabicyclo[2.2.2]octan-6-ol,	17.526	0.76
21	Isoaromadendrene epoxide	18.808	1.93
22	1,2,3,5-Cyclohexanetetrol	19.038	6.43
23	2,6,9,11-Dodecatetraenal	19.847	5.36
24	1,6-Anhydro-.alpha.-d-galactofuranose	20.725	5.70
25	Hexadecanoic acid, methyl ester	21.219	1.46
26	Hexadecanoic acid, ethyl ester	21.919	0.99
27	3-Heptanol, 2-methyl	22.310	3.81
28	9,12-Octadecadienoic acid, methyl ester	23.291	1.07
29	Linoleic acid ethyl ester	24.076	1.23

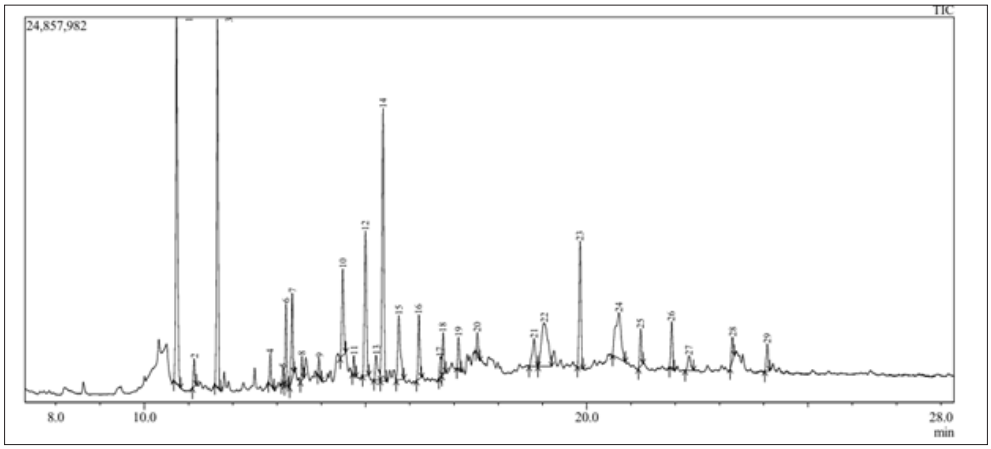

Figure 2: The chromatographic profiles of the volatile compounds in the methanolic oil extract of *Citrus reticulata* pericarp

Table 4: Chemical Composition of Citrus reticulata Pip Methanolic Oil Extract

Peak #	Compound	Retention Time	Area (%)
1	D-Limonene	10.739	2.09
2	Pentacosane	17.832	13.74
3	6-Octen-1-ol, 3,7-dimethyl-, propanoate	18.329	23.20
4	6-Octen-1-ol, 3,7-dimethyl-, propanoate	18.803	3.97
5	Ethanol, 2-(3,3-dimethylcyclohexylidene)	19.068	7.10
6	Succinic acid	19.695	1.17
7	Adipic acid, di(.beta.-citronellyl) ester	19.880	1.81
8	Trifluoroacetyl-lavandulol	20.836	3.40
9	Hexadecanoic acid, methyl ester	21.226	4.45
10	Oxirane, 2, 2-dimethyl-3-(3,7,12,16,20-pentamethylhenicosa	21.346	5.86
11	Oxirane, 2, 2-dimethyl-3-(3,7,12,16,20-pentamethylhenicosa	21.739	1.55
12	Hexadecanoic acid, ethyl ester	21.928	1.78
13	Oxirane, hexadecyl	22.358	6.12
14	9,12-Octadecadienoic acid, methyl ester	23.300	1.31
15	9,12-Octadecadienoic acid, ethyl ester	24.092	4.45
16	Octadecanoic acid, ethyl ester	24.352	1.14
17	Pentatriacontane	25.034	4.18
18	6-epi-shyobunol	27.606	11.14
19	Tricyclo[20.8.0.0(7,16)] triacontane	27.994	1.54

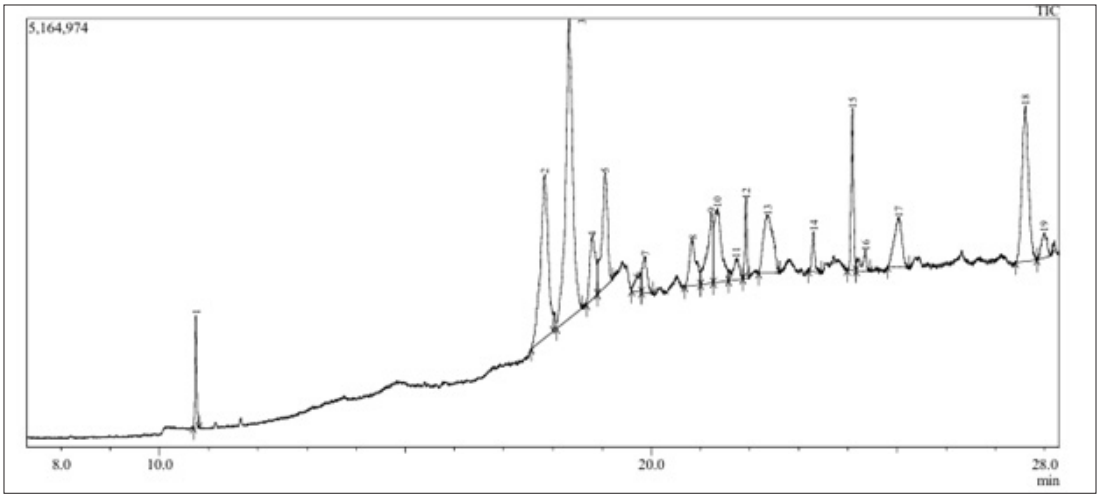

Figure 3: The chromatographic profiles of the volatile compounds in the methanolic oil extract of *Citrus reticulata* pip

Fourier Transform Infrared Spectroscopy analysis

Fourier Transform Infrared spectroscopy (FTIR) was used to characterize the methanolic oil extracts obtained from the *C. reticulata* pericarp and pip (seed). The resulting spectra, shown in Table 5 and Figures 4 & 5, confirmed the presence of various functional groups in both extracts. The peel extract exhibited characteristic absorption bands across 3307.56 to 55.89 cm-1, while the seed extract showed distinct peaks from 3272.54 to 417.97 cm-1. Fourier Transform Infrared spectroscopy provides insight into the functional groups and chemical constituents present in the methanolic oil extract of *Citrus reticulata* pericarp and seed, shown in Table 5. The results indicated the presence of hydroxyl groups found in alcohols and carbonyl functional groups, characteristic of phenolics, esters, and terpenoids, due to the presence of phytochemical constituents. The phenolic –OH groups reveal the potent radical scavenging activity of the pericarp and pip methanolic oil extracts, while hydrocarbon, alkene, and ester groups support the lipid and terpene bioactivity contributing to the *citrus reticulata* pericarp and pips methanolic oil extracts (Zheng et al., 2022).

The findings from the GC-MS and FTIR indicated that the methanolic oil extracts from the pericarp and pip of *Citrus reticulata* contains active compounds that account for antimicrobial and antioxidant properties. This may help in

reducing stress, combating microbial infections and inflammation. Also, the oil extracts from both the pericarp and the seed methanolic oil extracts can be used in the food industry as natural preservative agents and flavor enhancers. The oil extracts can be used in the pharmaceutical industries as antimicrobial additives, and used as anti-aging substances in the cosmetics industry

Table 5: Fourier transform infrared spectroscopy analysis of methanolic oil extract of citrus reticulata pericarp and pip

Peak value (cm^{-1}) Pericarp	Peak value (cm^{-1}) pip	Functional group	Compound
3307.56	3272.54	–OH group	Alcohol
2938	2934.7	C–H group	Alkanes
2833.7	-	C–H group	Alkanes
1634.8,1514.2	1607.72	C=C group	Alkanes
1367.6	1397.70	C–H Stretching	Alkanes
1266.6	1019.02	C–O Stretching	Ethers
101.31	925.16	C–O Stretching	Ethers
55.89	603.90	C–I stretching	Alkyl halides

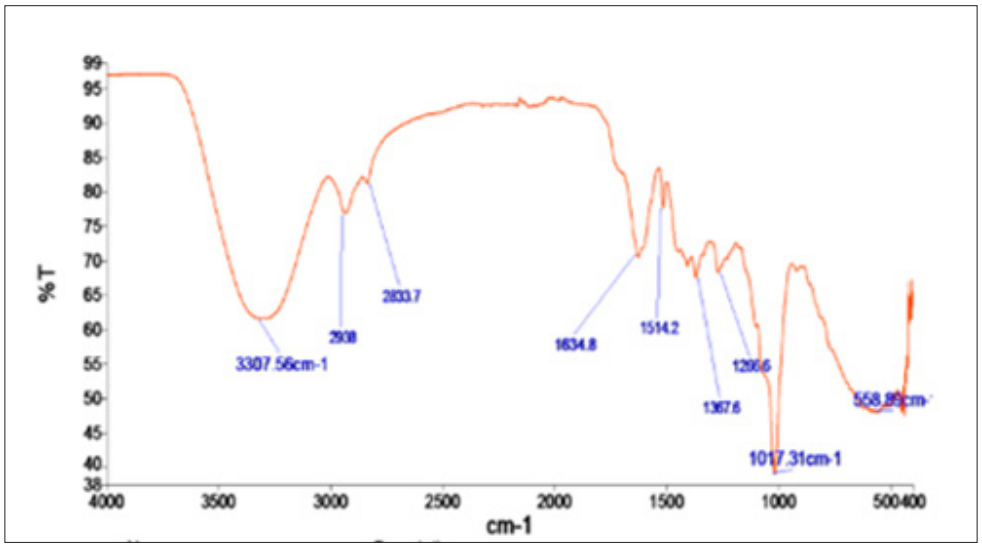

Figure 4: Fourier transform infrared spectroscopy analysis of methanolic oil extract of *citrus reticulata* pericarp

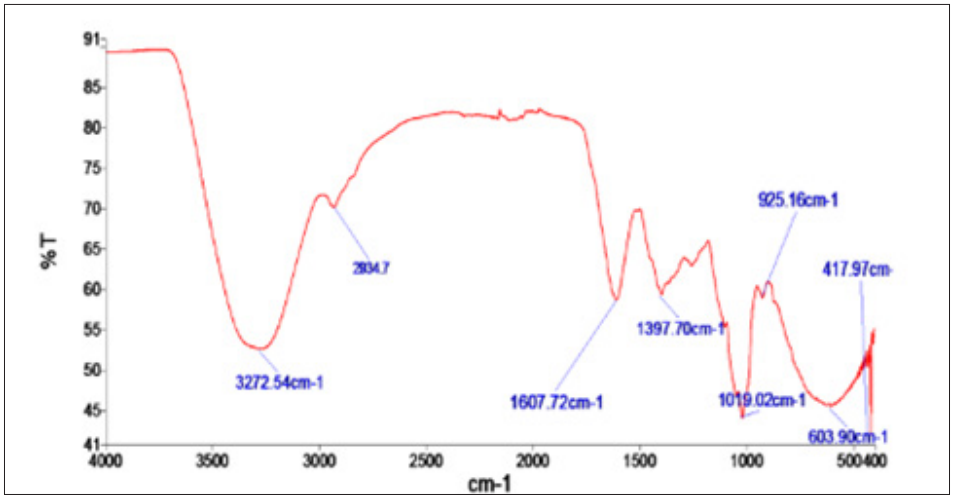

Figure 5: Fourier transform infrared spectroscopy analysis of methanolic oil extract of *citrus reticulata* pip

Conclusion

This comparative study has shown that the physicochemical properties showed that the pericarp had the best and highest oil yield. The saponification and iodine value indicated that the methanolic crude oil extracts are non-drying which indicate their usage in soap manufacturing and vegetable oil based creams and the pips methanolic oil has low rancidity. The methanolic oil extracts from *C. reticulata* contain important phytochemical and active compounds which could be significant to antioxidant and antimicrobial properties. This study identified the potential

of *C. reticulata* wastes as a valuable source of natural active compounds. These findings support further exploration of *Citrus reticulata* extracts for possible pharmaceutical and nutraceutical applications, encouraging their integration into valuable products to promote environmental and economic benefits, and also promoting health as a source of antioxidant and therapeutic agents.

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