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Microwave-Assisted Extraction of Carotenoids from Orange and Mango Peels Using Green Coconut Oil versus Solvent-Based Methods

Jubril Babatunde[1*], Ugoeze Ucheoma Elele[2], Azeh Yakubu[2], Musah Monday[2] and Kolo Salamatu Ibrahim[1]

[1]*Department of Food Science and Technology, Ibrahim Badamasi Babangida University, Lapai, Nigeria*
[2]*Department of Chemistry, Ibrahim Badamasi Babangida University, Lapai, Nigeria*

(*)Corresponding Author's: babatundejubril@gmail.com, 08053334762

Abstract

This study was designed to compare and determine the effects of microwave-assisted green extraction (MAE) and conventional extraction (CE) techniques on the carotenoid content, polyphenolic compounds, and antioxidant activities of mango and orange peels. The MAE was carried out using coconut oil as a green solvent, while the CE involved solvents such as Hexane:Acetone (2:1), Ethanol:Water (20:1), Ethyl acetate:Petroleum ether (1:1), 100% Hexane, and 100% Acetone. Results showed that the total carotenoid content (TCC) obtained from MAE (19.27 ± 0.08 µg/g for orange peels and 25.85 ± 0.08 µg/g for mango peels) was significantly higher ($p < 0.05$) than those from all conventional methods, with the highest CE values being 16.62 ± 0.09 µg/g and 17.55 ± 0.28 µg/g, respectively. Polyphenol contents ranged from 6.39 ± 0.23 to 27.54 ± 0.06 mg GAE/g for orange peels and 5.44 ± 0.08 to 19.51 ± 0.06 mg GAE/g for mango peels, with MAE yielding approximately 1.7-fold higher values compared to the best-performing CE. Furthermore, total antioxidant capacity (TAC) of the carotenoid extracts from MAE (46.94 ± 0.15 and 37.20 ± 0.15 mg AAE/g for orange and mango peels, respectively) was significantly greater ($p < 0.05$) than those obtained from CE. In addition to enhancing the quality and yield of carotenoids extracted from mango and orange peels, MAE demonstrates clear environmental advantages. These findings highlight the potential of microwave-assisted green extraction as a sustainable method for recovering valuable bioactive compounds from fruit peels for use in functional foods, nutraceuticals, natural colorants, and cosmetic formulations.

Keywords: Sustainable extraction, Green extraction, Mango, Antioxidant capacity, Orange, Carotenoids, Bioactive compounds.

Introduction

The growing demand for natural, plant-derived compounds as alternatives to synthetic additives in the food, pharmaceutical, and cosmetic industries has intensified in recent years. This shift is largely driven by health concerns associated with synthetic pigments and food additives, which have been linked to adverse effects such as hyperactivity and allergic reactions, especially in sensitive individuals and children [1]. In response, consumers and regulators increasingly advocate for natural pigments with added health benefits, particularly antioxidants, in food and personal care products [2].

To meet this demand, researchers are exploring the recovery of bioactive compounds such as carotenoids, polyphenols, and dietary fiber from underutilized plant materials and agro-industrial by-products [3]. Mango (*Mangifera indica*) and orange (*Citrus sinensis*) peels, typically discarded as waste, are rich sources of these compounds and hold significant promise for valorization [4]. In Nigeria, It was reported in 2021 that Nigeria's citrus fruit production was 4.11 million tonnes. Citrus fruit production in Nigeria increased from 1.5 million tonnes in 1972 to 4.11 million tonnes in 2021, growing at an average annual rate of 2.20. Large volumes of these peels are generated through fruit processing, presenting both an environmental burden and a sustainable opportunity.

Modern extraction technologies have been developed to improve the yield, safety, and environmental sustainability of phytochemical recovery. Techniques such as supercritical fluid extraction [5], ultrasound-assisted extraction [3], enzyme-assisted extraction, and microwave-assisted extraction (MAE) [2] offer advantages over conventional solvent-based methods, including reduced extraction time, energy consumption, and solvent use. When coupled with edible oils as green solvents, these technologies align with circular economy principles by producing clean, food-safe extracts [6].

Carotenoids are a class of C_{40} isoprenoids responsible for the yellow, orange, and red coloration in many fruits and vegetables. They are generally categorized into (i) carotenes (e.g., β-carotene, lycopene) which consist of only hydrogen and carbon and can be cyclic or linear, (ii) oxycarotenoids (xanthophylls, lutein) which contain hydrogen, carbon, and oxygen in the form of hydroxy, epoxy, or oxygen groups and (iii) xanthophylls (e.g., lutein, zeaxanthin), distinguished by the presence or absence of oxygen atoms [5]. In addition to their role as natural colorants, carotenoids exhibit antioxidant, anticancer, and pro-vitamin A activities, making them valuable in the development of functional foods, nutraceuticals, and cosmetics [7].

This study aims to evaluate and compare microwave-assisted green extraction (using coconut oil) and conventional solvent extraction methods for recovering carotenoids, polyphenols, and antioxidants from mango and orange peels. The findings are expected to support sustainable waste valorization and promote the industrial application of fruit peel extracts as natural functional ingredients.

Materials and Methods

Sample Preparation

Mango (*Mangifera indica*) and orange (*Citrus sinensis*) fruits were purchased from the Lapai local market, Niger State, Nigeria. The peels were manually separated from the pulp, washed thoroughly with distilled water, and freeze-dried at −50 °C for 48 hours (Model: Biobase, BK-FD10P). The dried peels were ground using a laboratory grinder (Model: Waring Excella) and stored in a labelled plastic zipper bags. All samples were immediately stored in a deep freezer at −4 °C until further analysis.

Extraction Methods for Carotenoids from Orange and Mango Peels

Both conventional extraction (CE) and microwave-assisted extraction (MAE) methods were employed for carotenoid recovery. The influence of each method on total carotenoid content (TCC), total phenolic content (TPC), and antioxidant activity was evaluated.

Conventional Extraction (CE)

Conventional extraction was performed following the method of [8], with slight modifications.

Five solvent systems were used:

- Hexane:Acetone (2:1 v/v)
- Ethanol:Water (20:1 v/v)
- Ethyl acetate:Petroleum ether (1:1 v/v)
- Hexane (100%)
- Acetone (100%)

Five grams of each sample (mango or orange peel) was mixed with 20 mL of solvent and vortexed briefly. The extraction was repeated four times until the extracts showed no visible yellow coloration. To aid in solvent removal and phase separation, 20 mL of 0.1% NaCl solution was added. The pooled extracts were evaporated using a hot-air oven at 45 °C until the final volume reached 50 mL. Extracts were stored at −20 °C in amber vials prior to analysis to prevent light degradation.

Microwave-Assisted Extraction (MAE) Using Coconut Oil as Green Solvent

Microwave-assisted extraction was carried out based on the procedure of [2], with modifications. Five grams of dried peel sample was placed in a microwave-safe extraction flask containing 50 mL of coconut oil (1:10 w/v, sample to oil). Extraction was performed in a domestic microwave oven (Samsung MS32J5133BTSA) set at 130 W for a total of 30 minutes. To avoid thermal degradation, the flask was removed every 5 minutes, cooled in an ice bath for 1 minute, and returned for continued exposure, maintaining the sample temperature below 45 °C. Post-extraction, the mixtures were centrifuged at 4500 rpm for 45 minutes (Search-Tech 80-2KS, UK), and the supernatants were stored at −20 °C for further analyses.

Determination of Total Carotenoid Content (TCC)

The TCC of each extract was determined using the method of [3], with slight modifications. For MAE extracts, 3 g of the oil-based sample was diluted with cyclohexane to a final volume of 10 mL. For CE extracts, 10 mL of the solvent-based extract was used directly. Absorbance was measured at 470 nm using a spectrophotometer (SPECORD-250 Plus, Analytik Jena, Germany). Total carotenoids were calculated using the following formula:

$$C = \frac{A\ X\ 10^6}{2000\ \ X\ 100\ X\ d}$$

Where:

- **C** is the carotenoid concentration (mg/kg),
- **A** is the absorbance at 470 nm,
- $\mathbf{E_0}$ is the extinction coefficient of lutein (2000), and
- **d** is the path length of the cuvette (1 cm).

Determination of Total Phenolic Content (TPC)

Total phenolics were determined using the Folin-Ciocalteu colorimetric method as described by [3]. Two milliliters of each extract was mixed with 1 mL of Folin-Ciocalteu reagent and 0.8 mL of 7.5% sodium carbonate. The mixtures were incubated at room temperature for 30 minutes, and absorbance was measured at 765 nm. Results were expressed as milligrams of gallic acid equivalents per gram of dry sample (mg GAE/g).

Determination of Total Antioxidant Capacity (TAC)

The total antioxidant capacity of extracts was determined using the phosphomolybdenum method described by [2]. Two milliliters of each extract was combined with 1 mL of reagent solution (0.6 M sulfuric acid, 28 mM sodium phosphate, and 4 mM ammonium molybdate). The reaction mixtures were incubated in a water bath at 50 °C for 90 minutes. After cooling to room temperature, absorbance was measured at 695 nm against a reagent blank. Results were expressed as milligrams of ascorbic acid equivalent per gram of dry sample (mg AAE/g).

Statistical Analysis

All experiments were performed in triplicate. Data were expressed as mean ± standard deviation. Statistical comparisons between extraction methods and sample types were carried out using one-way analysis of variance (ANOVA), followed by Tukey's post hoc test at a 95% confidence level ($p < 0.05$) using SPSS version 26.0 (IBM Corp., Armonk, NY, USA).

Results and Discussion

This study evaluated the efficiency of microwave-assisted extraction (MAE) using coconut oil (a green solvent) and five conventional extraction (CE) methods in recovering bioactive compounds specifically total carotenoids content (TCC), total phenolic content (TPC), and total antioxidant capacity (TAC) from mango and orange peels. All experiments were performed in triplicate ($n = 3$), and results are expressed as mean ± standard deviation.

Total Carotenoids Content (TCC)

The TCC of mango and orange peels extracted using MAE and conventional solvent systems is presented in **Table 1** and **Figure 1**. For orange peels, the TCC ranged from 2.54 ± 0.06 µg/g (Hexane) to 16.62 ± 0.09 µg/g (Acetone) among conventional extractions, while MAE achieved a significantly higher value of 19.27 ± 0.08 µg/g ($p < 0.05$). Similarly, TCC in mango peels ranged from 3.02 ± 0.10 µg/g (Hexane) to 17.55 ± 0.28 µg/g (Ethyl Acetate:Petroleum Ether) for CE, with MAE yielding the highest content at 25.85 ± 0.08 µg/g.

These findings show that MAE consistently outperformed all conventional methods in both fruit samples. This superior extraction efficiency may be attributed to the microwave-induced cell wall rupture and localized heating, which enhance mass transfer and solvent penetration into plant tissues [2]. Moreover, the use of coconut oil as a green solvent may have contributed to improved carotenoid solubility due to its high lipid affinity, viscosity, and emulsifying properties, which facilitate better pigment dispersion and extraction.

The carotenoid extraction trend for orange peels followed the order: MAE > Acetone > Ethanol:Water > Hexane:Acetone > Ethyl Acetate:Petroleum Ether > Hexane, While for mango peels, the order was: MAE > Ethyl Acetate:Petroleum Ether > Ethanol:Water > Acetone > Hexane:Acetone > Hexane.

These trends demonstrate that polar solvent systems (e.g., Acetone and Ethanol:Water) performed better than non-polar solvents (e.g., Hexane), confirming that carotenoids in fruit peels are more effectively extracted with polar or mixed solvents, particularly under microwave-enhanced conditions.

Additionally, the results from this study align with literature values, which report TCC in orange peels typically ranging between 10–20 µg/g depending on the extraction method [8], thereby supporting the reliability of our data.

The differences in extraction efficiency among methods were statistically significant ($p < 0.05$), confirming the effectiveness of MAE as a green alternative for carotenoid recovery.

Table 1: Total Carotenoid Contents (μg/g) of Orange and Mango Peels extracts

Sample code	Solvent	Solvent Ratio	Solvent volume (ml)	Orange TCC (μg/g)	Mango TCC (μg/g)
H:A	Hexane : Acetone	2 : 1	20	10.11 ± 0.09^{c}	6.11 ± 0.09^{b}
E:W	Ethanol : Water	20 : 1	20	12.51 ± 0.28^{d}	13.94 ± 0.56^{d}
EA:PA	Ethyl Acetate : Petroleum Ether	1 : 1	20	5.85 ± 0.11^{b}	17.55 ± 0.28^{e}
H	Hexane	1 : 0	20	2.54 ± 0.12^{a}	3.02 ± 0.11^{a}
A	Acetone	1 : 0	20	16.62 ± 0.09^{e}	7.44 ± 0.12^{c}
MAE	Coconut oil	1:0	20	19.27 ± 0.08^{f}	25.85 ± 0.08^{f}

Values represent means values of triplicate analysis ± Standard Deviation. Different letters (a, b, c, d, e, f) in the same column indicate a significant statistical difference in the observed data ($p < 0.05$)

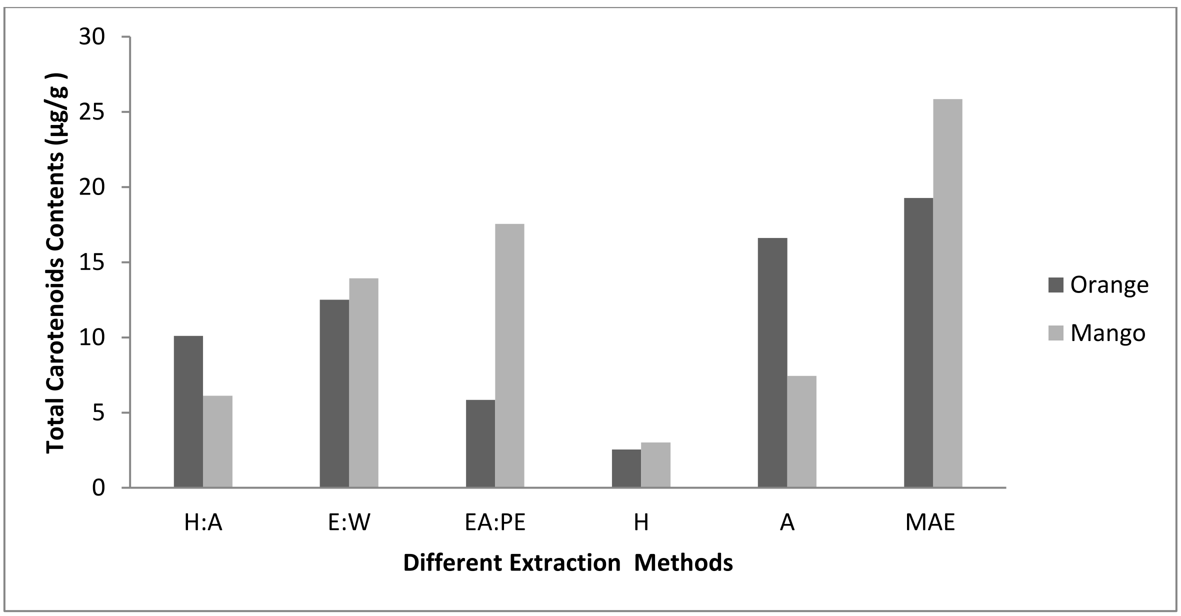

Figure 1: Effect of different extraction methods on the Total Carotenoid Contents (TCC) (μg/g) of orange and mango peel extract.

Total Phenolic Content (TPC)

The total phenolic content (TPC), a key indicator of antioxidant potential, was evaluated for both orange and mango peel extracts and expressed as milligrams of gallic acid equivalents per 100 grams of extract (mg GAE/100 g). The values are presented in Table 2 and Figure 2. Overall, orange peels yielded significantly higher phenolic content than mango peels ($p < 0.05$).

For orange peels, TPC ranged from 5.15 ± 0.11 mg GAE/100 g (Hexane) to 27.54 ± 0.06 mg GAE/100 g (MAE). Notably, the MAE result was approximately 1.7-fold higher than the best-performing conventional extraction method (Ethanol:Water, 16.68 ± 0.06 mg GAE/100 g). In the case of mango peels, TPC values ranged from 4.42 ± 0.05 mg GAE/100 g (Hexane) to 19.51 ± 0.06 mg GAE/100 g (MAE), again showing the superior performance of MAE over all conventional techniques ($p < 0.05$).

These results suggest that microwave-assisted extraction (MAE) using coconut oil as a green solvent is significantly more effective in liberating phenolic compounds from plant matrices compared to solvent-based conventional extraction. The enhanced efficiency may be attributed to microwave-induced rupture of cell walls, which facilitates the breakdown of cellular structures and the release of intracellular phenolics [8].

Furthermore, the polar nature of phenolic compounds allows for better solubility in lipid-rich solvents like coconut oil when coupled with microwave irradiation. However, it is important to note that although microwaves aid in extraction, the localized heat generated may also cause minor degradation of heat-sensitive phenolic compounds—a tradeoff often observed in rapid thermal processes.

Our findings align with those reported by [4], who emphasized the role of green solvents and innovative technologies in improving polyphenol recovery from agro-industrial wastes. The variation in TPC between mango and orange peels is also consistent with their known compositional differences, where citrus peels typically contain higher concentrations of flavonoids and other phenolic acids.

Table 2: Total Phenolic Contents (mg GAE/g) and Total Antioxidant Capacity (mg AAE/g) of orange and mango peels extracts

Sample code	**TPC (mg GAE/g)**		**TAC (mg AAE/g)**	
	Orange	Mango	Orange	Mango
H:A	6.39 ± 0.23^{b}	5.44 ± 0.08^{b}	17.61 ± 0.21^{b}	17.37 ± 0.21^{b}
E:W	5.15 ± 0.11^{a}	12.98 ± 0.11^{c}	17.13 ± 0.18^{a}	19.70 ± 0.18^{c}
A	16.68 ± 0.060^{c}	4.42 ± 0.05^{a}	23.44 ± 0.19^{c}	15.00 ± 0.15^{a}
MAE	27.54 ± 0.06^{d}	19.51 ± 0.06^{d}	46.94 ± 0.15^{d}	37.20 ± 0.15^{d}

Values represent means values of triplicate analysis ± Standard Deviation. Different letters (a, b, c, d,) in the same column indicate a significant statistical difference in the observed data ($p < 0.05$).

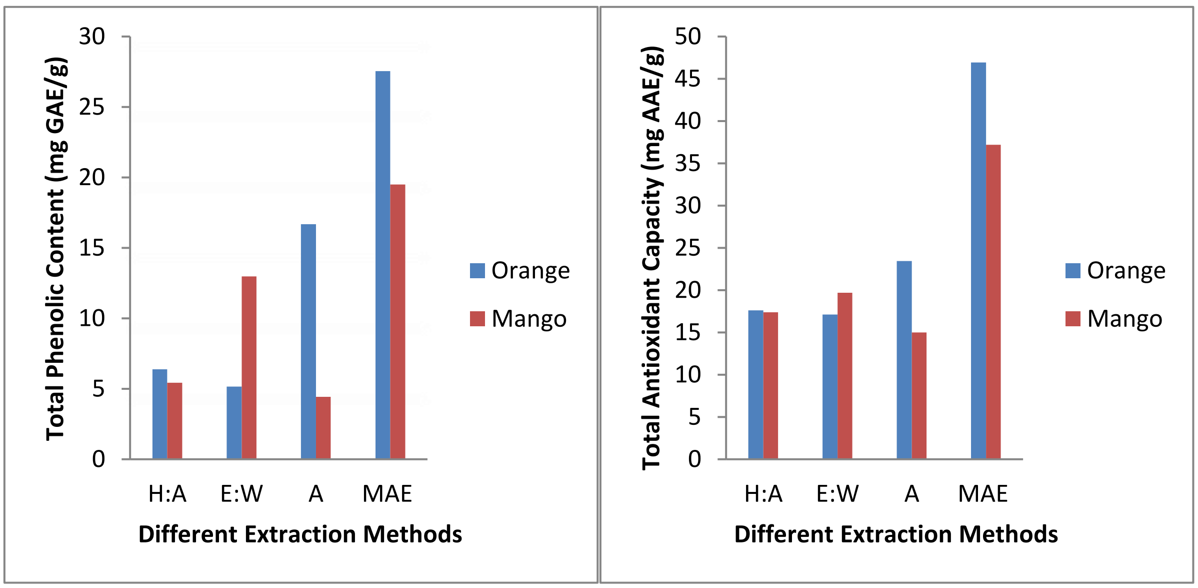

Figure 2: Effect of different extraction methods on Total phenolic contents (TPC) (mg gallic acid equivalent (GAE)/g of extract) and Total Antioxidant Capacity (TAC) (mg ascorbic acid equivalent (AAE)/g of extract) of Orange and Mango peels.

Total Antioxidant Capacity (TAC)

The total antioxidant capacity (TAC) of both orange and mango peel extracts was assessed and expressed in milligrams of ascorbic acid equivalents per gram of dry sample (mg AAE/g). The values are summarized in Table 2 and Figure 2.

For orange peels, TAC ranged from 17.13 ± 0.18 mg AAE/g (Hexane) to 46.94 ± 0.15 mg AAE/g (MAE), while mango peels ranged from 15.00 ± 0.15 mg AAE/g to 37.20 ± 0.15 mg AAE/g, also peaking with the MAE method. In both cases, microwave-assisted extraction yielded significantly higher antioxidant capacity than all five conventional solvent systems ($p < 0.05$).

Compared to previous reports, the TAC of the MAE orange peel extract in this study was higher than the 22.4 mg AAE/g reported for ethanolic orange peel extracts [2]. The enhanced antioxidant capacity observed here can be attributed to the microwave-assisted breakdown of cell walls and improved solubilization of phenolic and carotenoid compounds [8].

The higher antioxidant activity in orange peels relative to mango peels is consistent with their naturally higher flavonoid and phenolic content. Antioxidants neutralize free radicals primarily through hydrogen donation, and the high values recorded here reflect the strong reducing potential of both peel types [8].

The observed TAC extraction trends were as follows:

- **Orange peels**: MAE > Acetone > Hexane:Acetone > Ethanol:Water > Hexane
- **Mango peels**: MAE > Ethanol:Water > Hexane:Acetone > Acetone > Hexane

These trends further highlight the superior performance of MAE, especially when combined with a green solvent like coconut oil.

Conclusion

The effect of various extraction methods on the total carotenoid content (TCC), total phenolic content (TPC), and total antioxidant capacity (TAC) of mango and orange peels was determined. Mango peels exhibited higher TCC, while orange peels demonstrated superior TPC and TAC values. A statistically significant difference ($p < 0.05$) was observed across all solvent systems, underscoring the critical role of solvent selection in maximizing the recovery of bioactive compounds.

Microwave-assisted extraction (MAE), combined with coconut oil as a green solvent, yielded the highest values of TCC, TPC, and TAC compared to all conventional methods evaluated. This suggests that MAE not only enhances extraction efficiency but also aligns with sustainable and environmentally friendly processing goals.

These findings support the potential scalability of MAE for industrial applications in the food, nutraceutical, pharmaceutical, and cosmetic sectors particularly for the valorization of agro-industrial fruit waste. The ability to extract natural antioxidants and pigments using green solvents like coconut oil further emphasizes the relevance of this approach in clean-label product development.

Recommendations for Future Work

Future studies should explore the comparative efficiency of other green extraction technologies—such as ultrasound-assisted extraction (UAE), pressurized liquid extraction (PLE), supercritical fluid extraction (SFE), and enzyme-assisted extraction (EAE)—in recovering carotenoids and phenolic compounds from similar matrices. Such work will be instrumental in validating the effectiveness, cost-efficiency, and scalability of these technologies relative to conventional solvent extraction systems.

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