ULTRASOUND ASSISTED SUPERCRITICAL CO_2 EXTRACTION OF CAROTENOIDS FROM GAC FRUIT

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Summary and Conclusion

CHAPTER V

SUMMARY AND CONCLUSION

The food industry is shifting towards health-oriented products due to consumer demand for improved health, fitness and longevity. This trend, often influenced by marketing, has led to the "super fruit" concept, promoting nutrient-rich fruits. Interest in Gac fruit is growing beyond its native regions, with numerous companies developing innovative health-focused products.

Gac fruit, a tropical vine is valued in India for its nutritional richness, including carotenoids, polyphenols, vitamin E and omega-3 fatty acids. It finds applications in food, health and cosmetic industries, often replacing synthetic colourants. Understanding its engineering properties is vital for optimizing post-harvest processes like sorting and packaging. Mass-based modelling, which predicts fruit mass, improves efficiency, especially for irregularly shaped fruits and offers a cost-effective, accurate grading solution, reducing processing costs.

UAE is an eco-friendly, efficient method that enhances mass transfer and reduces extraction time, especially when combined with SFE, improving yield and quality. SFE, using CO_2 at supercritical conditions (304.25 K, 7.38 MPa), preserves extract quality while being environmentally safe and thermally gentle, making both methods ideal for carotenoid recovery.

The study evaluated the engineering properties of fresh Gac fruits, including physical, thermal, textural and frictional characteristics. Mass modelling of the fruits was conducted based on these physical properties. The effectiveness of four models linear, quadratic, S-curve and power was analyzed, with the most accurate model evaluated using metrics such as the coefficient of determination (R^2) and the standard error of estimate (SEE).

Ultrasound pretreatment of Gac pulp was optimized using RSM, with an amplitude of 80% and a duration of 30 min identified as optimal for maximizing carotenoid recovery, yield and antioxidant activity.

Supercritical CO_2 extraction was performed on both untreated and ultrasound-treated samples under optimized conditions (30 MPa, 328.15 K, CO_2 flow rate 15 g/min and ethanol co-solvent at 3.75 g/min). The extracted oil was analyzed for carotenoid concentration and antioxidant activity, while SEM and FTIR revealed morphological and structural changes in the samples post-extraction.

The results of the above experiments are summarized as following:

Gac fruit has average dimensions with a length of 119.16 ± 25.06 mm, width of 75.72 ± 11.72 mm and thickness of 72.76 ± 11.49 mm. The arithmetic mean diameter (Da) is 89.22 ± 15.89 mm and the geometric mean diameter (Dg) is 86.86 ± 14.94 mm. The aspect ratio (AR) is 64.32 ± 4.72 and the sphericity value (Ø) is 0.734 ± 0.034. Its ellipsoid volume (Vellp) is 373,965 ± 194,809 mm^3 and the average mass is 313.72 ± 149.41 g, with peel (55.36 ± 20.9 g), yellow pulp (136.4 ± 63.05 g), arils (80.84 ± 46.78 g) and seeds (41.03 ± 25.27 g).

True density (ρt) is 439.9 ± 18 kg/m^3, bulk density (ρb) is 764.6 ± 27 kg/m^3 and porosity (ε) is 42.46 ± 1.2%. The interaction coefficients with materials are 0.549 ± 0.004 for stainless steel, 0.3619 ± 0.008 for plywood and 0.1809 ± 0.004 for glass. Thermal conductivity values are 0.5265 ± 0.001 W/m°C for the peel, 0.5856 ± 0.007 W/m°C for the pulp and 0.5392 ± 0.014 W/m°C for the aril. Thermal diffusivity values are $0.1966 \times 10^{-7} \pm 0.0007$ m^2/s for the pulp, $0.1915 \times 10^{-7} \pm 0.0008$ m^2/s for the peel and $0.1927 \times 10^{-7} \pm 0.0006$ m^2/s for the aril. Mechanically, Gac fruit has a hardness of 5.35 ± 0.57 N, cohesiveness of 0.31 ± 0.08 and adhesiveness of -0.01 ± 0.003 J.

Gac fruit has high carbohydrate content, with 98.36 ± 0.45 mg/g in the aril, 35.87 ± 0.86 mg/g in the yellow pulp. Fat content is also significant, especially in the aril (21.44 ± 0.13%), followed by the yellow pulp (3.06 ± 0.30%). The ash content is highest in the peel (6.14 ± 0.56%), then the aril (4.00 ± 0.36%) and the yellow pulp (2.44 ± 0.43%). Carotenoid content includes with the aril showing the highest concentrations: 355.97 ± 1.23 mg/100 g of β-carotene and 298.40 ± 1.08 mg/100 g of lycopene.

In mass modelling, the power model was identified as the most suitable approach. Among the dimensions, geometric mean diameter, surface area and ellipsoid volume stood out as the optimal physical properties, achieving the highest R^2 value of 0.976 and a minimal standard error of estimate (SEE) of 0.07433.

In the ultrasound treatment, increasing the amplitude from 50 - 80% significantly improved the extraction yield, carotenoid recovery and antioxidant activity of the Gac fruit sample. The yield of β-carotene rose from 70.04 to a maximum of 84.49 mg per 100 g of dry sample, peaking at 80% amplitude and 34 min. Carotenoid recovery similarly increased from 68.16% to a high of 82.83% under these conditions. Antioxidant activity also improved from 39.07 - 56.06% as the amplitude increased from 50 to 80%, though it decreased slightly to 54.48% at 100% amplitude, with the highest antioxidant activity achieved at 80% amplitude and 34 min.

Ultrasound pretreatment proved to be a highly effective enhancement technique for SC-CO_2 extraction of Gac oil. By using ultrasound to pre-treat the Gac pulp, the extraction process became more efficient, as it disrupted the fruit's cellular structure, improving the release of oil and bioactive compounds. This resulted in a notable reduction in the extraction time and an increase in the oil yield from 33 - 41%, showcasing a significant improvement.

Furthermore, the UAE enhanced the recovery of carotenoids, yielding a higher concentration of 0.481 μg/mL compared to 0.476 μg/mL obtained through conventional supercritical extraction. This slight but meaningful increase demonstrates the method's effectiveness in preserving and extracting these valuable compounds. Additionally, the antioxidant activity of the oil improved remarkably, rising from 60.48% in normal extraction to 71.60% with ultrasound assistance, indicating a superior quality of extracted oil with enhanced bioactive properties.

Scanning electron microscopy showed that untreated samples had smooth, intact cell walls with minimal disruption. Ultrasound treatment created rough, porous surfaces due to cavitation effects. Supercritical CO_2 treatment increased surface roughness and combining both methods amplified these changes. This enhanced porosity improved compound release during extraction.

FTIR analysis of Gac oil confirmed the presence of carotenoids and bioactive compounds, with a strong peak at 1750 cm^{-1} indicating carotenoids and triglycerides. The ultrasound-treated oil showed a larger peak, suggesting a higher concentration of β-carotene.

Following conclusions were derived based on the findings:

- Engineering properties like dimensions, density and texture make Gac fruit suitable for post-harvest machine design. Its high carbohydrate and carotenoid content, particularly β-carotene and lycopene in the aril, highlights its nutritional and antioxidant benefits.
- The power model, based on properties like geometric mean diameter and ellipsoid volume, was the best fit for mass modelling, achieving high accuracy ($R^2 = 0.976$).
- Using RSM, ultrasound pretreatment was optimized at 80% amplitude for 30 min. The highest β-carotene yield (84.49 mg/100 g dry sample) and carotenoid recovery (82.83%) were achieved under these conditions.
- Ultrasound assisted supercritical extraction improved the yield by 8%, with a carotenoid concentration of 0.481 µg/mL and boosted antioxidant activity by 11% compared to untreated supercritical extraction.
- SEM analysis confirmed that ultrasound treatment disrupted cell walls, creating a porous structure that facilitated carotenoid release, while FTIR likely indicated the presence and structural integrity of the carotenoids post-extraction.

Scope of future work

- Conduct bioavailability studies to assess the absorption efficiency of extracted carotenoids in food products or supplements.

- Examine the stability of extracted carotenoids and antioxidants during storage and within food products to evaluate how processing influences their nutritional value over time.
- Assess how ultrasound-assisted supercritical extraction compares with other extraction methods, such as microwave-assisted or enzymatic extraction, in terms of yield, purity and antioxidant properties.
- Explore the use of extracted Gac oil rich in carotenoids in various food formulations to enhance colour, nutrition and shelf life.