

**MICROWAVE HYDRO-DISTILLATION AND ULTRASOUND
ASSISTED EXTRACTION OF BIOACTIVE COMPONENTS
FROM NUTMEG PERICARP**

by

NOORBINA RAZAK

(2022-18-001)

THESIS

Submitted in partial fulfillment of the requirement for the degree of

MASTER OF TECHNOLOGY

IN

AGRICULTURAL ENGINEERING

(Processing and Food Engineering)

Faculty of Agricultural Engineering and Technology

Kerala Agricultural University



Department of Processing and Food Engineering

KELAPPAJI COLLEGE OF AGRICULTURAL ENGINEERING AND

FOOD TECHNOLOGY

TAVANUR, MALAPPURAM - 679573

KERALA, INDIA

2024

Summary and Conclusion

CHAPTER V

SUMMARY AND CONCLUSION

Bioactive compounds are phytochemicals which promotes the health beyond the basic nutritional value of the food. They contribute to the antioxidant, anti-inflammatory, antimicrobial and other therapeutic properties which render them highly valuable across various industries. Nutmeg fruit is the donor of two high economic valued spices, in which the fleshy part named as pericarp is wasted because of its unknown benefits. Although it has some limited application in the production of jam, jelly, squash, and pickles, they are considered as of low economic value and is underutilised. After the fruit got matured, the fleshy pericarp splits itself revealing the highly valued nutmeg seed and mace and is collected. The pericarp is then disposed or left stacking under the tree, causing fungal attack due to its highly perishable nature leading to environmental pollution. However, it is a rich source of active phytochemicals such as monoterpenes, ethers, aldehydes, polyphenols, flavonoids, and tannins which contributes to maintaining health and protection from chronic diseases which supports health and protection against chronic diseases.

Diversifying nutmeg pericarp by extracting its valuable compounds as essential oil and oleoresin could enhance its economic value. Essential oils are the volatile aromatic compounds extracted from the plant material through hydrodistillation. After the extraction of essential oil, the non-volatile compounds such as fixed oils, resins and pigments are remaining in the plant matrix which is referred to as oleoresin, and are extracted through solvent extraction method. These conventional extraction methods often result in lower extraction efficiency and quality, longer extraction time, high energy requirement and environmental pollution. To circumvent these issues, eco-innovative technologies such as Microwave Assisted Hydro-distillation (MAHD) for essential oil extraction and Ultrasound Assisted Extraction (UAE) for oleoresin extraction offers sustainable results by improving extraction efficiency and quality while reducing the extraction time, solvent consumption, and energy consumption.

Microwaves are the electromagnetic waves in the frequency ranging from 300 MHz to 300 GHz. The microwaves generate internal volumetric heating which is rapid and uniform causing the effective heat and mass transfer across the cell. The ionic conduction and dipole rotation induced heating evaporates the in-situ water causing the rupture of cell wall and oleiferous glands facilitating the release of essential along with the escaping water vapour. This mechanism extracts the essential oil within a shorter duration with reduced energy consumption. For the extraction of oleoresin, the ultrasonic pretreatment was employed to improve the extraction efficiency by enhancing mass transfer. The ultrasound is the sound wave in the frequency range of 20 kHz to 100 kHz which could alter the physico-chemical properties of plant material. They alter the cell structure by the phenomenon named as cavitation which is the production, growth, and collapse of microscopic bubbles. Sonication facilitates higher mass transport of bioactive constituents from the plant material to the solvent media via three combination effects mainly cavitation, mechanical agitation and thermolysis. It completes the extraction in a short time, reducing the thermal damage to the valuable components.

Therefore, these two technologies were adopted in the current study to optimize the process parameters for the efficient extraction and to obtain better quality extracts with significant economic potential. The MAHD process for the essential oil extraction was carried out in a microwave reactor of 800 W which acts as the radiation source with an extraction unit mounted above the reactor. The extraction unit consists of oil collection and separation column, condenser, supporting stand and an energy meter. The supporting stand provides additional support to the extraction system and the energy consumed for the extraction was measured using a single-phase energy meter. A digital control panel in the reactor sets the microwave power and exposure time for the experiment. The water vapour and volatile essential oil released due to the internal volumetric heating of microwave was passed out of the reactor and then condensed and collected in the oil collection and separation column. After completion of the extraction process, the oil and water collected in separate layers due to the density difference and oil is collected and kept in an amber coloured for storage.

An ultrasound bath of 10 L capacity with chiller was utilised for the ultrasonic pretreatment for the extraction of oleoresin. The ultrasound bath has the provision to set the temperature and time of sonication. The ultrasound generated by the piezoelectric transducer provided at the base of the ultrasound bath generates cavitation effect facilitating the cell wall rupture and enhancing the mass transfer.

Design-Expert version 12 was used to frame the experimental design and optimisation of parameters. For the MAHD process of volatile essential oil extraction, Box-Behnken design framed the experimental design with the selected process variables of solid to water ratio (1:4 to 1:8 g/ml), exposure time (1 to 2 h) and microwave power (320 to 640 W) and the responses were essential oil yield, specific gravity, and energy consumption. UAE was employed for extracting the remaining non-volatile oleoresin from the MAHD extracted samples, and the Central Composite design framed the experimental design. The process variables such as solid to solvent ratio (1:4 to 1:6 g/ml), sonication time (10 to 30 min) and types of solvent (ethanol and ethyl acetate) were selected for optimizing the response variables of oleoresin yield, total phenolic content (TPC) and total flavonoid content (TFC).

The optimised conditions for essential oil yield were determined as, 1:8 g/ml, 1.5 h with 320 W power for obtaining a maximum yield of 0.4% with minimum energy consumption of 0.6 kWh and as specific gravity of 0.917. Maximum oleoresin yield, TPC and TFC of 5.524%, 66.449 mg GAE/g and 4.284 mg QE/g respectively were observed for the process variables of solid to solvent ratio of 1:5.42 g/ml, sonication time of 30 min, and ethanol as the solvent for the extraction of oleoresin.

The comparison of MAHD and UAE with conventional methods were done for both the essential oil and oleoresin. For the essential oil extraction, both MAHD and HD showed similar extraction yield of 0.4%. However, a significant reduction in the extraction time was noticed in the case of MAHD (1.5 h) when compared to HD (4.5 h). Also, MAHD required only about half energy than HD for extracting the same amount of yield. The effect of ultrasound pretreatment in solvent

extraction was also compared with conventional solvent extraction, which demonstrated that UAE achieved a higher yield in a shorter time (5.6% in 1.5 h) than conventional solvent extraction (4.8% in 2.5 h).

The characterization of the extracted bioactive compounds from MAHD and UAE were compared with conventional hydrodistillation and solvent extraction respectively for determining the efficiency and quality of extraction. The optimized samples from novel technologies exhibited improved physicochemical, and microstructural properties when compared to conventional extraction processes. Chromatographic analysis for identifying and quantifying bioactive compounds in essential oil showed that MAHD extracted high concentration of major compounds such as myristicin, elemicin and terpenes, indicating superior quality of essential oil. Also, the UAE extracts exhibited high antioxidant activity with high TPC and TFC contents demonstrating the efficiency of ultrasound in obtaining high quality extracts. The SEM images illustrated the detailed insights into the microstructural changes in the material. The images revealed that MAHD and UAE outperformed conventional extraction in effectively rupturing the cell wall structure. This enhanced cell wall rupture facilitated a higher mass transfer thereby improving the extraction efficiency within a short time.

These findings suggests that the extraction of bioactive compounds from nutmeg pericarp employing MAHD and UAE holds a significant economic potential with high extraction efficiency and quality by minimizing environmental pollution and supporting a circular economy.

Suggestions for future work

1. The synergistic effect of ultrasound and microwave in extracting the essential oil can be studied
2. For extracting oleoresin, ultrasonic bath was utilised in this study, the effect of ultrasonic probe in extraction could also be put to further research
3. Conventional commercial level solvent extraction methods such as percolation extractor may be employed after US pretreatment for better scale up.