

Performance of Green Solvent and Microwave in Developing Sustainable Extraction of Functional Compound and Hydrodistillation Essential Oils from Cinnamon

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ABSTRACT

Southeast Asian cinnamon contains functional compounds and essential oils that act as natural and beneficial antioxidants. This research developed and evaluated for Microwave-Assisted Hydrodistillation (MAHD) to combine simultaneous extraction of phenolic compounds and distillation of essential oil using a green solvent - Natural Deep Eutectic Solvent: Citric Acid-Sucrose (NADES-CAS). This study aimed to evaluate the performance of NADES-CAS in collecting functional compounds (phenolic and proanthocyanidins) and essential oils using MAHD. The MAHD was conducted using NADES-CAS with a molar ratio of 1:1 in concentrations of 1% under microwave power of 640, 720, and 800 W. The performance was evaluated using Peleg's equation as a kinetic approach. The microwaves increased the value of EC, TDS, and solvent temperature but decreased the density and viscosity, supporting MAHD. Peleg's kinetic model describes an

increase in microwave power increase the rate constant (B_0) and capacity constant (C_e). The B_0 values of phenolic and proanthocyanidin compounds were in the range of 0.0264-0.0397 mg/mL.min and 0.0388-0.1117 mg/mL.min, respectively. Meanwhile, the C_e value of these compounds were in the range of 0.5349-0.6607 mg/mL and 0.5529-0.9439 mg/mL, respectively. The model accurately predicting the profiles of each compound concentrations during MAHD. The combination of MAHD and NADES successfully distilled essential oils from cinnamon powder with significant components such as Cinnamaldehyde and minor components such as caryophyllene, benzaldehyde, and

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pinene. This innovation is potential to be applied to accelerate the simultaneous extraction of phenolic compounds and distillation of essential oils from cinnamon.

Keywords: Cinnamon, essential oil, green solvent, microwave-assisted hydrodistillation

INTRODUCTION

Cinnamon, a tropical spice native to Southeast Asia, is prized for its aroma and bioactive compounds. It is derived from the inner bark of *Lauraceae* trees and used in both culinary and medicinal contexts (Pages-Rebull et al., 2024). Its compounds exhibit antioxidant, anti-inflammatory, antiparasitic, anticancer, and antidiabetic properties (Zhang et al., 2024), with important benefits in reducing lipids to combat heart disease and high cholesterol (Maiercan et al., 2017). Functional compounds in cinnamon can act as natural antioxidants and antimicrobial food preservatives, which can be extracted for use in food products. Additionally, cinnamon bark contains valuable essential oils which can be obtained through distillation (Deshi et al., 2024).

Extraction is a process of separating target compounds from a mixture using suitable solvents, thus facilitating the isolation and preservation of bioactive substances (Wang et al., 2024). Functional antioxidant compounds in cinnamon, such as polyphenols, flavonoids, saponins, tannins, and phenols, can be effectively extracted. Meanwhile, essential oils, which also have high economic and functional value, can be obtained through both extraction and distillation methods (Loto, 2020).

Although conventional extraction is widely applied, it has several limitations, including high energy consumption, longer processing times, and a risk of degrading volatile compounds. Besides conventional extraction, microwave-assisted extraction (MAE) and ultrasound-assisted extraction (UAE) have been widely developed in previous research (Izza et al. 2023; Purdi et al., 2023). Both of these methods are expected to accelerate the extraction process, maximise the diffusion process, use low energy, and maintain the structure of active compounds (Guddi & Sarkar 2024). However, these methods cannot directly separate the essential oils. Thus, the extracted essential oils must be separated using evaporation and condensation in a distillation system.

To save energy and cost, an innovative method combining extraction and distillation simultaneously in a unit called a Microwave-Assisted Hydrodistillation (MAHD) method is proposed. In previous research, Jeyaratnam et al. used MAHD for extracting cinnamon oil. However, it focussed on analysing the results of cinnamon essential oil (Jeyaratnam et al., 2016a; Jeyaratnam et al., 2016b). The present research is proposed with a novelty, which is not only in analysing the simultaneous extraction of bioactive compounds and essential oils from cinnamon using MAHD, but also in exploring the mechanisms and kinetics of extracting bioactive compounds from cinnamon powder while distilling the cinnamon oils simultaneously.

NADES is reported as an environmentally friendly solvent for extracting bioactive compounds, such as phenolic compounds, more effectively than organic solvents (Adeel et al. 2020b). NADES is a green solvent that is safe and non-flammable. It has the right chemical-physical properties for cinnamon extraction (Cravotto et al. 2024b; Mulia et al. 2015). Additionally, NADES can extract both polar and nonpolar compounds. NADES mainly utilises carboxylic acid compounds, choline chloride (ChCl), and hydrogen bond donors (Cravotto, et al. 2024a). A type of NADES used in this MAHD research is citric acid and sucrose (CAS). This solvent is expected to achieve a good synergy and interaction with the microwave, thereby enhancing the extraction process. NADES-CAS has also proven effective in extracting bioactive compounds from sorghum, rice, and others (Susanti et al. 2021). This solvent has also been reported to maintain some functional compounds in cinnamon extract, such as polyphenols, flavonoids, saponins, tannins, and phenols that are susceptible to degradation by the process (Segatto et al. 2022). Research on applying microwaves and utilising green solvents to accelerate the extraction of antioxidant bioactive compounds from sorghum, rice, and other sources has been conducted by previous research (Izza et al. 2023; Susanti et al., 2021).

Additionally, the novelty of this research lies in the design of the MAHD system and the application of Natural Deep Eutectic Solvents-Citric Acid Sucrose (NADES-CAS) as a food-grade, environmentally safe solvent for the extraction of bioactive compounds and cinnamon oils. In this research, the physical characteristics of NADES-CAS are explored, and its effects on extraction performance are analysed, which has not been explicitly addressed in previous studies. Previous research has evaluated and demonstrated the characterisation of NADES-CAS and its potential as a green solvent for extraction (Savi et al., 2019). Furthermore, NADES-CAS has been applied in microwave-assisted extraction of olive oil (Bonacci et al., 2020) and ultrasound-assisted extraction of date sugar (AlYammahi et al., 2023).

The simultaneous extraction and distillation in MAHD using NADES-CAS is expected to be an innovative method that increases energy efficiency and reduces processing time through an environmentally friendly process. The microwave radiation through the solvent likely interacts with the cinnamon powder and solvent, causing uniform heating and vibration, leading to faster and more effective extraction and release of bioactive compounds and essential oils from the cinnamon powder (Adeel et al. 2020a). This research also explores the extraction mechanism using a kinetic approach, which has not been studied in the previous related paper, as a novelty of this paper. MAHD utilises microwaves, and NADES-CAS is expected to accelerate the extraction and distillation processes simultaneously, thereby making them more efficient.

In the current research, the application of green solvent needs to be combined with the right MAHD power to obtain positive synergy and accelerate the collection of functional cinnamon compounds. This study aimed to design and test the performance of MAHD

and green solvent to accelerate the collection of essential oils and bioactive compounds of cinnamon simultaneously by determining the optimal microwave power. First, this research characterised the properties of NADES-CAS under microwave conditions. Then, it was applied to test its performance in MAHD at several microwave powers for the extraction of phenolic and proanthocyanidin compounds, as well as its capability to collect essential oils from cinnamon powder. The extraction performance was evaluated through a kinetic approach using a Peleg model. Additionally, the quality of the resulting essential oil was also evaluated. The result of this research is expected to be applied to enhance the simultaneous extraction of bioactive components and essential oils from cinnamon, compared to conventional methods. It can also serve as a reference for increasing the use of green solvents over organic solvents, which have high toxicity and can harm both health and the environment, thereby supporting sustainable chemistry and pharmacy. The successful integration of NADES-CAS with the MAHD process highlights an environmentally friendly strategy for the extraction of cinnamon compounds.

MATERIALS AND METHODS

Materials and Reagents

The material used in this extraction process was cinnamon obtained from the Beringharjo Market, Yogyakarta, Indonesia. The primary reagents used in this research were citric acid, sucrose, and distilled water to produce NADES-CAS. Other reagents used for the evaluation of phenolic and proanthocyanidin contents were Folin–Ciocalteu reagent, Na_2CO_3 , butanol, HCl, iron reagent, and $\text{FeNH}_4(\text{SO}_4)_2$. All reagents were analytical grade from Merck (Darmstadt, Germany).

Preparation of NADES CAS

The green solvent, 1 M NADES-CAS, was prepared from citric acid (CA) and sucrose (S) in a molar ratio of 1:1 in 1 L of aquadest. Then, the mixture was homogenised using a probe ultrasonic with a power amplitude of 80% at a frequency of 20 Hz to accelerate the homogenous condition using its acoustic power that induced bubbles, compression, and rarefactions in the mixture (Medina-Torres et.al, 2017). The ultrasonic equipment was from Hangzhou Dowell Ultrasound Tech. Co., Ltd. with a model number DW-SD20-1200H, a power of 1.2 kW, and a 220 V / 20 kHz voltage. The initial period of the ultrasonic method was 30 minutes, and then it was stopped temporarily to adjust the environmental temperature to prevent overheating. After that, the process continued for 30 minutes. It was sufficient to ensure a homogenous mixture of NADES-CAS. The mixture of CA and S was diluted in distilled water to reach its concentration of 1%.

The Extraction and Hydrodistillation using MAHD

The extraction and hydrodistillation of volatile compounds from cinnamon powder were conducted in a system of MAHD constructed from the modification of an Electrolux Microwave EMM2308X with a capacity of 23 L, maximum microwave power of 800 W, and a voltage of 220–240 V/ 50 Hz. The microwave had panel control to set the temperature, a magnetic stirrer speed in a microwave flask, microwave power, and time. The extraction temperature was controlled by a temperature controller and a CKC Tinner DH485 (H5CN) indicator. A magnetic stirrer was installed at the bottom of the microwave flask to make stirring. The extraction scheme with MAHD is shown in Figure 1. The extraction was conducted at the stirrer speed of 1600 RPM and a temperature of 100 °C using 1% NADES-CAS. Furthermore, the flask containing the solvent was inserted into the microwave chamber. The temperature profile of the solvent was measured using a thermocouple. This process was conducted at microwave power levels of 640, 720, and 800 W, corresponding to 80%, 90%, and 100% of the maximum microwave power, respectively. The extraction of phenolic compounds, proanthocyanidin compounds, and essential oils was conducted in a flask exposed to microwave radiation. The phenolic and proanthocyanidin compounds, as antioxidant compounds, were measured from the bulk of solvent during the extraction. The essential oil was evaporated and distilled in the distillation system and accommodated in the essential oil container.

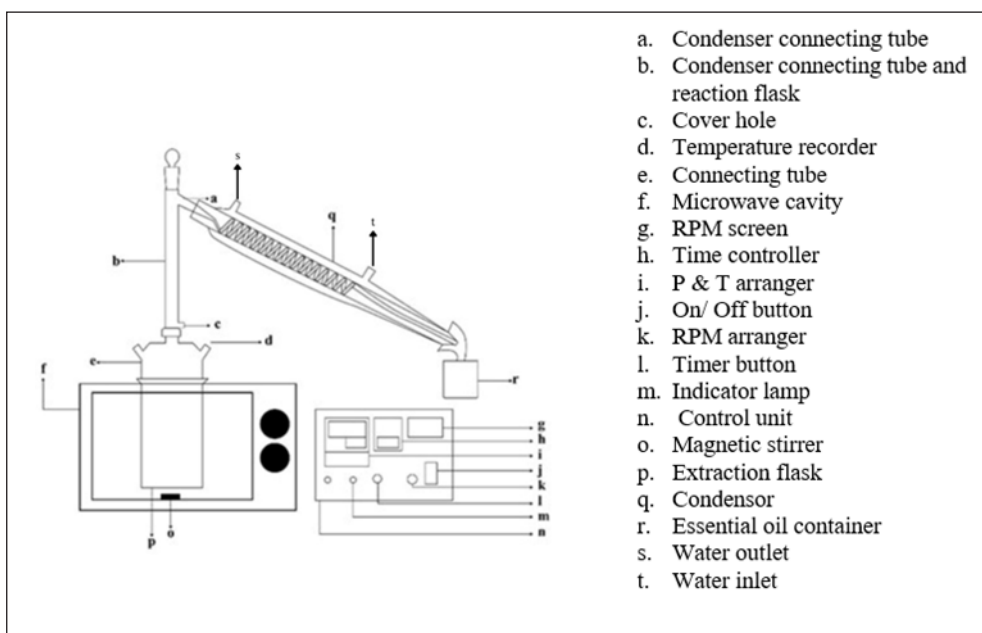


Figure 1. The schematic of Microwave-Assisted Hydrodistillation

Determination of Proanthocyanidins (PA) Content in the Extract

The PA content was determined through the acid butanol assay (de Souza et al., 2018; Susanti et al., 2020). It has been reported as the best method for determining PA concentration and has been widely reviewed in previous studies (Susanti et al. 2020). In this method, a 6 mL acid butanol was mixed with 1 mL of the extract sample and 0.2 mL of $\text{FeNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ reagent, then vortexed until homogeneous and incubated at 90 °C. After 50 minutes, the sample was removed and waited until a normal temperature. The final step was to read the absorbance value using a UV-Vis Spectrophotometer (Mesulab, type N₂S, China) with a wavelength of 550 nm. The result was stated as mg/mL.

Determination of Total Phenolics Content (TPC) in the Extract

Polyphenol content was determined through the Folin-Ciocalteu Test method (Kalpoutzakis et al. 2023). This method has been considered the best method for determining polyphenol content (Yilmaz et al. 2021). This method used seven test tubes containing 15 mL of distilled water and 1 mL of Folin and Ciocalteu's phenol reagent. Each test tube was filled with 1 mL of sample test solution, 1 mL of calibration standard solution, or 1 mL of distilled water. Each test tube was mixed well and left for 6 minutes. Then, 3 mL of sodium carbonate solution was stirred in each tube. Then, the test tubes were placed in the heating block for 120 minutes. As much as 1 mL of solution was transferred into the cuvette and measured for its absorbance at 765 or 760.5 nm after focussing on the spectrophotometer with a blank. The standard solution was measured first, then the sample test solution (Kupina et al. 2019).

The results of cinnamon extraction were tested for polyphenol content. The polyphenol test was carried out using the Folin-Ciocalteu method. The extract of cinnamon (0.5 ml) was reacted with 2.5 mL of 0.2 N Folin Ciocalteau and 2 mL of Na₂CO₃. Next, it was incubated at 50 °C for 5 minutes, and then its absorbance was tested using a UV-Vis Spectrophotometer (Mesulab, type N₂S, China) at a wavelength of 550 nm.

The Evaluation of Extraction Rate and Maximum Capacity of Extraction using the Peleg Model

The performance of MAHD was analysed using the Peleg kinetic model, which describes the relationship between water content and solute absorption time (Oroian 2017). This model was relevant for predicting TPC and PA concentration in extracts during MAHD. In the Peleg model, the C_0 value indicates the initial TPC and PA concentration, while B_0 (extraction rate constant) and C_e (capacity constant) values indicate the extraction rate and capacity. Additionally, this model was also relevant to releasing solutes into water. Using the Peleg model, the extraction curve (TPC vs time and PA vs time) was fitted to the absorption curve (water content vs time). The equation of the Peleg model is presented in Equation(1). For the initial condition $C_0 = 0$, Equation 1 was rearranged into Equation 2. Equation 2 is expected to provide a reasonable prediction of the PA concentration in the extract during

MAHD. The TPC and PA concentration in the solvent at a given time is denoted as $C(t)$ in mg/mL. The application of the Peleg model as a kinetic approach was evaluated using some validation parameters, namely the Sum Squares of Error (SSE), Root Mean Squared Error (RMSE), Chi-square (X^2), and Coefficient of Determination (R^2) (Azeez et al. 2019).

$$C(t) = C_0 + \frac{t}{\frac{1}{B_0} + \frac{t}{C_e}} \quad [1]$$

$$C(t) = \frac{t}{\frac{1}{B_0} + \frac{t}{C_e}} \quad [2]$$

Determination of Physicochemical Characteristics of Aqueous Extract

Physicochemical characteristics of the resulting aqueous extract were determined, including total dissolved solids (TDS), electrical conductivity (EC), pH, viscosity, and density. During the MAHD process, liquid samples were collected at 30 minutes time of intervals from minute at 30 to 150 to monitor changes in extract properties over time. TDS and EC values in the cinnamon extract were measured using the HORIBA B-173 Conductivity Meter. The EC value indicates the presence of phenolic compounds, while the TDS value indicates the solid material contained in the granules. The density was measured using the ratio between the mass and volume using a pycnometer. Meanwhile, viscosity was determined using a Viscometer Brookfield RTV (Ametec Inc, America) operated at 20 rpm with spindle number 1 (Susanti et al., 2025). The pH value was measured using a Digital pH Meter with mech DrGray.

Gas Chromatography–Mass Spectrometry (GC–MS) Analysis

GC–MS analysis was performed to verify the presence of volatile constituents in the cinnamon essential oils resulting from the best-performing MAHD power treatment. A portion of the extracted oils was introduced into the GC–MS system for compound separation and identification. The instrument was equipped with an Elite-5MS capillary column, and helium served as the carrier gas throughout the run. The detected molecular ion fragments were interpreted by comparing their mass spectra with reference profiles from the NIST Library incorporated in the instrument's software.

RESULTS AND DISCUSSION

Characteristics of the NADES-CAS under the MAHD System

The capability of a solvent to extract bioactive compounds depends on its ability to reach the position of the compound, to bind and bring them out from the inner part of natural resources to the bulk of the solvent. This research evaluated the characteristics of NADES-

CAS as a green solvent in the extraction of bioactive compounds and essential oils from cinnamon through the MAHD method. The parameters of the NADES-CAS characteristics included density, viscosity, total dissolved solids, electric conductivity, and temperature during the MAHD process.

Density of the NADES-CAS

The density of the solvent was evaluated at various MAHD powers, namely 640 W (80%), 720 W (90%), and 800 W (100%). The density is one of the parameters used to evaluate the solvent properties to support the extraction process, besides the solubility in water (Kurniawan et al. 2022). The density of the solvent is shown in Figure 2. It illustrated the relationship between time and density under the MAHD system at various power values of 640, 720, and 800 W.

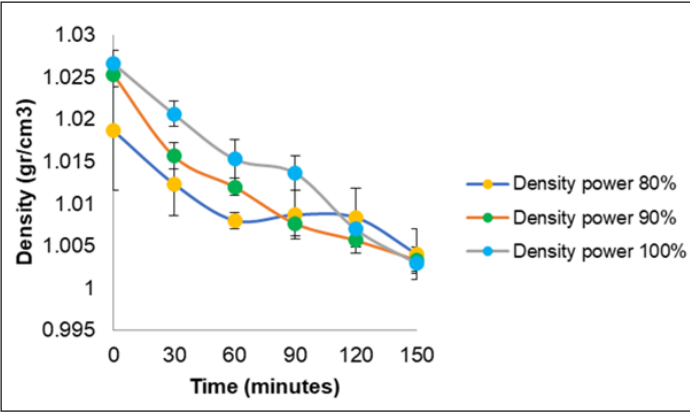


Figure 2. Density value of NADES-CAS during the MAHD process

The density values tended to decrease with increasing extraction time. The longer the extraction time under the microwave, the lower the density of solvent, although the change was not significant. The higher the power of MAHD, the higher the solvent density. The increase in power caused a higher temperature and the evaporation of solvent, which affected its density. This proves that the greater the power used in extraction, the lower the density of the solvent. The results showed that the density value of NADES was higher (1.005 mg/mL to 1.027 mg/mL) than that of pure water (1.0000 mg/mL) (Pires et al., 2022; Susanti et al., 2025).

Viscosity of the NADES-CAS

Viscosity plays a vital role in supporting the solvent's mass transfer into the cinnamon's inner part to collect the substances. Figure 3 shows the profile of the solvent's viscosity

during the MAHD. There was a downward trend in the solvent viscosity corresponding to an increase in the extraction time, although it was not statistically significant. Nevertheless, it was supposed to affect the diffusion of solvent into the pore and internal part of the material to accelerate the extraction.

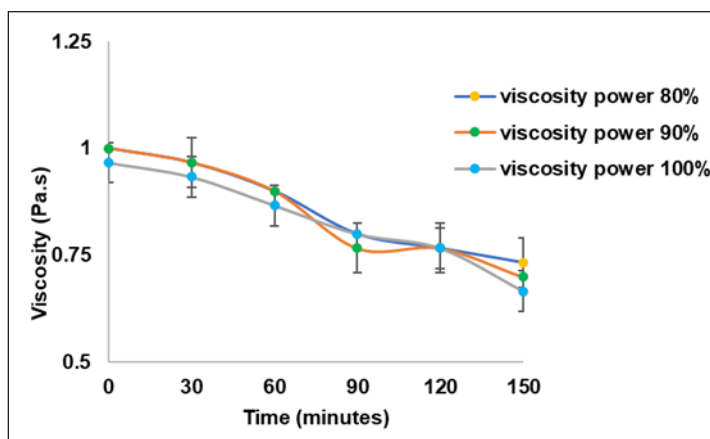


Figure 3. Viscosity value of NADES-CAS during the MAHD process

The decrease in viscosity during the MAHD can be correlated with the increased movement of molecules due to the kinetic energy from microwaves. This particle's movement was expected to accelerate the diffusion of the solvent into the material, thereby extracting the functional compound. The increase in time can cause viscosity to decrease (Mahyati & Azis 2019). The viscosity profiles exhibited similar patterns across the different MAHD power levels. In the extraction method using MAHD with 1% NADES-CAS at various powers, controlling this viscosity is a crucial factor in optimising extraction results to produce essential oils.

Total Dissolved Solids (TDS) of the NADES-CAS

The physical property parameters characterising NADES in this research can be measured by determining the TDS value. Figure 4 shows that the TDS value of NADES-CAS increased significantly with increasing microwave power, with the highest value of 782 mg/L obtained at 800 W and the lowest value of 260.3 mg/L at 640 W. The increase in TDS indicated an increase in the concentration of dissolved solids during extraction. A higher microwave power resulted in more intense heating, thus promoting greater evaporation of water while the solutes remain in the system. When the volume of water decreased, the solution became more concentrated, resulting in higher TDS values. This also reflected the release of more solid compounds from the cinnamon matrix into the NADES solvent during extraction.

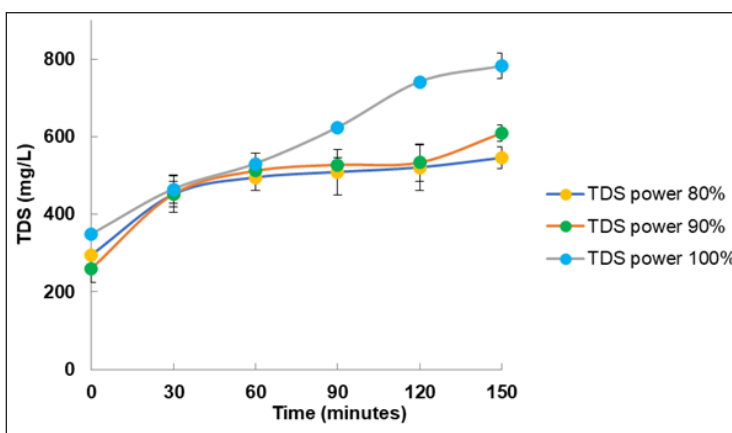


Figure 4. Total dissolved solids of NADES-CAS during the MAHD process

This finding aligns with Várady et al. (2022), who reported that microwave-induced heating enhanced water evaporation, thus increasing solute concentration in extraction systems. Similarly, Igwegbe and Onukwuli (2019) found that higher extraction intensity resulted in higher TDS values due to increased dissolution and release of solid materials into the solvent. Thus, the observed trend was consistent with established mechanisms of microwave-assisted extraction. Overall, the increasing TDS value with higher microwave power confirms that microwave heating promoted both water evaporation and the release of solid compounds into NADES. Therefore, TDS serves as a reliable indicator of the concentration of extracted materials and reflects the efficiency of the MAHD–NADES extraction process.

Electrical Conductivity (EC) of the NADES-CAS

The electrical conductivity (EC) of NADES-CAS increased progressively during the extraction process across all microwave power levels (Figure 5). Additionally, the EC of NADES-CAS increased progressively with increasing power levels.

The increase in EC showed the enhancement of ionic mobility when the internal temperature of the NADES increased under microwave heating. Higher microwave power resulted in faster temperature elevation, which reduced the intermolecular forces in the solvent, promoted ion movement, and ultimately increased conductivity. This heightened ion mobility also improved the ability of NADES-CAS to dissolve polar phenolic compounds from cinnamon, since polar molecules interact more efficiently with highly conductive ionic media. These findings are in line with those of Liu et al. (2018), who reported that temperature was a major determinant of ionic conductivity in liquid systems. Additionally, Fu et al. (2018) demonstrated that an increase in temperature enhanced ion mobility and increased EC of ionic liquid–based solvents, which was consistent with the trend observed

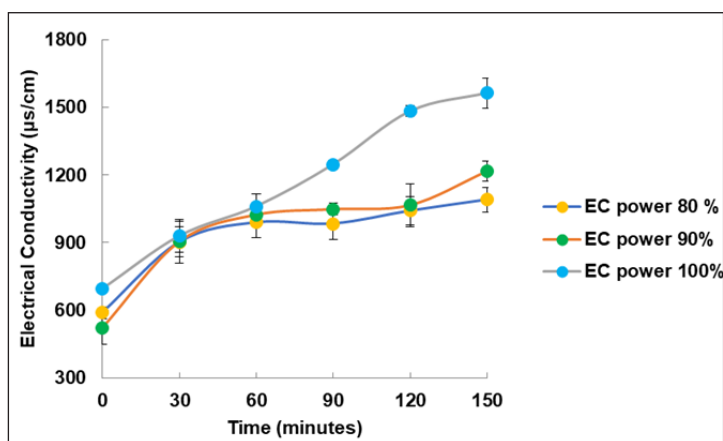


Figure 5. Electrical conductivity of NADES-CAS during the MAHD process

in this study. The increase in EC with increasing microwave power confirms that thermal stimulation significantly enhanced ionic activity in NADES-CAS. This improvement supported more efficient solubilisation of polar phenolics during extraction, indicating that higher microwave power contributed to better extraction performance through increased ionic conductivity.

Solvent Temperature Response at Each Microwave Power

Ionic migration occurs when the electric field generated by microwaves triggers the movement of ions through the process of electrophoresis, where ions move due to changes in the electric field. Heat is generated through friction between ion molecules due to the solvent's resistance to ion migration. Meanwhile, dipole rotation occurs when a continuously fluctuating electric field rotates the dipole molecules in the solvent. In the distillation process using MAHD, the solvent temperature increased along with ion migration activity and dipole rotation, where the rate of temperature increase depends on the dielectric constant and the solvent's response to microwaves. This study used different power variations, starting from 80%, 90% and 100%. The resulting data showed that the power of 80% achieved a temperature of 100 °C in 14-15 minutes of heating using MAHD. Meanwhile, the power of 90% needed 11-12 minutes, and the power of 100% needed the fastest time, namely 9 minutes, to achieve a temperature of 100 °C.

Profile of Liquid Extract under MAHD

Electrical Conductivity (EC) of the Extract under MAHD

One of the physical property parameters of the cinnamon extract was EC. The EC value can be seen in Figure 6. The EC value depends on the number of dissolved ions as well as

the type and concentration of ions in the solution. Electrical conductivity (EC) of cinnamon extract is often evaluated to understand its properties in microemulsions, where it interacts with other compounds like tea polyphenols and gallic acid (T. Wang et al. 2023).

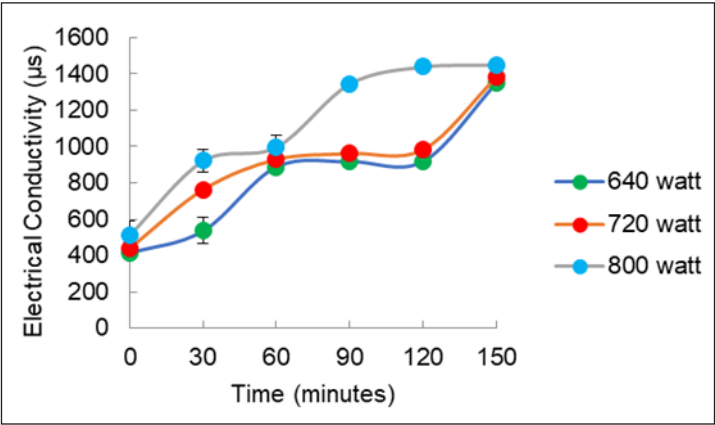


Figure 6. Electrical conductivity (EC) of the extract under MAHD

An increase in EC values with extraction time can be observed in Figure 6, while MAHD at 800 W showed higher EC values compared to the other power levels. This proves that the EC value increased with increasing time and power. In addition, MAHD increased the temperature and pressure on the sample, thereby increasing the number of ions in the solution. The success of an extraction can be seen from the EC value, which was not too high or too low. The EC value obtained in this research was 1450 µs at 800 W, 1383 µs at 720 W, and 1349 µs at 640 W.

Total Dissolved Solids (TDS) of the Extract under MAHD

Besides EC, another physical property parameter of liquid cinnamon extract is the TDS value. The results of TDS values are presented in Figure 7. The high TDS value in the cinnamon extract can identify the presence of compounds such as polyphenols, cinnamaldehyde, and antioxidants. The TDS value of cinnamon extract represents the amount of dissolved substances, such as active and volatile compounds. The value of TDS shows the combination of dissolved organic and inorganic substances as ionised molecules (Rahayu et al. 2019). Based on Figure 7, there was an insignificant increase in the TDS value. TDS value increased with the length of the extraction time. The TDS value obtained from this research was 725 ppm at 800 W, 691 ppm at 720 W, and 674 ppm at power at 640 W. The MAHD at a power of 800 W obtained the highest TDS value because it can increase the temperature and pressure on the sample, accelerating the release of compounds dissolved in the material. The success of an extraction can be seen from the TDS value,

the enhanced release of organic and inorganic constituents from the material resulted in higher TDS values (Izza et al., 2020).

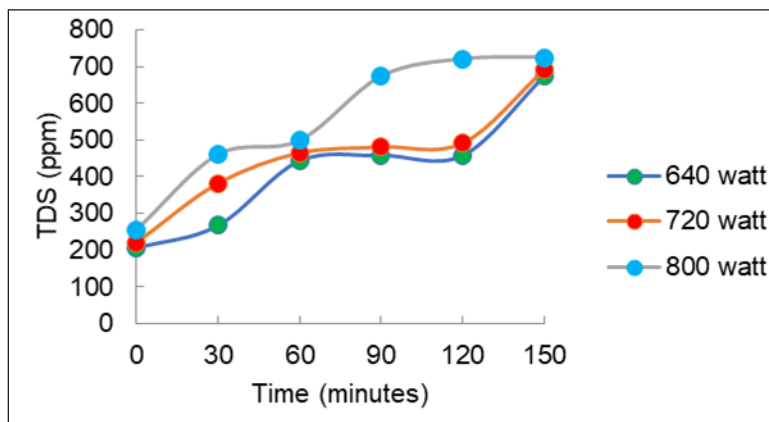


Figure 7. Total dissolved solids (TDS) of the extract under MAHD

Potential of Hydrogen (pH) of the Extract under MAHD

Based on Figure 8, the pH of the extract decreased over time and became more acidic as both the extraction time and microwave power increased. Specifically, the extract exposed to 800 W showed a lower pH value compared to those exposed at 640 W and 720 W. pH measurements were conducted every 30 minutes over 150 minutes at a constant temperature of 100 °C under power levels of 80%, 90%, and 100%. The decrease in pH under higher microwave power can be attributed to enhanced thermal dissociation within the NADES. When the temperature increased, the hydrogen-bonding equilibrium between the hydrogen bond acceptor and donor (HBD, citric acid) shifted, promoting the release of more protons (H^+) into the solution. This increased proton availability resulted in a more acidic (lower pH) environment (Karadendrou et al. 2022). The strength and concentration of hydrogen bonds in the NADES strongly influenced this behavior: a higher HBD content (citric acid) contributed to greater acidity when heated, due to stronger hydrogen-bond interactions and greater dissociation.

These findings are supported by previous studies. Skulcova et al. (2018) stated that the acidity of deep eutectic solvent was significantly influenced by the nature of the HBD, and that pH frequently decreased with temperature increase due to enhanced dissociation of the acid component. A comprehensive review also confirmed that temperature and the chemical identity of the HBD strongly affected the pH behaviour of NADES (Jančíková et al., 2022). This result observed that increasing microwave power and time led to a more acidic NADES-CAS (lower pH), which was consistent with the known physicochemical behaviour of acid-based eutectic systems. This pH behaviour thereby contributed to the

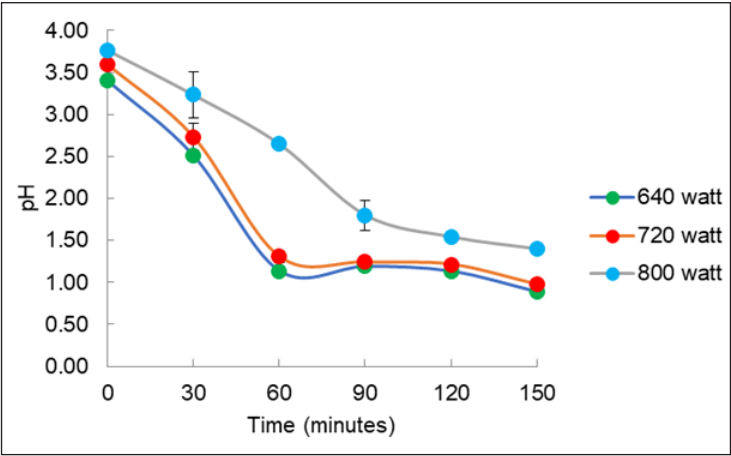


Figure 8. Potential of Hydrogen (pH) of extract under MAHD

enhanced efficiency of phenolic extraction under high-power, high-temperature MAHD conditions.

Total Phenolic Content of the Extract during MAHD

The profile of changes in the amount of Total Phenolic Compound (TPC) extracted in the solvent during the Microwave-Assisted Hydrodistillation (MAHD) process is presented in Figure 9. The TPC value increased with extraction time at all MAHD power levels. At 80% power, TPC increased from approximately 0.30 to 0.40 mg/mL. At higher power levels (90% and 100%), TPC values increased from about 0.45 to 0.65 mg/mL with extraction time. When comparing the power level 90% and 100% at the same extraction time, the difference in TPC values was relatively small, generally within the range of 0.01–0.05 mg/mL The TPC value at a power of 80% was lower than that at powers of 90% and 100% because the energy applied to the cinnamon extract was insufficient to optimally break down the material matrix to limit the release of phenolic compounds. In contrast, higher power levels (90% and 100%) provided greater thermal and microwave-induced molecular movement, enhancing cell wall breakdown and facilitating phenolic diffusion into the solvent. The minimal difference between powers of 90% and 100% indicated that the extraction approached saturation at a power of 90%, where further increases in power no longer produce significant gains. These observations are consistent with prior studies. For example, a microwave extraction study on *Moringa oleifera* leaves (Kheyar et al., 2025), broccoli (Jokićet al., 2025), and *Anoectochilus roxburghii* (Xu et al., 2017) demonstrated that increasing microwave power significantly increased TPC yield. Overall, the TPC extraction profile demonstrated that a microwave power of 90% was sufficient to achieve near-maximum TPC.

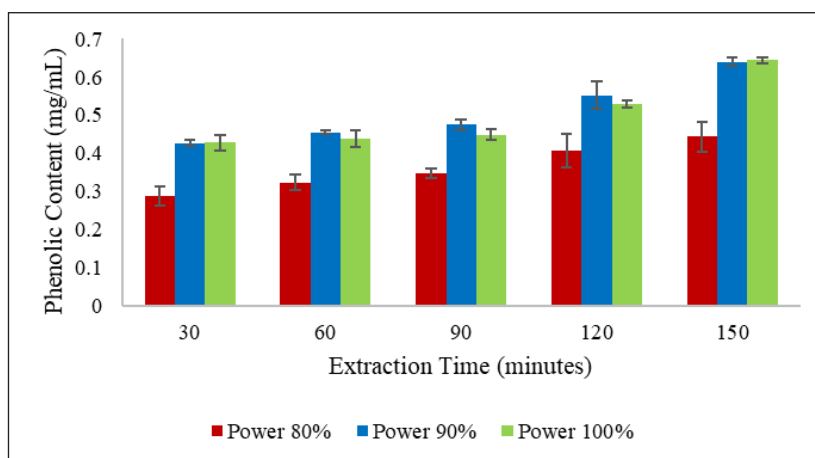


Figure 9. Total phenolic content of the extract during MAHD

The Total Proanthocyanidins Content of Liquid Extract during MAHD

Each solvent has a different dielectric constant, which significantly affects the performance of the extraction process. In this case, the concentration of citric acid and sucrose in distilled water (aquadest) plays an important role in increasing the effectiveness of compound extraction. Citric acid is known to be hygroscopic, allowing it to absorb moisture from its surroundings and thereby maintain the stability of the system. In addition, this compound exhibits good stability against light and air exposure, making it less prone to degradation over time (Luque-Uria et al., 2025). The increase in extraction efficiency is influenced not only by the thermal effects of the solvent but also by the solvent's ability to disrupt and release compounds bound within the material matrix. An effective solvent can enhance the diffusion rate of bioactive constituents, thereby promoting a more efficient release of matrix-bound compounds (Song et al. 2020).

The proanthocyanidin is included in phenolic compounds with many active compounds and was measured using a UV-Vis spectrophotometer. Figure 10 shows that the Total Proanthocyanidin Content (PA) value increased during the Microwave-Assisted Hydrodistillation (MAHD) process, ranging from 0.14 to 0.27 mg/mL. The higher PA results indicated a higher concentration of extracted proanthocyanidin compounds. At a power of 800 W (100%), PA reached a higher value (0.32–0.41 mg/mL), compared to that at powers of 80% and 90%, resulting PA of 0.15–0.26 mg/mL. The PA value of 0.41 mg/mL was achieved at a temperature of 100 °C, a power of 800 W, and an extraction time of 150 minutes. The increase in PA value at high power was due to higher extraction energy, optimal temperature, and better mass transfer efficiency (Izza et al., 2023; Susanti et al., 2020; Susanti et al., 2021). However, power and extraction time must be optimised to ensure energy efficiency and prevent degradation of active compounds.

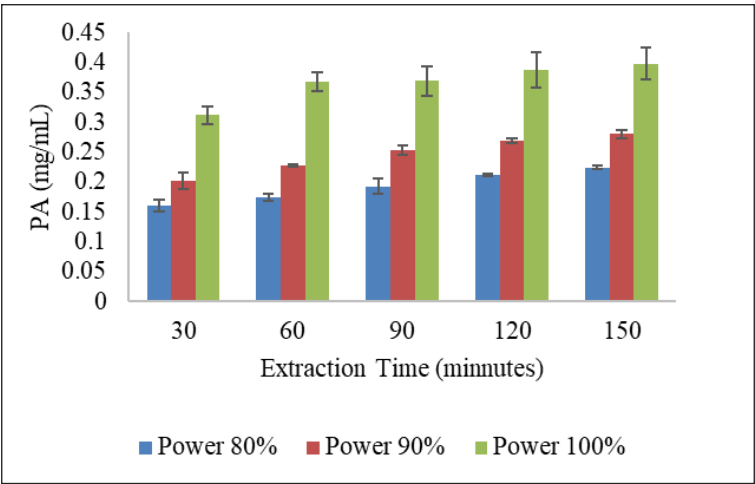


Figure 10. Total proanthocyanidins content of the extract during MAHD

The Application of the Peleg Model in the Extraction

Application of the Peleg Model in the Profile of Total Phenolic Content

The Peleg model interprets the change of TPC during extraction and determines the initial extraction rate constant (B_0) and maximum extraction capacity value (C_e). Table 1 shows that the extraction rate constant (B_0) increased as microwave power rose from 640 W to 800 W. The highest B_0 value was obtained at 800 W (0.03969 mg/mL·min), while the lowest value was obtained at 640 W (0.02642 mg/mL·min). A similar pattern was observed in the equilibrium concentration (C_e), where C_e increased with power, although the difference between 720 W and 800 W was relatively small (0.06785 to 0.066072 mg/mL). Figure 11 further confirms that the Peleg model predictions closely followed the experimental data under all power levels.

The increase in B_0 reflects the faster initial extraction rate when greater microwave power is applied. Higher power enhances internal heating through rapid dipole rotation and ionic conduction, resulting in cell disruption and accelerated release of polyphenols. However, the limited increase from 720 W to 800 W suggests the system approaches thermal saturation, where additional energy no longer significantly accelerates diffusion. The C_e behaviour indicates that 720 W is already sufficient to reach near-maximum extraction capacity. At the lowest power (640 W), both B_0 and C_e were markedly reduced, implying insufficient heat to overcome mass-transfer resistance and fully disrupt the cinnamon matrix.

These observations are in line with previous studies showing that the microwave transmitted to the solvent caused an increasing temperature, causing an increase in the extraction rate value (Sonar & Rathod 2020). The ability of the Peleg model to accurately describe extraction kinetics has also been reported for phenolic extractions from plant

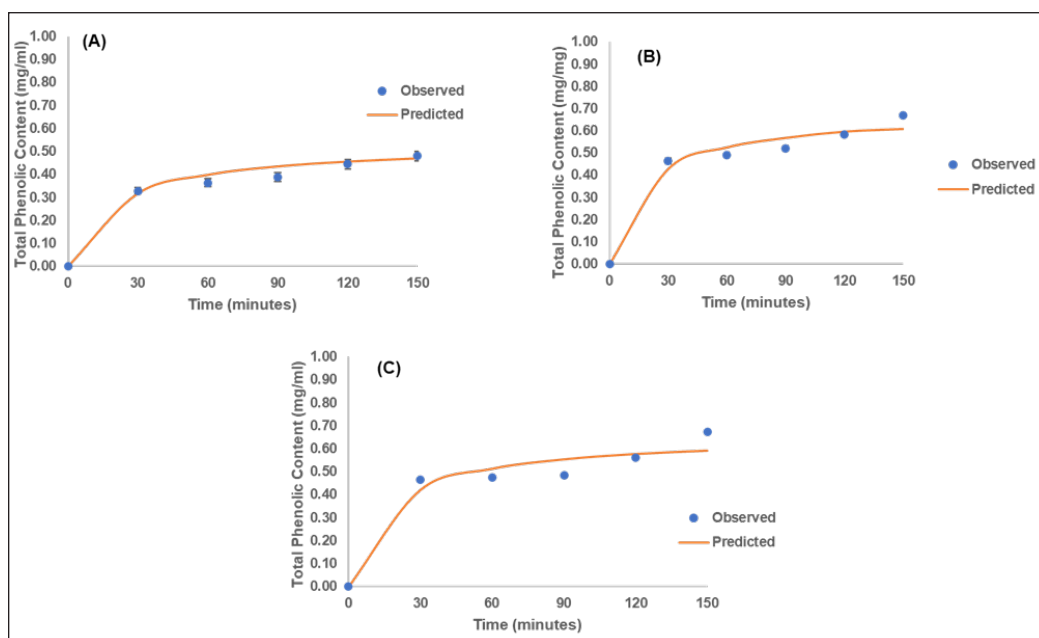


Figure 11. Observed and predicted total phenolic content during MAHD at (A) 640, (B) 720, (C) 800 W

Table 1

Peleg model parameters and validation statistics for the extraction of total phenolic content (TPC) from cinnamon at different microwave-assisted hydrodistillation (MAHD) power levels

The Peleg Model Parameters				Validation Parameters		
Power	B_0	C_e	SSE	RMSE	X^2	R^2
(W)	(mg/mL.min)	(mg/mL)				
640	0.0264	0.5349	0.0079	0.0481	0.0031	0.9810
720	0.0389	0.6785	0.0096	0.0561	0.0038	0.9655
800	0.0397	0.6607	0.0155	0.0718	0.0061	0.9422

matrices, where B_0 correlates with the initial diffusion rate and C_e represents the maximum achievable concentration under given conditions (Pamungkas et al., 2025; Susanti et al., 2025). Overall, the close agreement between predicted and experimental data indicates that the Peleg model is a reliable tool for characterising and forecasting the extraction of TPC from cinnamon behaviour under MAHD conditions.

Application of the Peleg Model in the Profile of Proanthocyanidin Content

Proanthocyanidin (PA) was one of the main target compounds in cinnamon extract due to its strong antioxidant activity (Paquet-Durand et al. 2015). The extraction performance was quantitatively evaluated using the Peleg kinetic model, which provided the extraction rate

constant (B_0) and the maximum extraction capacity (C_e). As shown in Table 2, the highest B_0 value (0.11171 mg/mL·min) was obtained at 800 W, while the lowest B_0 (0.03878 mg/mL·min) was obtained at 640 W. This trend indicates that higher microwave power significantly accelerated the release of PA into the NADES-CAS solvent. As discussed previously, the Peleg model also performed best in predicting the profile of proanthocyanidin during the MAHD. Prediction in each power of MAHD, as shown in Figure 12, illustrated the match value between the predicted proanthocyanidin concentration using the Peleg model and the observed data. It was also supported by the values of several validation parameters, such as SSE, RMSE, X^2 , and R^2 , presented in Table 2.

Table 2
Peleg model parameters and validation statistics for the extraction of proanthocyanidin (PA) from cinnamon at different microwave-assisted hydrodistillation (MAHD) power levels

The Parameters of the Peleg Model			The Validation Parameters			
Power	B_0	C_e	SSE	RMSE	X^2	R^2
(W)	(mg/ mL.min)	(mg/mL)				
640	0.0388	0.5529	0.0024	0.0263	0.0009	0.9939
720	0.0425	0.7300	0.0023	0.0276	0.0009	0.9932
800	0.1117	0.9439	0.0018	0.0214	0.0007	0.9989

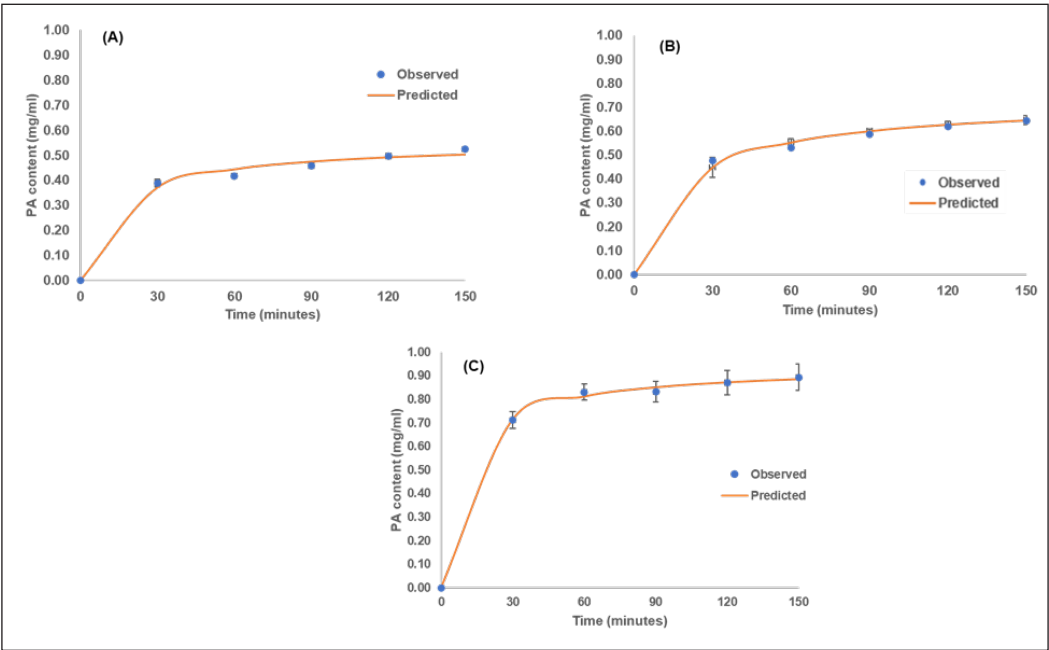


Figure 12. Observed and predicted Proanthocyanidin content during MAHD at (A) 640, (B) 720, (C) 800 W

The increase in B_0 with greater microwave power reflects the enhanced mass-transfer rate during extraction. At 800 W, the rapid internal heating of the solvent and sample matrix allowed the system to reach 100 °C more quickly than at 720 W or 640 W. The elevated temperature enhanced cell wall rupture, reduced solvent viscosity, and accelerated diffusion, enabling more efficient solubilization of PA compounds. Consequently, high power improved both the initial extraction rate and the approach toward the maximum PA concentration. The high extraction temperature increased the diffusion and weakened the plant cell wall and membrane integrity, thereby enhancing solvent penetration and facilitating the release of proanthocyanidins into the extract. Several studies noted that the rise in proanthocyanidin yield at elevated temperatures was associated with greater diffusion rates and the thermal or physical disruption of cell wall structures (Zhang et al., 2018; Thilakarathna et al., 2022; Shi et al., 2022).

The maximum extraction capacity constant (C_e) value was proportional to the initial extraction rate (B_0). At a power of 800 W, the extraction process produced the highest C_e value of 0.9439 mg/mL. It indicates the optimal maximum capacity for releasing proanthocyanidins from cinnamon. The presence of NADES supported the stability of the PA compound at high temperatures, likely due to strong hydrogen-bond interactions between NADES components and the proanthocyanidin molecules, and protected against thermal degradation (Li, 2022; Hikmawati et al., 2021; Silva et al., 2024). At a lower power of 640 W, the extraction resulted in the lowest B_0 value of 0.03878 mg/mL.min, and the lowest C_e value of 0.5529 mg/mL.

The Essential Oil from MAHD

This research assessed the combination of NADES-CAS and microwave for its ability to extract the functional compounds and essential oils from cinnamon. As discussed, the combination proved successful in extracting phenolics, including proanthocyanidins, which are antioxidant compounds in cinnamon. The phenolic compound was released in the bulk of the solvent and remained in the extraction flask. At the same time, the essential oils evaporated with water vapour after they were released from the internal part of the cinnamon. The heat generated from the interaction of the microwave with cinnamon and solvent material supported this evaporation. The evaporation was also supported by the NADES, which created an abrasion and enlarged the pore to support the evaporation of essential oil from the internal part of the cinnamon powder. The essential oil was separated from the water vapour through a distillation process using a cold condenser that settled on the top of the microwave system. The maximum yield of essential oil was achieved at a microwave power of 800 W, which resulted in 0.4% of cinnamon powder. Meanwhile, microwave powers of 720 and 640 W only resulted in 0.22% and 0.18% of cinnamon powder, respectively.

The essential oil extracted from cinnamon bark (*Cinnamomum* spp.) powder has a distinctive aroma due to its several essential oil compounds. The main components are dominated by aromatic aldehyde, terpenoid, and phenylpropane compounds, which play an important role in biological activity and their application in various industries. The essential oil resulting from the MAHD was evaluated to identify the active constituents in the essential oil using the gas chromatography-mass spectrometry (GC-MS) method. The results of GC-MS analysis allow the identification of components based on compound name, molecular weight, and chemical structure, providing detailed information on the chemical profile of cinnamon bark essential oil (Chokkavarapu & Mandla 2019).

Table 3 describes that the essential oil from MAHD of cinnamon essential powder had several main compounds, concluding Cinnamaldehyde (74.982%), Caryophyllene (0.982%), Benzaldehyde (0.438%), and Pinene (0.12%). Cinnamaldehyde is the main component of cinnamon bark, giving a distinctive sweet and spicy aroma. It has antimicrobial and anti-inflammatory activities. The molecular formula in Cinnamaldehyde is C_9H_8O , which consists of 9 carbon atoms, 8 hydrogen atoms, and 1 oxygen atom. The compound contains a benzene (phenyl) ring connected to a carbon-aldehyde chain (CHO) (Friedman, 2017; Guo et al., 20204; Kim et al., 2020). Cinnamaldehyde was the dominant component in the sample, with a total contribution of 74.982%. The presence of two peaks at different retention time (RT) indicates the presence of trans-cinnamaldehyde and cis-cinnamaldehyde isomers. The presence of two peaks can also be caused by heating on the GC-MS device, which can convert some of the trans-cinnamaldehyde to cis-cinnamaldehyde. Cinnamaldehyde was the major compound of the essential oil from cinnamon obtained using MAHD with NADES-CAS solvent. Meanwhile, the others were detected as Caryophyllene, Benzaldehyde, and Pinene.

Table 3
The major volatile compound in the essential oil from cinnamon

No	Retention Time (min)	Name of Compound	Peak Area %
1	12.825; 15.704	Cinnamaldehyde	74.982
2	21.841; 20.606; 22.647	Caryophyllene	0.982
3	7.24; 10.112	Benzaldehyde	0.438
4	7.165; 8.526	Pinene	0.12

CONCLUSION

The combination of Microwave-Assisted Hydrodistillation (MAHD) and the Natural Deep Eutectic Solvent Citric Acid–Sucrose (NADES-CAS) was successfully applied for the simultaneous extraction of phenolic compounds, proanthocyanidins, and essential oils from cinnamon powder. The NADES-CAS (1:1 molar ratio, 1% concentration) demonstrated distinct physicochemical changes under increasing microwave power

(640–800 W), including increased EC, TDS, and temperature, and decreased density, viscosity, and pH. Application of the Peleg model showed high accuracy in predicting the extraction kinetics, with extraction rates (B_0) in the range of 0.0264–0.0397 mg/mL·min for phenolics and 0.0388–0.1117 mg/mL·min for proanthocyanidins, and maximum capacities (C_e) in the range of 0.5349–0.6607 mg/mL for phenolics and 0.5529–0.9439 mg/mL for proanthocyanidins, respectively. Higher microwave power increased both B_0 and C_e values, confirming the acceleration effect on mass transfer during extraction. The MAHD–NADES system also produced cinnamon essential oil with a yield of 0.18–0.40%, dominated by cinnamaldehyde, along with caryophyllene, benzaldehyde, and pinene.

Practically, these findings demonstrated that combining MAHD with a green solvent can shorten extraction time, enhance mass transfer efficiency, and simultaneously recover high-value bioactive compounds and essential oils. This approach offers a scalable, energy-efficient alternative for the extraction industry, particularly for phytochemical and functional ingredient production, enabling faster processing with reduced solvent use and improved yield consistency.

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