INSTANT CONTROLLED PRESSURE DROP ASSISTED EXTRACTION OF ESSENTIAL OIL FROM CINNAMON LEAVES

By

AMEERA C (2020-02-006)

NAVYA PRAVEEN K (2020-02-021)

ALEX K SANI (2020-02-026)

AKASH BALAKRISHNAN (2020-02-041)

DEPARTMENT OF PROCESSING AND FOOD ENGINEERING
KELAPPAJI COLLEGE OF AGRICULTURAL ENGINEERING AND TECHNOLOGY
TAVANUR-679573, MALAPPURAM
KERALA, INDIA
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PROJECT REPORT

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KELAPPAJI COLLEGE OF AGRICULTURAL ENGINEERING AND TECHNOLOGY

TAVANUR –679573, MALAPPURAM

KERALA, INDIA

2024

DECLARATION

We hereby declare that this project report entitled "**INSTANT CONTROLLED PRESSURE DROP ASSISTED EXTRACTION OF ESSENTIAL OIL FROM CINNAMON LEAVES**" is a bonafide record of project work done by us during the course of project and that the report has not previously formed the basis for the award of any degree, diploma, associateship, fellowship or other similar title of any other University or Society.

AMEERA C	(2020-02-006)
NAVYA PRAVEEN K	(2020-02-021)
ALEX K SANI	(2020-02-026)
AKASH BALAKRISHNAN	(2020-02-041)

Place : Tavanur

Date : 31-05-2024

CERTIFICATE

Certified that this project report entitled "**INSTANT CONTROLLED PRESSURE DROP ASSISTED EXTRACTION OF ESSENTIAL OIL FROM CINNAMON LEAVES**" is a bonafide record of project work done jointly by Ms. Ameera C, Ms. Navya Praveen K, Mr. Alex K Sani and Mr. Akash Balakrishnan under our guidance and supervision and that it has not previously formed the basis for the award of any degree, diploma, associateship, fellowship or other similar title, of any other University or Society.

Project Guide:

Dr. Prince M V

Professor and Head,

Department of Processing and Food Engineering

KCAET, Tavanur

Co-Guide:

Dr. Ashitha. G N

Assistant Professor (C).

Department of Processing and Food Engineering

KCAET, Tavanur

Place : Tavanur

Date :

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DEDICATED TO TEACHERS

AND FRIENDS

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SYMBOLS AND ABBREVIATIONS

%	Percentage
℃	Centigrade
&	And
/	Per
=	Equal to
Δ	Delta
Γ	Gamma
α	Alpha
AD	Alzheimer`s disease
AGRI TECH	Agricultural Technology
ANOVA	Analysis of variance
ASE	Assisted Extraction
ASTA	American Society of Travel Advisors
β	Beta
C.	Cinnamomum
cm	Centimeter
deg	Degree of arc
DIC	Instant Controlled Pressure Drop
EAE	Enzyme Assisted Extraction
et al.	And others
Fig.	Figure

g	Gram
g/g	Gram/gram
g/ml	Gram per milliliter
GSH	Glutathione
h	Hour
HaCaT	Human non cancer keratinocytes
HD	Hydro-distillation
HP	Horse power
H_2S	Hydrogen sulphide
i.e	That is
IL	Interleukin
KCa	Kilo calorie
KCAET	Kelappaji College of Agricultural Engineering and Technology
Kg	Kilo gram
Kg/cm^2	Kilogram per centimeter square
KPa	Kilo pascal
KW	Kilo watt
m	Meter
m^2	Meter square
mm	Millimeter
MAE	Microwave Assisted Extraction

M.C	Moisture Content
MFA	Multi Flash Autovaporisation
MPa	Mega Pascal
min	Minute
mg/g	Milligram per gram
ml	Milli liter
NO	Nitric Oxide
N	Newton
PCA	Principle Component Analysis
Ph	Potential of hydrogen
PLE	Pressurized Liquid Extraction
RMSE	Root Mean Square Error
RS	Resistant starch
RI	Refractive Index
s	Second
Syn	Synonym
SFE	Supercritical Fluid Extraction
TNAU	Tamil Nadu Agricultural University
TNF	Tumor Necrosis Factor
TPC	Total Phenolic Content
UAE	Ultrasound Assisted Extraction
USA	United States of America

INTRODUCTION

CHAPTER I

INTRODUCTION

Essential Oils (EOs) are complex aromatic liquids found in various plant parts like flowers, herbs, leaves, fruits, bark, seeds, and roots, synthesized as secondary metabolites. They are hydrophobic, lipophilic, soluble in organic solvents, and immiscible with water, making them valuable and rare substances with low extraction yields. EOs exhibit biocidal activities like bactericidal, virucidal, and fungicidal properties, making them useful in medical applications and as food preservatives. With over 100 different terpenic compounds in their chemical composition, EOs have broad antimicrobial activities against bacteria, fungi, molds, viruses, pests, and insects. In pharmaceutical and food industries, EOs are included in various dosage forms and food products for their medicinal properties, preservation capabilities, and innovative applications in food packaging to combat pathogens (Asbahani *et al.*, 2015).

Extraction is an important step for the separation, identification, and use of valuable compounds from different plants. The choice of an acceptable technique to obtain maximum yield and highest purity varies according to the nature of the target compound. Chemical and mechanical processes like solvent extraction and steam distillation are used for the extraction of compounds from plants (Danlami *et al.*, 2014). The major goal of all oil extraction processes is to achieve high yields without compromising the inherent quality of oil. Numerous technologies have been developed for oil extraction which are classified into traditional, conventional (dry/wet rendering, mechanical pressing, solvent or chemical extraction), and advanced techniques enzyme-assisted extraction (EAE), microwave-assisted extraction (MAE), ultrasound-assisted extraction (UAE), supercritical fluid extraction (SFE). Presently, both conventional and advanced/modern techniques are employed for oil extraction (Rani *et al.*, 2021).

Traditionally essential oil extraction has been achieved via hydro-distillation. This technique is based on the principle that, at boiling temperature, the combined vapor pressures equal the atmospheric pressure. The charged steam rises and encounters a narrow tube cooled by an outside source (cold water or antifreeze). Afterward, the steam is condensed and collected in a vessel; hence, since the essential oil is less dense, it moves at the top while the water goes down. Some advantages of this procedure are the low cost of the equipment and its simplicity. On the other hand, it also has disadvantages, such as it can consume a lot of energy and the processing time is often long, which could trigger negative chemical changes on the EO. Regarding energy consumption, in the study conducted by Allaf *et al.* 2013 on orange peels, the energy balances of HD vs. the instant controlled pressure drop technology (DIC) point out that above a certain amount of extraction efficiency, the energy consumption significantly decreases by using the DIC technology (Teresa-Martínez *et al.*,2022).

The techniques such as microwave-assisted extraction, ultrasound-assisted extraction, pressurized liquid extraction, and supercritical fluid extraction developed for extraction of valuable components from plants and seed materials have been successfully used to effectively reduce the major short-comings of the traditional method such as soxhlet extraction. These include shorter extraction time, increase in yield of extracted components, decrease in solvent consumption, and improvement of the quality of extracts (Danlami *et al.*, 2014).

Instant Controlled Pressure Drop (DIC) is an innovative thermomechanical process utilized in various applications, including microbial decontamination, extraction of volatile active molecules, and enhancement of drying processes. The DIC technology involves subjecting a material to high-pressure saturated steam of 100 to 900 kPa for a few seconds followed by an abrupt pressure drop towards a vacuum (5 kPa). This sudden pressure drop leads to the instantaneous auto-vaporization of water and other volatile compounds, causing rapid cooling that prevents thermal degradation.

DIC is particularly effective in expanding and texturing plant materials, which improves solvent diffusion and enhances extraction efficiency. This process is highly valued in food processing, pharmaceuticals, and essential oil extraction for its ability to maintain the quality and efficacy of bioactive compounds while ensuring energy efficiency and ecological benefits (Nader *et al.*, 2022).

Cinnamon belongs to the Lauraceae family. The genus Cinnamomum comprises approximately 250 species which are widely distributed in China, India and Australia Cinnamon is considered a remedy for various disease conditions including respiratory, gastro-intestinal, endocrinal and gynecological ailments. Almost every part of the cinnamon tree, specially the bark and leaves have some medicinal or culinary use. The essential oils obtained from barks and leaves, vary significantly in chemical composition, which suggests that they might vary in their pharmacological effects as well. (Wijesinghe *et al.*, 2021). The leaves of C.verum are rich sources of various chemical volatiles, including Cinnamaldehyde. Further, these molecules are found to have strong insecticidal, larvicidal, and antimicrobial properties. It is also noteworthy that these essential oils have no toxic effects observed and are also found to be safe in germinating grains. Hence, it is possible that the C. verum essential oils may evolve as a promising green pesticide and antimicrobial agent in the near future (Narayanan kutty *et al.*, 2021). Phenolic compound eugenol belongs to the group of phenylpropanes is the major active component of C. verum leaf EO which is responsible for its bioactivity. Eugenol can interfere with cell membrane integrity and nonspecific permeability by altering the membrane fatty acids and disrupt the plasma membrane of bacterial cells. Further, eugenol can inhibit bacterial enzymes of some bacterial species such as *Escherichia coli* and *Bacillus subtilis* (Wijesinghe *et al.*, 2020). The antifungal and antioxidant properties of cinnamon leaf essential oil are due to volatile components such as eugenol and cinnamaldehyde. These volatile phenolic compounds are able to damage the fungal cells (Melgarejo-Flores *et al.*, 2013).

It could be noted from the above statements that application of an Instantaneous Pressure drop to the cinnamon leaves could result in an increased structure swelling and higher diffusivity constant. This coupled with subsequent hydro-distillation could provide efficient extraction of high quality essential oil in less time and energy. In such a system each plant material behaves differently to the pressure drops. Therefore, the process parameters leading to efficient extraction of quality essential oil needs to be optimized.

To the best of our knowledge, there are no previous studies regarding DIC technology as a texturing pretreatment of cinnamon leaf before hydro-distillation (HD). Then, this comparative study explores the impacts on the essential oil yield obtained by only HD and by coupling DIC to HD.

Taking the above facts into consideration, this research entitled “Instant Controlled Pressure Drop assisted extraction of Cinnamon Leaf Essential Oil” was undertaken with the following objectives:

1. To optimize the process parameters of the instant controlled pressure, drop assisted extraction of essential oil from cinnamon leaf

2. To study the physico-chemical characteristics of the extracted essential oil

REVIEW OF LITERATURE

CHAPTER II

REVIEW OF LITERATURE

This chapter deals with the review of research work reported on the scenario of cinnamon and benefits of cinnamon. Reviews on application of instant pressure drop assisted in extraction of essential oil and d has been elaborately presented.

2.1 CINNAMON

Cinnamon is one of the oldest known spices and has been used in cooking and traditional herbal medicine for millennia. The botanical name *Cinnamomum* derives from the Hebraic and Arabic term *amomon*, meaning fragrant spice plant. Cinnamon was one of the first spices to reach the Mediterranean (Spence, 2024). Cinnamon belongs to the Lauraceae family. The genus Cinnamomum comprises approximately 250 species which are widely distributed in China, India and Australia (Wang *et al.*, 2009). Cinnamon is the name given to the bark of the thin lateral shoots taken from the foot of several tropical evergreen trees of the genus *Cinnamomum*. Trees tend to be harvested every two to three years. the cinnamon tree typically grows to a height of 7–10 m (though there are reports of trees as tall as 17 m) (Spence, 2024). Cinnamomum cassia is also named Chinese Cinnamon, which has been found both wildly and cultivated in Southeast Asia since ancient age, then introduced into Indonesia, South America and Hawaii. Cinnamomum zeylanicum originated from the island Sri Lanka and India. Cinnamomum tamala originates from the south slopes of Himalayas. Cinnamomum burmannii, also known as Indonesian Cinnamon, is normally found in West Sumatra. Cinnamomum pauciflorum is occurring in southwest China, north-eastern India, Assam, and Khassia hills. Among, the major species with high economic values are C. cassia and C. burmannii. These five species have been used in the traditional medicines of India and China (Wang *et al.*, 2009). It is currently also one of the world's most commonly used spices (Spence, 2024).

2.1.1 Area and production

Cinnamon is an evergreen perennial plant widely grown across Asia as a spice and for its essential oils, with about 80% of the world's commercial cinnamon coming from Sri Lanka (Jayawardena and Smith, 2010). Cinnamon is indigenous to Sri Lanka, which contributes around 90% of the global trade of true cinnamon. Given its wide range of industrial applications, including food and beverage, liqueur, perfumery, nutraceutical, cosmeceutical and oral care industries, and with the advent of green economy, there is growing global demand for Ceylon cinnamon. Because of its exquisite organoleptic properties combined with insignificant content of coumarin, a carcinogen, in its bark, Ceylon cinnamon, if strategically and vigorously marketed, could command a clear competitive edge in the global market over its competitor, cassia, which is of inferior quality and carries appreciable amounts of coumarin. However, its low productivity, low export volume, high cost of production, dearth of peelers and limited value addition are key constraints to enhancing the cinnamon industry in Sri Lanka (Samaraweera *et al.*, 2020).

There is more production of cassia which is produced in South East Asia, primarily in countries, such as Indonesia, China, and Vietnam. Cinnamon produced in India is also of the cassia variety and is stronger and more intense in flavor. Cinnamon produced in Sri Lanka is of highest quality as it releases a more delicate aroma and is priced much higher than cassia cinnamon. Mexico and United States are leading importers of true cinnamon produced in Sri Lanka. Cinnamon is a favorite household for Asian family and also has become popular in western culturesin the last one century. Key producers are Indonesia, China, Vietnam, and Sri Lanka and key importers are Mexico, USA, India, and Bangladesh. In fact, such was the trade volumes of cinnamon and at one point of time it was traded as currency for exchanging goods at maritime ports of Indo Pacific region (Singh and Singh, 2018).

The greater part of the spice sold as cinnamon in the USA and Canada (where true cinnamon is still generally unknown) is actually cassia. In some cases, cassia is labelled ‘Chinese cinnamon’ to distinguish it from the more expensive true cinnamon, which is the favourite form used in Mexico and Europe. ‘Indonesian cinnamon’ can also refer to Cinnamomum *burmanii*, which is also commonly sold in the USA, simply labelled as cinnamon (Lungarini *et al.*, 2008).

2.1.2. Varieties of cinnamon

Cinnamon corresponds to a wide variety of plants, belonging to the Cinnamomum species; among these species of the Lauraceae family, four are highlighted in the food, pharmacological, and cosmetic industries, including Cinnamomum cassia (sin. C. aromaticum) known as Cassia or cinnamon Chinese, this being the most common, C. burmannii, C. loureiroi, and C. zeylanicum (syn. Cinnamomum Verum). Table 2.1 presents the information about the main species and their respective commercial names.

Table 2.1 Main species and commercial name of Cinnamomum

Scientific name	Commercial name
C.zeylanicum sin. C. verum	Ceylon cinnamon True cinnamon Mexican cinnamon
C.burmannii	Indonesian cinnamon Korintje cinnamon Padang cassia
C.loureiroi	Saigon cinnamon Vietnamese cassia Vietnamese cinnamon
C.aromaticum	Cassia cinnamon Chinese cinnamon

(Ju *et al.*, 2023)

2.1.3 General composition of cinnamon

The composition varies depending on the geographical origin of the spice and the processing conditions. According to different authors, the following range of variation was observed: carbo- hydrates, 16.6-22.6%; fibre, 25.6-30.5%; moisture, 5.4-11.4%; protein, 3.0-4.5%; volatile oil, 0.3-2.8%; and fixed oil, 0.3 - 1.9% .The variation in the composition of various cassia bark is as follows: moisture (6.5-11.9%); crude fibre (12.0-28.8%); carbo- hydrate (6.9-32.0%); protein (3.1 - 3.4%) fixed oil (0-2.1%): volatile oil (0.5 - 5, 1 deg / 0) and cold alcohol extract (4.6-16.7%) (Leela, 2008). The nutritional composition of cinnamon is given in Table 2.2. Of these, the constituent of commercial importance is the volatile oil.

Table 2.2 Nutritional composition of cinnamon per 100 gm.

Composition	ASTA
Water (g)	10.00
Food energy (KCal)	355
Proteins (g)	4.50
Fat (g)	2.20
Carbohydrate (g)	79.8
Ash (g)	3.50
Calcium (g)	1.60
Phosphorous (mg)	50
Sodium (mg)	10
Potassium (mg)	400
Iron (mg)	4.10
Thiamine (mg)	0.140
Riboflavin (mg)	0.21
Niacin (mg)	1.90
Ascorbic acid (mg)	40.00
Vitamin A activity (RE)	25

(Parthasarathy, 2008)

2.2. CINNAMON LEAF

The leaf of the Indian cassia, known as tajpat in Hindi, is a spice that has a clove like odour and a faint pepper-like aroma. It is a popular flavouring agent in north Indian vegetarian and n-vegetarian preparations. Apart from G. tamola. C. sulphuratum, C. bejolghota and C. impressinervium are also traded as teipot in North-east India. The essential oil is used in the flavouring and formulation of liquors and confections (Ravindran *et al.*, 2003).

2.3. CINNAMON LEAF ESSENTIAL OIL

The essential oil derived from cinnamom leaves is rich in eugenol, that from the roots in camphor and that from the buts shows a high amount of sesquiterpenes (α-bergamotene and a-copaene). It has been established that the oils and extracts from cinnamon possess a distinct antioxidant activity, which is especially attributed to the presence of phenolic and polyphenolic substances, Free radicals are generated in consequence of the metabolism of normal or pathological cells (Schmidth *et al.*, 2006).

2.3.1 Composition of cinnamon leaf essential oil

The essential oil of Cinnamomum verum from Palni Hills, Tamil Nadu, contains a total of 19 components, representing 97.6% of the oil composition. The major constituents present in the essential oil are:

- Eugenol: It is the predominant component, constituting approximately 81.7% of the oil composition. Eugenol is known for its anesthetic, anti-inflammatory, antioxidant, antispasmodic, antiulcer, and vasodilator properties.
- Linalool: This component makes up about 3.8% of the essential oil. Linalool is recognized for its analgesic, anesthetic, antispasmodic, anti-inflammatory, hypnotic, sedative, and anti-allergic properties.
- Benzyl Benzoate: Another significant constituent, accounting for around 3.9% of the total oil composition. Benzyl benzoate exhibits antiasthmatic, antispasmodic, antitumor, hypotensive, and myorelaxant properties (Chakraborty *et al.*, 2015).

2.3.2 Importance of cinnamon (cinnamomum verum) leaf essential oil

Cinnamon is an evergreen, bushy tree belonging to the Lauraceae family, known for its highly aromatic bark and leaves. Commercially, two types of essential oils are extracted from cinnamon: bark oil and leaf oil. Bark oil is utilized in perfumes, flavorings, liquors, and medicines. Leaf oil is used in perfumes, toiletries, seasonings, and the production of eugenol and vanillin. Leaf oil is particularly rich in eugenol (Joy, 1998).

Cinnamon (Cinnamomum zeylanicum Blume, synonym C. verum) is a widely used spice with numerous applications in the perfume, food, and pharmaceutical industries. The essential oil extracted from C. verum bark primarily contains cinnamaldehyde. Recent in vitro studies have demonstrated that C. verum essential oil effectively inhibits food spoilage and the growth of pathogenic bacteria. Beyond its antimicrobial properties, this oil offers various health benefits, making it a promising alternative as a food preservative. However, to utilize cinnamon oil as a food additive, the mechanism behind its antibacterial action needs to be clarified. Cinnamon is recognized for its antioxidant, anti-inflammatory, antimicrobial, anti-diabetic, and anticancer properties. Additionally, it can help prevent heart disease, high cholesterol, and neurological disorders such as Alzheimer's and Parkinson's diseases. Some of the most studied health benefits of cinnamon oil include:

- Reducing inflammation
- Lowering blood sugar levels
- Decreasing bad cholesterol
- Combatting infections
- High antioxidant content
- Boosting the immune system
- Enhancing libido
- Fighting parasites

Cinnamon oil has shown promise in treating cancers such as stomach cancer and melanoma. Compounds like cinnamaldehyde and eugenol have demonstrated effectiveness against leukemia and lymphoma and have been used as nutraceuticals to combat colon and liver cancer cells. Additionally, cinnamon extract is a promising natural substance for oral products to control bad breath by inhibiting the growth of

Solobacterium moorei, killing biofilm, and reducing hydrogen sulfide (H_2S) production. As an antimicrobial agent, cinnamon oil in mouthwash is an alternative to chlorhexidine and is non-toxic to oral keratinocytes, making it suitable for daily use.

Cinnamon is reported to have various health benefits due to its antioxidant content and effects on diabetes, neurological disorders, microbial infections, and cardiovascular diseases, attributed to its bioactive components. The pharmacological properties stem from polyphenolic constituents like phenolic acids, coumarin, proanthocyanidin, and volatile essential oils. Cinnamaldehyde is noted for reducing the production and expression of nitric oxide (NO), interleukin (IL)-1β, IL-6, and tumor necrosis factor (TNF)-α in lipopolysaccharide (LPS)-activated BV2 microglia, thus exhibiting anti-neuroinflammatory properties. It also aids in neuroprotection by potentially inhibiting tau protein aggregation, a hallmark of Alzheimer's disease (AD). Furthermore, cinnamaldehyde and eugenol protect the gut from damage caused by inflammation, infections, and oxidative stress (Blaszczyk *et al.*, 2021).

The phenolic compound eugenol, a major active component of Cinnamomum verum leaf essential oil (EO), belongs to the group of phenylpropanes and is responsible for much of the oil's bioactivity. Various scientists have proposed hypotheses to explain eugenol's antimicrobial action. Schmidt *et al.* (2006) studied the anti-Candida activity of C. verum leaf oil and suggested that its antifungal effect is due to cytochrome P-450 mediated conversion of eugenol into quinone methide. This cytotoxic quinone methide depletes intracellular glutathione (GSH) levels and reacts with cellular macromolecules, leading to cell apoptosis.

Linalool, another component of C. verum leaf EO present in small amounts, also contributes to controlling the virulence mechanisms of Candida spp., including inhibiting germ tube formation and the transition to the hyphal form. A review by Marchese *et al.* (2017) identified several other mechanisms of action of cinnamon leaf EO on various bacterial species. Eugenol can disrupt cell membrane integrity and nonspecific permeability by altering membrane fatty acids and disrupting the plasma membrane of bacterial cells. Additionally, eugenol can inhibit bacterial enzymes in some species, such as Escherichia coli and Bacillus subtilis (Wijesinghe *et al.*, 2020). The antimicrobial activity of the essential oil of Cinnamomum verum was evaluated against five microorganisms: two Gram-negative bacteria (Escherichia coli and Pseudomonas aeruginosa), one Gram-positive bacterium (Staphylococcus aureus), the yeast Candida albicans, and the mold Aspergillus niger (Ainane *et al.,* 2019).

Studies have shown that the antimicrobial effects of essential oils cause structural and functional damage to bacterial cells. This damage includes effects on the cell wall and cytoplasmic membrane, leading to protein denaturation, coagulation of the cytoplasm, inactivation of essential enzymes, and functional alterations of genetic material, ultimately resulting in cell destruction (Cruz-Valenzuela *et al.*, 2019).

Based on in vitro toxicology study results and pharmacodynamics of Cinnamomum verum leaf oil on both sessile and planktonic Candida cells, true cinnamon leaf essential oil shows potential as a therapeutic alternative for Candida infections. The toxicity of true cinnamon essential oil on host tissues was evaluated using an in vitro cell culture model of the human non-cancer keratinocyte (HaCaT) cell line. These toxicology assessments help determine an effective, non-toxic dose and aid in designing dosing regimens by integrating both the pharmacodynamics and pharmacokinetics of C. verum leaf oil (Wijesinghe *et al.*, 2020).

Essential oils of cinnamon leaf demonstrated antimicrobial effects against Listeria monocytogenes in pasteurized milk stored at 7°C and 35°C. This antimicrobial activity depended on the composition and concentration of the essential oils, the food composition, and the duration and temperature of exposure (Cava *et al.*, 2007). Cinnamon leaf oil is intended to be added to feed for all animal species without withdrawal. The maximum proposed use level in complete feed is 25 mg/kg for salmonids and ornamental fish, cats, and dogs, 40 mg/kg for chickens for fattening, and

50 mg/kg for other species/categories. The proposed use level in drinking water is 3 mg/L, using propylene glycol as an emulsifier (Azimonti, 2022).

2.4. CONVENTIONAL METHODS OF ESSENTIAL OIL EXTRACTION

Volatile oils are obtained by different techniques and extraction methods that prioritize the integrity of the bioactive compounds and their respective biological activities. Among the methods most used in the extraction of essential oils, there are traditional techniques, such as hydrodistillation, steam distillation, solvent extraction, and cold pressing, and the innovative ones, such as ultrasound-assisted extraction (UAE), microwave-assisted extraction (MAE), pressurized extraction (PLE) and supercritical fluid extraction (SFE) (Ju *et al.*, 2023).

2.4.1 Hydrodistillation

In order to isolate essential oils by hydrodistillation, the aromatic plant material is packed in a still and a sufficient quantity of water is added and brought to a boil; alternatively, live steam is injected into the plant charge. Due to the influence of hot water and steam, the essential oil is freed from the oil glands in the plant tissue. The vapor mixture of water and oil is condensed by indirect cooling with water. From the condenser, distillate flows into a separator, where oil separates automatically from the distillate water. Mechanism of Distillation Hydrodistillation of plant material involves the following main physicochemical processes:

i) Hydrodiffusion: Diffusion of essential oils and hot water through plant membranes is known as hydrodiffusion.

ii) Hydrolysis: It is defined as a chemical reaction between water and certain constituents of essential oils. Esters are constituents of essential oils and, in the presence of water, especially at high temperatures, they tend to react with water to form acids and alcohols.

iii) Decomposition by heat.

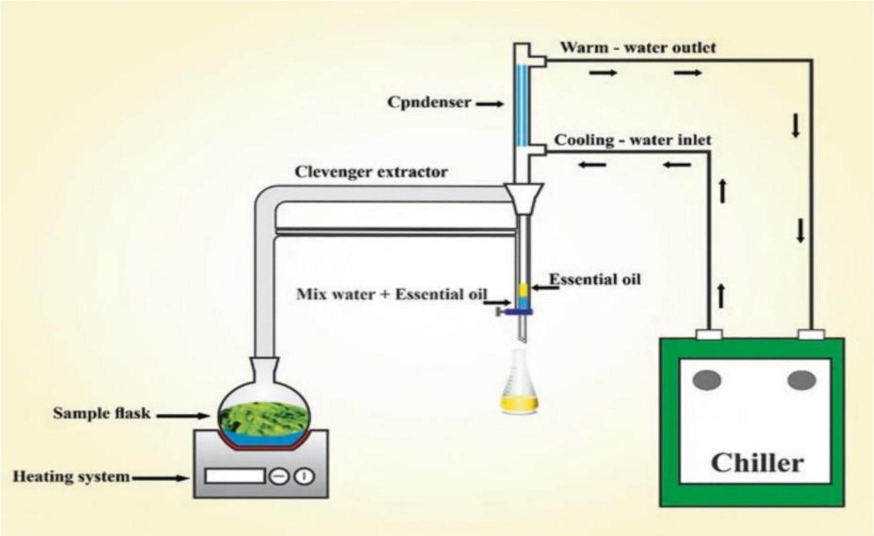


Fig. 2.1 Schematic diagram of hydro-distillation (Ju *et al.*, 2023).

2.4.2 Steam distillation

Steam distillation is the process of distilling plant material with steam generated outside the still in a satellite steam generator generally referred to as a boiler. As in water and steam distillation, the plant material is supported on a perforated grid above the steam inlet. A real advantage of satellite steam generation is that the amount of steam can be readily controlled. Because steam is generated in a satellite boiler, the plant material is heated no higher than 100° C and, consequently, it should not undergo thermal degradation. Steam distillation is the most widely accepted process for the production of essential oils on large scale. Throughout the flavor and fragrance supply business, it is a standard practice. An obvious drawback to steam distillation is the much higher capital expenditure needed to build such a facility. In some situations, such as the large-scale production of low-cost oils.

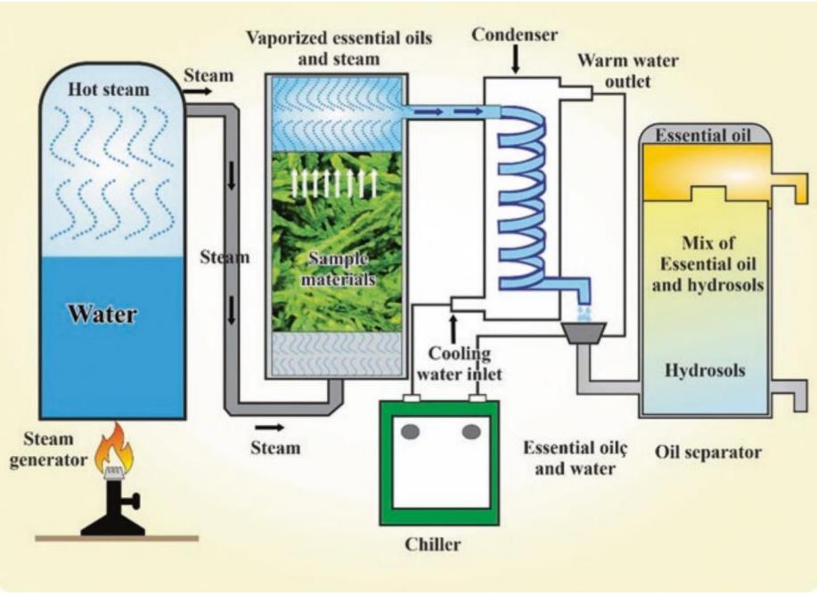


Fig. 2.2 Schematic diagram of steam distillation (Ju *et al.*, 2023).

2.4.3 Microwave assisted extraction

Microwave assisted extraction is the treatment of plant material with microwave irradiation during extraction to enhance the recovery of secondary metabolites and aroma compounds. The forced heating of water in core of material may cause liquid vapourisation within the cells, which may lead to the rupture of cell walls and/or plasma membranes. This extraction method is rapid compared to conventional method like hydrodistillation, soxhlet extraction. MAE technique produced more oxygenated compounds, is more cost effective and environmental friendly (Sethunga *et al.*, 2022).

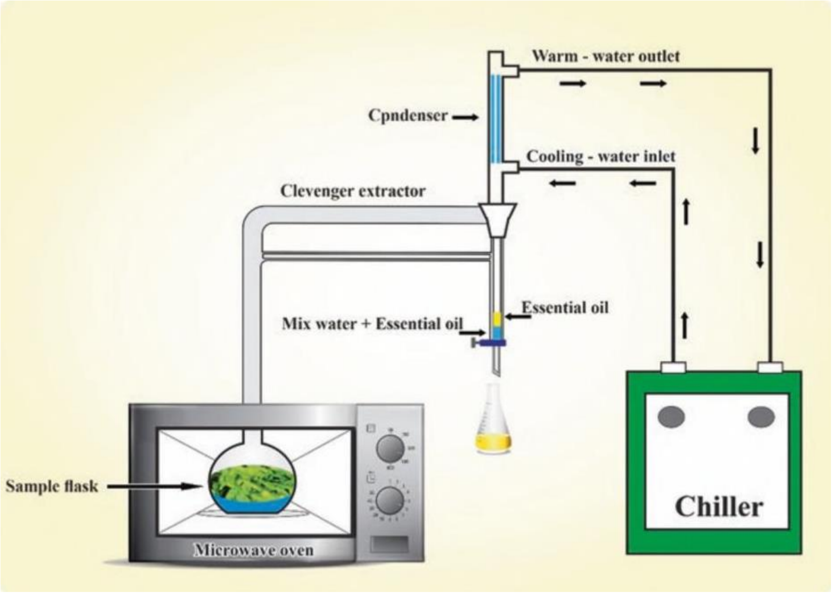


Fig. 2.3 Schematic diagram of microwave assisted extraction (Ju *et al.*, 2023).

2.4.4 Ultrasound Assisted Extraction

Ultrasound-assisted extraction (UAE) yields more in less time and at lower temperatures by enhancing mass transfer and reducing solvent use. It's great for extracting heat-sensitive components, as it operates at low temperatures. Ultrasonic waves create high-speed solvent jets, opening cell walls and promoting solvent penetration. Cavitation ruptures cell walls, allowing selective compounds to dissolve in the solvent. UAE is performed using ultrasonic baths in labs and ultrasonic heads at pilot scale (Sethunga *et al.*, 2022).

2.4.5 Supercritical Fluid Extraction

Supercritical fluid extraction (SCFE) isolates extracts from plants using supercritical fluids, offering cost-effective and environmentally friendly benefits. By adjusting temperature and pressure, these fluids exhibit both gas and liquid properties, enhancing extraction efficiency. Carbon dioxide is commonly used due to its safety and effectiveness, although water is an alternative with higher requirements. Recent advancements include polar modifications and enzymatic pre-treatment, improving extraction capabilities. Factors like solvent flow rate and temperature affect SCFE, yielding extracts with superior properties. However, challenges include CO2 retention risks and industrial application difficulties (Sethunga *et al.*, 2022).

2.4.6 Enzyme Assisted Extraction

Enzyme assisted extraction (EAE) has been developed to enhance the efficiency of extraction, increasing the yields in an eco-friendly way by using enzymes and reducing the use of solvents, extraction time, energy inputs, and environmental issues. In EAE, hydrolytic enzymes such as alpha-amylase, cellulase, pectinase, and protease enzymes or blends hydrolyze the cell wall components such as cellulose, hemicelluloses, lignin, and pectin . Factors like enzyme concentration, pH, and temperature influence EAE, which can also serve as a pre-treatment for essential oil distillation, yielding higher yields compared to traditional methods (Sethunga *et al.*, 2022).

2.5 INSTANT CONTROLLED PRESSURE DROP (DIC)

Instant controlled pressure drop (DIC: De´tente Instantane´e Controle´e(French acronym)) is a thermomechanical process of great specificity and relevance in texturing, in microbiological decontamination, in extraction by autovaporization of volatile active molecules etc. DIC was initially defined, designed, studied, and patented in its different application areas by Professor Allaf’s team in 1988.

The general concept of DIC is based on subjecting the product to an abrupt pressure drop towards the vacuum (about 5 kPa) after a treatment at high temperature (up to 180 °C) during a short time period (5–60 s), depending on the desired outcome. At first, the material is placed in a processing vessel where a primary vacuum is established. This can ensure, in the next step, a more intimate contact between the saturated steam and the exchange surface. Then, the product is exposed to a saturated steam pressure bringing up speedily, partially by convection and primarily by condensation, the product’s exchange surface to the steam temperature; then, a conduction process will enable the heat transfer within the product. Subsequently, the abrupt pressure drop towards the vacuum induces an instantaneous autovaporization of water and other volatile molecules. This induces a similarly instantaneous cooling, which stops the thermal degradation. The instantaneous autovaporization generates an adequate amount of vapor acting as a source of mechanical expansion stress in the internal pores. As the pressure abruptly drops, the temperature level immediately drops to stabilize at the equilibrium level depending on the total pressure. The level of the vacuum pressure is defined according to the glass transition of the material. The speed of temperature drop implies maintaining the rheological viscoelastic behavior. Thus, the mechanical stresses produced by the generated vapor can trigger structural modification.

The autovaporization of the volatile molecules can be carried out in successive cycles (multi-flash autovaporization). It leads to the definition of a highly efficient operation, especially used for the direct extraction of essential oils performed in few minutes. The short duration of the operation allows the preservation of the residual non-volatile compounds usually in an expanded matrix. DIC autovaporization of volatile molecules has also been defined, studied, and optimized as a fast, efficient, and high-quality essential oil extraction technique. The multi-flash autovaporization (MFA) involves successive DIC cycles intending to evaporate the adequate molecules. Its efficiency is closely related to the molecule's volatility that is only a function of the exchange surface temperature. Thus, DIC-MFA leads to a significantly interesting extraction yield much higher than the steam distillation and hydrodistillation processes. However, it is worth noting that the instantaneity of DIC autovaporization inevitably leads to a fog-like presence of the various essential oil (EO) compounds and water; thus, an EO water emulsion is obtained. Conventional evaporation or solvent separation step can be added as a way to achieve the separation between water and the EO molecules (Nader *et al.*, 2022).

2.5.1 Components of DIC Equipment

DIC equipment is mainly composed of four components as presented in Figure 2.4

- A processing vessel, which is an autoclave with a heating jacket where the product to be treated is placed.
- A pneumatic valve, which ensures a nearly instant liberation of steam pressure contained in the treatment vessel to the vacuum tank.
- A vacuum system composed of a vacuum pump and a tank with a cooling jacket. The tank volume is usually 100–130 times higher than the volume of the processing vessel. A water ring pump maintains the tank pressure at about 2.5–5 kPa.
- An extract collection trap used to recover condensates (Hamoud-Agha and Allaf, 2019).

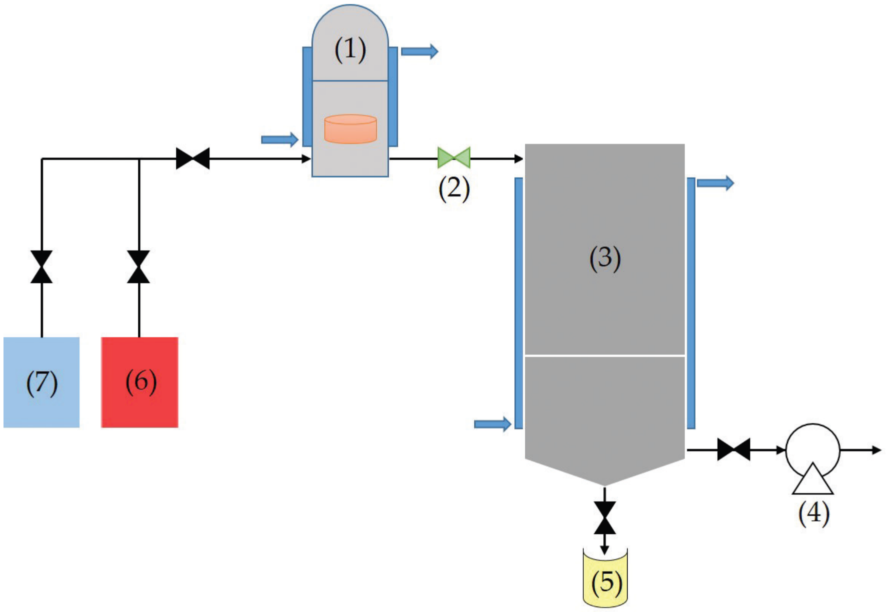


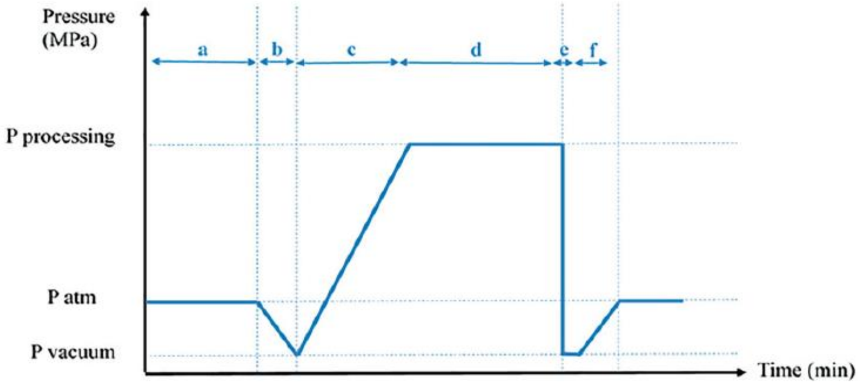
Fig. 2.4 Schematic presentation of a typical DIC reactor (1) Treatment vessel, (2) controlled instant pressure drop valve, (3) vacuum tank with cooling jacket, (4)

vacuum pump, (5) extract collection trap, (6) steam generator and (7) air compressor.

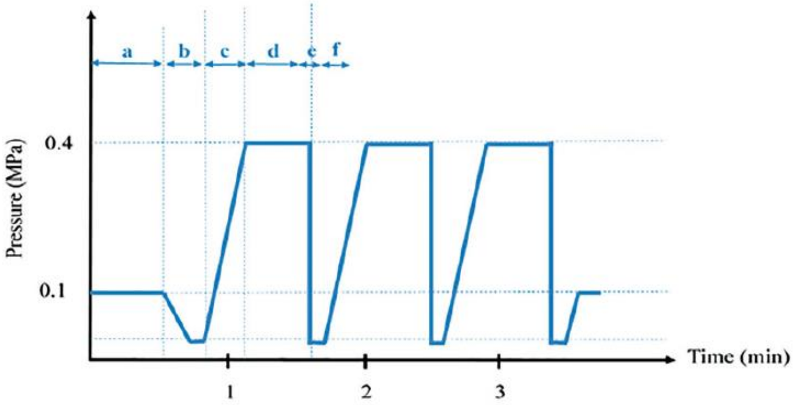
2.5.2 Procedure of DIC

1. Place the product in a processing vessel.
2. Apply a First Vacuum Step (5 kPa) Just before Injecting the Steam. This allows closer thermal contact between the steam (i.e., the heating fluid) and the product exchange surface.
3. Connect the Steam Generator to the Treatment Vessel.
4. Maintain the Product at High Saturated Vapor Pressure for a Short, Predetermined Period for Thermal and Moisture Homogenization.
5. Instantaneously Open the Main Vacuum Valve- An instant pressure drop towards the vacuum, at a rate ($\Delta P/\Delta t$) greater than 0.5 MPa/s, is performed in the processing vessel.
6. Injection of an Airflow to Reach the Atmospheric Level
7. Recovering of the Mixture from the Vacuum Tank
8. Recover the mixture from the Vacuum Tank
9. Multi-DIC Cycles- In some cases, multiple DIC cycles may be performed (Fig. 2.5 (b)). The multi-cycle DIC consists of n repetitions of steps (3-5) (Nader *et al.*, 2022).

The temperature and pressure level during DIC is shown in Fig. 2.5.



(a) DIC Treatment-single cycle



(b) Multi-cycle DIC

Fig. 2.5 DIC treatment cycle (a) Treatment vessel at atmospheric pressure; (b) initial vacuum of 5 kPa; (c) injection of saturated steam; (d) maintaining the pressure for a specific time; (e) abrupt pressure drop; (f) vacuum period followed by restoration of atmospheric pressure

2.6 DIC PRETREATMENT ON EXTRACTION

Abdallah *et al.* (2023) explores the intensifying effect of Instant Controlled Pressure Drop (DIC) pre-treatment on hesperidin recovery from orange byproducts, showcasing enhanced antioxidant and antidiabetic activities of the extracts. Comparison of extraction methods reveals that the DIC-ASE method yields the highest hesperidin content, indicating the efficacy of DIC in improving extraction efficiency. Principal Components Analysis (PCA) demonstrates significant differences between extracts obtained using different methods, emphasizing the impact of extraction techniques on the composition and properties of the extracts. Extracts obtained with DIC pretreatment exhibit improved antioxidant and in vitro antidiabetic activities, highlighting the potential of DIC in enhancing the bioactive properties of the extracts. The significance of DIC pre-treatment lies in its ability to intensify the recovery of bioactive compounds, improve extract quality, and enhance functionality, making it a promising technique for valorizing orange byproducts.

Mkaouar *et al.* (2016) studied the impact of instant controlled pressure drop (DIC) technology on the extraction kinetics of polyphenols from olive leaves. The extraction process involved the use of ethanol (95%) as the solvent at a temperature of 55°C, with a ratio of 40 g/g dry basis for 3 hours. Solvent extraction kinetics of both untreated and DIC-treated olive leaves were conducted using an oil batch extractor system, where approximately 5 g of olive leaf powder was mixed with 250 ml of 95% ethanol at 55°C for both untreated and DIC-textured leaves. DIC treatment significantly increased the extraction yields of total polyphenol content (TPC) compared to untreated leaves, with TPC reaching 187.31 mg g^{-1} dry basis in DIC-treated leaves versus 67.76 mg g^{-1} in raw material extracts. Extraction time was reduced from 120 to 15 minutes with DIC treatment, showcasing a threefold increase in extracted polyphenols quantity compared to untreated leaves. The study demonstrated that DIC texturing is a promising method to intensify the extraction kinetics of olive leaf polyphenols, potentially increasing the industrial value of olive leaf extracts for various applications.

2.7 DIC ASSISTED HYDRO-DISTILLATION

Teresa-Martinez *et al.* (2022) conducted study on Instant Controlled Pressure Drop (DIC)Technology in Cardamom Essential Oil Extraction and Antioxidant

Activity. The study explores the impact of coupling DIC technology with hydrodistillation (HD) on essential oil extraction from cardamom seeds, revealing a significant increase in essential oil yield. The Essential Oil yield of DIC-HD (140°C and 30 s) was 4.43 compared to 2.52 for the control. DIC technology enhances the antioxidant scavenging capacity of essential oils extracted from cardamom seeds. The Antioxidant capacity of DIC-HD (165°C and 30 s) was 86% inhibition, whereas the control had 57.02% inhibition. The research emphasizes the potential of DIC technology to improve extraction efficiency and biological activities of essential oils, offering a novel approach to enhance the quality of extracted oils.

MATERIALS AND METHODS

CHAPTER III

MATERIALS AND METHODS

This chapter outlines the Instant Controlled Pressure Drop (DIC) system and the hydro-distillation system for extracting essential oil from cinnamon leaf (Cinnamomum verum). It details the process of evaluating and optimizing the process parameters for extracting cinnamon leaf essential oil and determining its physical properties. It also details about the processes of sun drying and heat pump drying to find the drying curve equation of best fit. Additionally, the chapter covers the identification and quantification of compounds in the oil using Gas Chromatography-Mass Spectrometry (GC-MS).

3.1 DRYING STUDY

3.1.1 Collection of raw material

Cinnomon leaves required for the study were collected from Instructional farm of KCAET, Tavanur. Leaves were separated from the stem and washed thoroughly to remove dust and dirt.

3.1.2 Moisture content determination

Moisture content was measured using infrared moisture analyzer (SHIMADZU MOC63U Uni Bloc). The sample was placed on a precision sample pan inside the moisture meter, ensuring even distribution for accurate measurement. The high-precision balance measured and recorded the initial weight of the sample.

An infrared lamp heated the sample evenly, preventing burning or overheating. As the sample heated, moisture evaporated. The moisture meter continuously measured the sample’s weight, tracking weight loss due to moisture evaporation in real time. The initial weight and real-time weight loss data were used to determine the moisture percentage.

Advanced algorithms processed the weight data to update the moisture content continuously. The measurement ended when weight loss stabilized, indicating most moisture had evaporated. Finally, the moisture meter displayed the moisture content as a percentage of the initial sample weight on its digital screen.

Plate 3.1 Infrared moisture analyzer

3.1.3 Drying of cinnamon leaves

A drying study was conducted in two methods: Heat pump drying and Sun drying to find the drying rate equation of best fit.

3.1.3.1 Sample preparation

Matured green colored cinnamon leaves about 200g were collected from farm of KCAET, Tavanur. Leaves were separated from the stem and cleaned to remove dust and dirt. Moisture content of leaves was measured using Infrared Moisture Meter.

3.1.3.2 Sun drying

About 100 g of the sample were taken. Arrange the leaves in a single layer on clean, dry trays, ensuring they are not overcrowded to allow for proper airflow. Place the trays in a sunny area with direct sunlight, ensuring they receive consistent exposure throughout the drying period. Depending on the intensity of the sunlight and ambient temperature, check the leaves periodically, flipping them over halfway through to promote uniform drying. The weight of the leaves was measured every 30 minutes. The experiment continued till the weight of sample showed constant value. Once the drying was complete, the final moisture content of the leaves was measured. The experiment was conducted in the first week of the month of march.

3.1.3.3 Heat Pump drying

About 100 g of the sample were spread in thin layers on trays and is kept inside heat pump dryer. The temperature of dryer is set at 55°C. The weight of the leaves was measured every 30 minutes. The experiment continued till the weight of sample showed constant value. Once the drying is complete, the final moisture content of the leaves is measured.

Plate 3.2 Heat pump drying

Based on these measurements, drying curves were plotted to compare the effectiveness of heat pump drying and sun drying.

3.1.4 Heat Pump Dryer

Drying of cinnamon leaf is done using IKE Closed-Loop Heat Pump Dehydration Dryer, model no: WRH-100B, typically constructed from stainless steel, is capable of handling batches between 20 to 100 kg and operates at a maximum power of 2.6 kW consumption within a temperature range of 50-65°C.

Its working principle involves drawing moist air from the drying chamber and passing it over an evaporator coil, where the moisture condenses and is removed. The dehumidified air is then reheated via a condenser coil before being recirculated back into the drying chamber to evaporate more moisture from the leaves. This process efficiently recycles heat, maintaining lower drying temperatures which preserve the quality of the leaves and minimize energy consumption.

Plate 3.3. Heat pump dryer

3.1.5 Drying characteristics of cinnamon leaves

The cinnamon leaves were dried employing two drying methods *viz.*, heat pump drying and sun drying. The sample was dried in heat pump dryer at 55°C to reduce drying time and production of good quality cinnamon leaves.

3.1.5.1 Moisture content

The moisture content of the dried samples obtained through both the heat pump drying and sun drying processes was determined using an infrared moisture meter. The samples were dried at 55°C until a constant weight was achieved. The moisture (wb) content was then calculated as follows:

$$\text{Moisture content (\%)} = \frac{w1-w2}{w1} \times 100$$

where, w_1 and w_2 are initial and final weight of the sample, respectively (Meena and Prince., 2022).

3.1.5.2 Moisture ratio

The moisture contents obtained for both drying processes were converted to dimensionless moisture ratio (MR) by the following equation.

$$MR = \frac{\text{Xt}-\text{Xe}}{\text{Xo}-\text{Xe}}$$

where, MR is the moisture ratio; Xo, Xe, and Xt are the initial moisture content, equilibrium moisture content, and moisture content at any drying time (g water/g dry material), respectively (Meena and Prince., 2022).

3.1.5.3 Thin layer drying models

Experimental data was modeled using various mathematical approaches, and the drying kinetics for each process were evaluated based on the drying curves. These mathematical models describe the moisture ratio (MR) as a function of temperature, linking the moisture gradient at any given time to the initial and equilibrium levels. The four most commonly used drying models—Newton, Page, Henderson and Pabis, and Logarithmic models were applied to the curve. All these equations are given below in Table 3.1.

Table 3.1 Mathematical models adopted for drying studies

Sl No.	Model	Equation
1	Newton	$MR = exp(-kt)$
2	Page	$MR = exp(-kt^{n})$
3	Henderson and Pabis	$MR = a\ exp(-kt)$
4	Logarithemic	$MR = a\ exp(-kt) + c$

where a, c, n are empirical constants, k is the drying constant and t is the drying time (Meena and Prince., 2022).

3.2 INSTANT CONTROLLED PRESSURE DROP SYSTEM

The Instant controlled pressure drop system developed by Banu *et al.* 2023 in Food Engineering Laboratory of KCAET, Tavanur was used for conducting experiments. The DIC system exposes the material to saturated steam briefly, followed by an immediate pressure drop. This process triggers auto-evaporation of water, facilitates product texturing, and initiates cooling. It induces a rapid transition from high steam pressure to vacuum, enhancing the biomaterial's overall diffusivity. The DIC system consists of the following components:

1. Steam generation system

2. Processing vessel

3. Vacuum system

4. Supporting frame

3.2.1 Steam generation system

The steam generator, a 10-liter capacity aluminum pressure vessel positioned outside the DIC cluster, utilized LPG as a heat source to convert water into steam. Steam pressure was monitored using a Dynamics pressure gauge, ranging from 0 to 7 kg/cm2, located on the vessel's lid (refer to plate 3.4). Steam from the generator flowed through an 8 mm diameter silicone tube to the processing vessel, where it interacted with the raw materials. The flow of steam into the processing vessel was regulated using a brass ball valve installed on the steam outlet pipe.

Plate 3.4 Steam generator

3.2.2 Processing vessel

A processing vessel, designed to endure sudden shifts from high steam pressure to vacuum conditions, was employed for treating the product. This vessel, made of 3 liters of stainless steel, served as the containment for the material being processed. Steam from the generator was directed into the processing vessel via a silicone tube and regulating valve. Additionally, a vacuum tank was connected to the vessel using a polyurethane tube with a 12mm outer diameter. The airflow between the vacuum tank and processing vessel was managed by a pneumatic regulating valve. Raw materials for processing were accurately measured and placed inside the vessel in a designated container. Before commencing treatment, the vessel was securely sealed to prevent any leakage through the lid.

Plate 3.5 Processing vessel

3.2.3 Vacuum system

The vacuum system of the DIC reactor comprises a vacuum tank, a vacuum pump, a controlled instant pressure drop valve, and associated measurement instruments. The vacuum tank, constructed from 1.5 cm thick PVC measuring 75 cm in length and 21.5 cm in outer diameter, is vertically mounted using a mild steel ring attached to the bottom frame. Both ends of the tank are sealed with 20 cm inner diameter PVC caps, giving it a total volume of 23 liters (refer to plate 3.6 (a)). Air from the vacuum tube flows through a 12 mm outer diameter polyurethane tube inserted through the upper cap, connected to a vacuum gauge via a 0.25 inch pneumatic push-type male connector. Air from the vacuum pump enters the vacuum tube through a 10 mm diameter polyurethane hose, facilitated by a pneumatic push-type male connector at the lower end.

The vacuum pump utilized (Make: Value, Model: VE115N) operates with a flow rate of 57 L/min and achieves an ultimate vacuum of 150 μHg (20 Pa), powered by a 230 V, 0.25 HP motor (plate 3.6 (b)). It is connected to the tank via a 10 mm diameter polyurethane tube at its base.

To measure the airflow rate from the vacuum tank to the processing vessel through the 12 mm diameter polyurethane tube, a vacuum gauge (Make: Manometer, 0-760 mm of Hg) is employed. This gauge is installed along the path of the tank using a 0.25 inch pneumatic push-type male connector, with its other end connected to a controlled instant pressure drop valve via a 0.25 inch female tee.

A pneumatic inline flow control valve (Make: Janatics, Model: C207) in conjunction with a brass ball valve serves as a controlled instant pressure drop valve. This assembly is installed on the polyurethane tube that connects the vacuum tank to the processing vessel. The valve setup is designed to adjust and decrease the flow rate within a pneumatic circuit. It effectively regulates the vacuum from the tank. One end of the flow control valve is connected to the vacuum gauge using a quarter-inch female tee, while the other end is linked to the ball valve via a quarter-inch brass hex nipple (refer to plate 3.6 (c)).

b) Vacuum tank

a) Vacuum pump

c) Instant controlled pressure drop valve

Plate 3.6 Vacuum system

3.2.4 Supporting frame

The support frame structure of the DIC equipment consisted of one-inch mild steel angles. A 10-gauge aluminum sheet covered the bottom of the frame. The vacuum tank was mounted vertically on a mild steel plate and supported by an adjustable one-inch mild steel ring secured with screws. The controlled instant pressure drop valve and vacuum gauge were mounted on a one-inch mild steel plate.

3.3 HYDRO-DISTILLATION UNIT

The Extraction unit consists of a Clevenger hydro distillation system -which consists of a round bottomed flask, Clevenger, a condenser, ad a heating mantle (plate 3.7).

a) Heating mantle

b) Clevenger **c) Condenser** **d) Round Bottom Flask**

Plate 3.7 Clevenger Apparatus

Water flows through the condenser. The volume of round bottom flask was 500 ml. The dimension of mouth of the mantle match with the roundness of flask. Clevenger was specific to higher density oils.

3.4 EXPERIMENTAL DESIGN

The process parameters which would influence the essential oil yield such as process pressure, no. of cycles of DIC, and solvent-sample ratio were taken as independent variables and their ranges for the study were fixed based on thorough review of literature and the preliminary studies conducted. Physical quality characteristics of extracted essential oil were taken as dependent variables.

3.4.1 Independent variables

a) Process steam pressure (kg/cm^2):

1) P1: 0.50 kg/cm^2

2) P2: 0.75 kg/cm^2

3) P3: 1.00 kg/cm^2

b) No. of cycles of DIC:

1) N1: 1

2) N2 :2

3) N3 :3

d) Solvent- sample ratio (ml/g)

1) A1: 7 ml/g

2) A2: 10 ml/g

3) A3: 13 ml/g

3.4.2 Dependent variables

3.4.2.1 DIC assisted Hydro-distillation system output parameters:

a) Essential oil yield

b) Energy consumption

3.4.2.2. Physical quality characteristics of cinnamon leaf essential oil:

a) Refractive index

b) Specific gravity

3.5 EXPERIMENTAL PROCEDURE

Experiments were conducted in the Food Engineering Laboratory of K.C.A.E.T, Tavanur.

3.5.1 Procedure For Essential Oil Extraction

3.5.1.1 Preparation of the raw material

Matured green colored cinnamon leaves about 1.3 Kg were collected from farm of KCAET, Tavanur. Leaves were separated from the stem and cleaned to remove dust and dirt. Moisture content of leaves was measured using Infrared Moisture Metre. The cleaned leaves were then spread in trays in thin layers and dried at 55°C for two hours forty-five minutes in a Heat Pump drier. The dried leaves were then kept in atmospheric temperature to cool down. The cooled leaves were then filled in LDPE pouches, sealed and stored in a dark compartment at ambient temperature (27±5 °C) for further use.

(a) Fresh leaves (b) Heat Pump drying (c) Prepared Sample

Plate 3.8 Raw material

3.5.1.2 DIC pretreatment process

The sample was subjected to abrupt pressure changes using the DIC system.

The process began with the steam generator, which was half-filled with water, sealed, and heated using LPG. During steam generation, the steam control valve remained closed. Thirty grams of dried cinnamon leaves were crushed by hand and placed in an open steel pan inside the processing vessel. Once the sample was positioned, the vessel was tightly sealed to ensure there were no leaks through the lid. The vacuum pump was then activated to create an initial vacuum within the processing vessel. This was done by drawing air out through a polyurethane hose connected to an opened pneumatic valve. The establishment of this initial vacuum facilitates a close exchange between the incoming steam and the product surface, enhancing the process's efficiency. Once the vacuum was adequately established, the pneumatic valve was closed, allowing the pressure changes to occur and optimize the hydration kinetics of the cinnamon leaves.

Steam from the steam generator, regulated by the steam control valve, was directed into the processing vessel via a silicone tube. Saturated steam was injected into the reactor at a fixed process pressure level as specified by the experimental design. During this phase, heat transfer occurred primarily through steam condensation, which released latent heat and ensured a very high coefficient of heat transfer. Once the desired conditions were achieved, the steam control valve was closed. The material was then subjected to an instant controlled pressure drop towards a vacuum by opening the valve. This rapid vaporization induced instant cooling of the samples. After maintaining the vacuum stage for a few seconds, the pressure in the vessel was released by opening the pressure release valve. The number of DIC cycles were done as per the experimental design. Finally, the sample was recovered from the processing vessel and prepared for the subsequent hydro-distillation extraction process.

Plate 3.9 DIC Pretreatment Process

3.5.1.3 Hydro-distillation with Clevenger Apparatus

First, the DIC pre-treated sample was filled into the round bottom flask. Then distilled water was poured in the ratios of 1:7, 1:10, and 1:13 as per the experimental design. The Clevenger apparatus was set up, ensuring the pipe fittings were proper and a minimum water flow was maintained through the condenser. The heating mantle temperature was set at 90-95°C and switched on.

The vapor produced, containing volatile oils, traveled through the condenser, where it condensed and collected in the graduated receiver. The oil settled at the bottom and the water above it because the density of cinnamon leaf essential oil is slightly higher than that of water. After a suitable distillation time of about 2-2.30 hours, the receiver screw was opened, and the essential oil was collected in amber bottles and stored under refrigerated conditions. The collected essential oil was then analyzed as per the experimental design.

(a) Clevenger apparatus **(b) Essential Oil Extraction** **(c) Extracted Oil**

Plate 3.10 Hydro-distillation with Clevenger Apparatus

3.5.2 Energy Measurement

Energy consumption was measured using energy meter as per the procedure explained by Claudia *et al*. 2017. A single-phase electronic energy meter (SPEM 01; 240V; 50 Hz) was connected to the hydro distillation unit to measure the energy consumed during the distillation process (Plate 3.11.).

Plate 3.11 Energy meter

3.6 STATISTICAL ANALYSIS

Response surface methodology (RSM) was chosen for the design of experimental combinations. This method is based on the multivariate non-linear model which is widely used for optimisation process. It is helpful to study the interactions of the various parameters that affect the process. The main advantage of RSM is reducing the number of experimental runs needed for providing sufficient information about statistically acceptable results (Montgomery, 2001). The dependent variables considered were Oil Yield(Y1), Refractive index (Y2), Energy (Y3) and Specific gravity (Y4). The independent variables considered were Steam Pressure (A), Number of cycles of DIC treatment (B), and Solvent-Solid ratio (C). The three levels of the process variables were coded as -1, 0 and +1. The values of independent variables at three levels were shown in Table 3.2.

Table 3.2 Values of independent variables at three levels of Box–Behnken design

Independent Variables	Symbol		Level	
	Coded	Uncoded	Coded	Uncoded
Steam Pressure	A	P	-1	0.50
			0	0.75
			+1	1.00
No. of cycles of DIC treatment	B	N	-1	1
			0	2
			+1	3
Solvent-Solid Ratio	C	A	-1	7
			0	10
			+1	13

The experiments were designed using Design Expert Software, Version 12.0 (State-Ease, Minneapolis, MN). The same software was used for statistical analysis of experimental data. According to Box-Behnken design for three independent factors, the total experiments to be conducted are found to be seventeen. Seventeen experiments were performed with three variables as shown in Table 3.3.

Table 3.3 Experimental design used for DIC assisted extraction of cinnamon leaf essential oil

Standard order	Run	Coded variables			Un-coded variables		
		Steam Pressure (kg/cm^2)	**No. of cycles of DIC treatment**	**Solvent Solid ratio (ml/g)**	**Steam Pressure (kg/cm^2)**	**No. of cycles of DIC treatment**	**Solvent-Solid ratio (ml/g)**
1	1	-1	-1	0	0.5	1	10
2	2	1	-1	0	1	1	10
3	17	-1	1	0	0.5	3	10
4	5	1	1	0	1	3	10
5	3	-1	0	-1	0.5	2	7
6	7	1	0	-1	1	2	7
7	4	-1	0	1	0.5	2	13
8	10	1	0	1	1	2	13
9	16	0	-1	-1	0.75	1	7
10	8	0	1	-1	0.75	3	7
11	14	0	-1	1	0.75	1	13
12	6	0	1	1	0.75	3	13
13	13	0	0	0	0.75	2	10
14	5	0	0	0	0.75	2	10
15	11	0	0	0	0.75	2	10
16	12	0	0	0	0.75	2	10
17	9	0	0	0	0.75	2	10

3.7 DETERMINATION OF PHYSICAL QUALITY CHARACTERISTICS OF ESSENTIAL OIL

3.7.1 Refractive Index

The refractive index of cinnamon leaf essential oil was measured using Abbe refractometer (Advance Research Instrument company, Model: R8, India) as shown in Plate 3.12. A refractometer is used to determine the concentration of a particular substance within a given solution. It operates based on the principle of refraction. When rays of light pass from one medium in to another, they are bend either toward or away from a normal line between the two medium.

The sample was positioned as a thin layer (about 0.1mm) between two prisms. The upper prism is securely mounted on a bearing, enabling it to rotate using the side arm indicated by dotted lines. The lower prism is hinged to the upper one, allowing separation for cleaning and sample introduction. The lower prism faces rough-ground: when light reflected in to the prism, this surface effectively becomes the source for an infinite number of rays that pass through the sample at all angles. The radiation is then refracted at the interface of the sample and the smooth-ground face of the upper prism. After this it passes into the fixed telescope.

Two Amici prisms, which can be rotated relative to each other, are used to gather the divergent critical angle rays of different colors into a single white beam, matching the path of the sodium D ray. The telescope's eyepiece has cross hairs, and during measurement, the prism angle is adjusted until the light-dark interference aligns precisely with the cross hairs. The prism's position is then determined from the fixed scale.

The experimental steps were as follows: First, the prism surfaces were cleaned with alcohol using a cotton plug. Then, 2-3 drops of the sample were placed between the prisms with a dropper, and they were pressed together. Light was allowed to fall on the mirror and was adjusted to reflect the maximum light into the prism box. The prism box was rotated using the lever until the boundary between the shaded and bright areas appeared in the field of view. If a band of color appeared at the light-shade boundary, it was sharpened by rotating the compensator. The lever was adjusted so that the light-shade boundary passed exactly through the center of the crosshair. Finally, the refractive index was read directly from the scale.

Plate 3.12 Abbe Refractometer

3.7.2 Specific Gravity

Specific gravity was determined by dividing the weight of one ml essential oil by the weight of one ml distilled water. Weights were calculated utilizing a balance with an accuracy of 0.001g (Gopika and Ghuman, 2014).

3.8 DETERMINATION OF CHEMICAL CONSTITUENTS

The cinnamon leaf essential oil extracted through Instant Controlled Pressure Drop assisted Hydro-distillation process was analyzed using GCMS (Shimadzu Model No. GCMS-QP2010) for volatile profiling using RTX5MS column (30m long). The temperature programming was as follows: Chromatography coupled with triple axis detector (Shimadzu GCMS-QP2010). Column: RTX 5MS 30 m x 0.250mm x 0.25 µm; Injection volume: 2 µL; split ratio: 50:1. Helium gas (99.9995%) flow rate: 1 mL/min. The estimation was executed using an EI (electron impact) mode along with 70 eV of ionization energy. The injector temperature: 280°C (constant). The column oven temperature program: 65°C for 2 min, ramped to 155°C @ 3°C/min, hold for 10 min ramped to 250°C @ 10°C/min, hold for 5 min. The volatiles were was identified after comparing the spectral configurations obtained with that of available mass spectral database (NIST-08 SPECTRAL DATA).

Plate 3.13 GC-MS apparatus

RESULTS AND DISCUSSION

CHAPTER IV

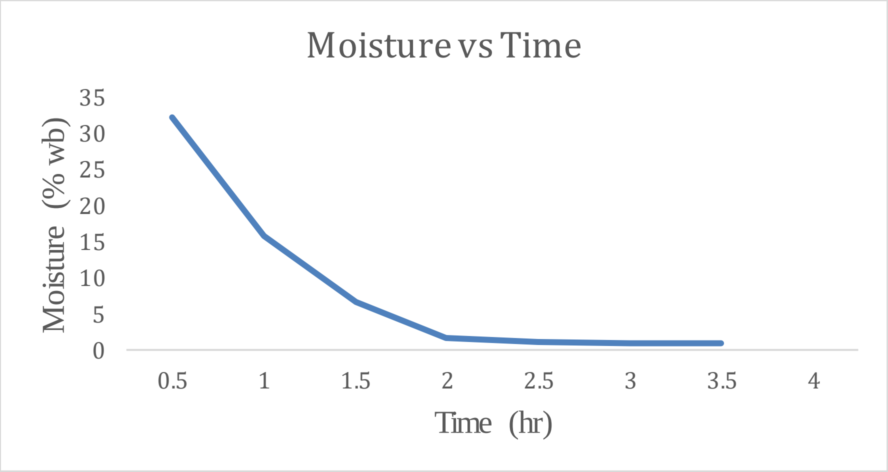
RESULTS AND DISCUSSION

This chapter deals with the evaluation of instant controlled pressure drop assisted hydro-distillation unit towards extraction of *Cinnamomum verum* leaf essential oil. The outcomes of the various experiments conducted to optimise the process parameters and the effect of process parameters on physical and chemical characteristics of the of the extracted essential oil are discussed.

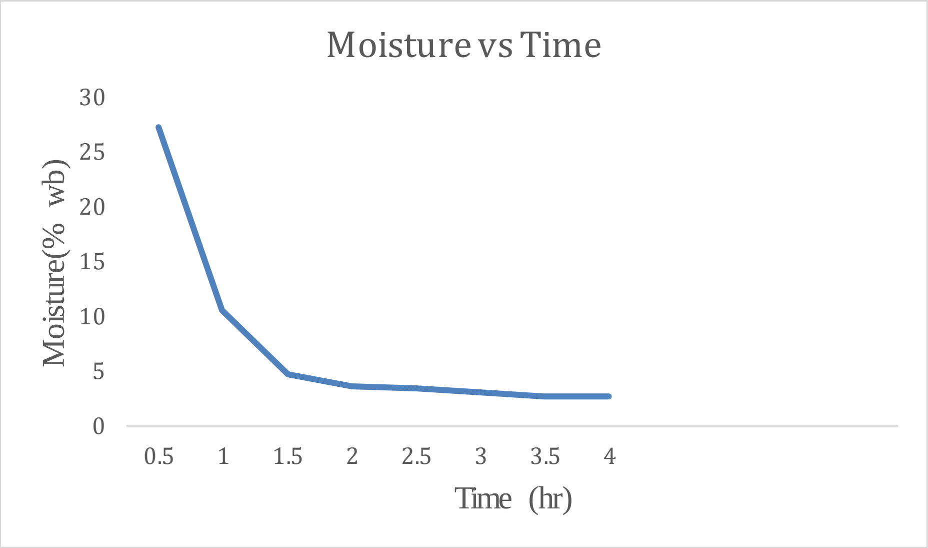
4.1 DRYING STUDY – EQUATION OF BEST FIT

4.1.1 Drying characteristics

The initial moisture content of cinnamon leaves before drying was found to be 53.82% (wb). and final moisture content reported after drying was around 0.92% (wb) in sun dried samples and 2.72% (wb) in heat pump dried leaves. Figure 4.1 (a) and (b) shows the variation of moisture content with time of sun dried and heat pump dried cinnamon leaves. It could be seen that the moisture content of all samples declined continuously as a function of time for both drying conditions, indicating that internal mass transfer was governed by moisture diffusion.



(a) Sun dried sample



(b) Heat pump dried sample

Fig. 4.1 Moisture vs Time graph

4.1.2 Mathematical modelling

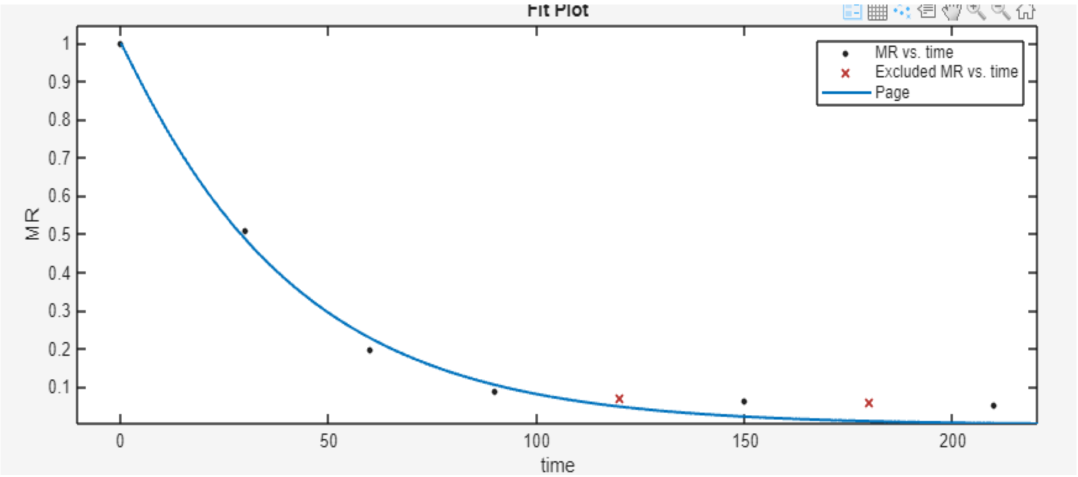
The moisture ratio against drying time for the experimental data of sundried and heat pump dried samples was fitted to 4 thin layer drying models as mentioned in Table 3.1. The results obtained employing software is shown in Appendix A (Table A.3 and A.4). These tables give the values of coefficient of determination (R^2), reduced mean square of deviation (χ^2) and root mean square error (RMSE). The model of best fit for explaining the drying kinetics of cinnamon leaf was selected based on higher values of R^2, and lower values of χ^2 and RMSE values.

Results revealed that the Page model gave a good fit for all the drying process amongst the selected models. Page model attained relatively higher R^2 value of 0.9992 for sun dried samples. R^2 values for heat pump dried sample was 0.9897. Furthermore, the lowest χ^2 and RMSE values were also observed by Page model. Various studies also stated that Page model gave a best fit to describe the drying behaviour such as for star fruit slices (Hii and Ogugo, 2014).

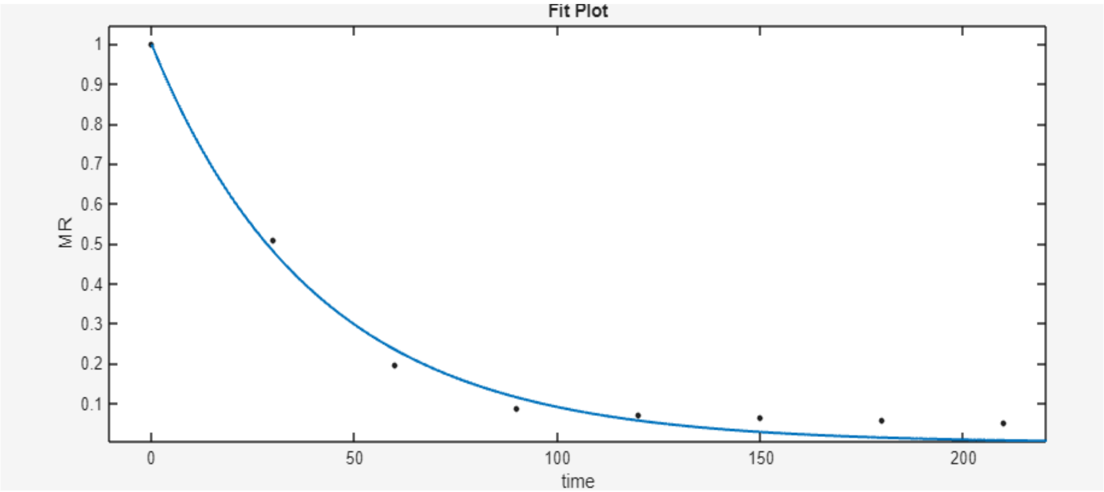
Table 4.1 showed the values of different constants of the drying models. Figure 4.2 (a) and (b) shows the plots of page model of sun dried and heat pump dried cinnamon leaf samples.

Table 4.1 Constants of drying models

Model	**Newton MR=exp(-k*x)**	**Page MR=exp(-k*x^n)**		**Henderson and Pabis MR=a*exp(-k*x)**		**Logarithmic MR=a*exp(-k*x)+C**		
Constant	k	k	n	k	a	k	a	c
Sun dried	0.678	0.0063	1.29	0.849	1	0.849	0.842	0.159
Heat pump dried	0.6787	0.025	0.98	0.849	1	0.849	0.852	0.147



(a) Sample sun dried



(b) Heat pump dried

Fig. 4.2 Plots of page model

4.2 STANDARDISATION OF THE PROCESS PARAMETERS OF THE INSTANT CONTROLLED PRESSURE DROP ASSISTED HYDRO-DISTILLATION EXTRACTION SYSTEM AND DETERMINATION OF PHYSICAL QUALITY CHARACTERISTICS OF THE CINNAMON LEAF ESSENTIAL OIL

As per the experimental design a series of experiments were performed. The three levels of steam pressures (0.5, 0.75 and 1 kg/cm^2), no. of stages of DIC treatment (1,2 and 3) and solvent-sample ratio (7,10 and 13 ml/g) were employed for the experiments as input variables. The experiments were performed as per the methodology described in Chapter III under the section 3.6. The results of the experiments conducted towards the instant controlled pressure drop assisted hydro-distillation with mean values of essential oil yield, refractive indices and energy are tabulated in Table 4.2.

The parameters were optimised through the optimisation process using Box-Behnken method of response surface methodology. Seventeen experimental data were used in the design to optimise the parameters as per response surface methodology.

Design Expert (Trial version 12.0, STAT-EASE Inc.), a statistical software was used to analyse the experimental data, for analysis of variance (ANOVA), regression coefficient calculations and for graphical analysis (response surfaces) of the experimental data.

Table 4.2. Effect of process variables towards extraction of *Cinnamomum verum* leaf essential oil

Sl. No.	**Sample**	**Essential oil yield (%)**	**Refractive index**	**Energy kW**
1.	$P_1N_1A_2$	2.86	1.3635	0.4
2.	$P_3N_1A_2$	3.23	1.363	0.367
3.	$P_1N_3A_2$	2.93	1.363	0.43
4.	$P_3N_3A_2$	3.3	1.363	0.3
5.	$P_1N_2A_1$	2.56	1.363	0.393
6.	$P_3N_2A_1$	2.5	1.362	0.35
7.	$P_1N_2A_3$	2.53	1.364	0.383
8.	$P_3N_2A_3$	3.13	1.364	0.327
9.	$P_2N_1A_1$	2.46	1.362	0.35
10.	$P_2N_3A_1$	3.06	1.364	0.333
11.	$P_2N_1A_3$	3.4	1.3655	0.317
12.	$P_2N_3A_3$	3.33	1.362	0.333
13.	$P_2N_2A_2$	2.76	1.364	0.317
14.	$P_2N_2A_2$	2.7	1.364	0.327
15.	$P_2N_2A_2$	2.76	1.365	0.317
16.	$P_2N_2A_2$	2.7	1.363	0.317
17.	$P_2N_2A_2$	2.78	1.361	0.327

4.3 EFFECT OF PROCESS PARAMETERS ON OUTPUT CHARACTERISTICS OF THE INSTANT CONTROLLED PRESSURE DROP ASSISTED HYDRO-DISTILLATION EXTRACTION SYSTEM

4.3.1 Essential Oil Yield

The essential oil yield of cinnamon leaf obtained in various combinations of experiments are shown in Table 4.2. The total yield of oil varied from 2.46 to 3.33 %. The maximum oil yield was obtained for a steam pressure of 0.75 kg/cm^2, no. of cycle of DIC treatment 3 and a solid-solvent ratio of 1:13.

A second order non-linear regression equation was utilised to relate between dependent and independent variables by using the experimental values. Following regression model was obtained to predict the yield (%) of cinnamon leaf essential oil.

Total yield of essential oil = $2.74 + 0.16A + 0.0837B + 0.2262C + 0.0000\,AB + 0.1650\,AC - 0.1675\,BC - 0.0212\,A^2 + 0.3613\,B^2 - 0.0388\,C^2$ …… (4.1)

Where A: Steam pressure (kg cm^{-2})

B: No. of cycles of DIC (N)

C: solvent solid ratio (g/ml)

The ANOVA table for the response "essential oil yield" is shown in Appendix A (Table A.5). From table A.5, it is inferred that the values of R- Squared, Adj R-Squared and Pred R-Squared for the total yield of oil were 94.99, 88.54 and 25.13% respectively. It could also be inferred that there is no significant variation in essential oil yield with variation in process parameter ($P>0.01$)

The relationship between steam pressure, no. of cycles of DIC and solid-solvent ratio on total yield of essential oil is illustrated by plotting 3D graphs representing the response surface generated by the model (Equation 4.1). The 3D responses were shown in figure 4.3.

From Figure 4.3, it can be inferred that there is an increase in yield of oil with an increase in no. of cycles of DIC. There is only slight variation in yield with increase in pressure and solid- solvent ratio. This increase in yield could be due to increased disruption of the cell walls, facilitating the release of essential oil.

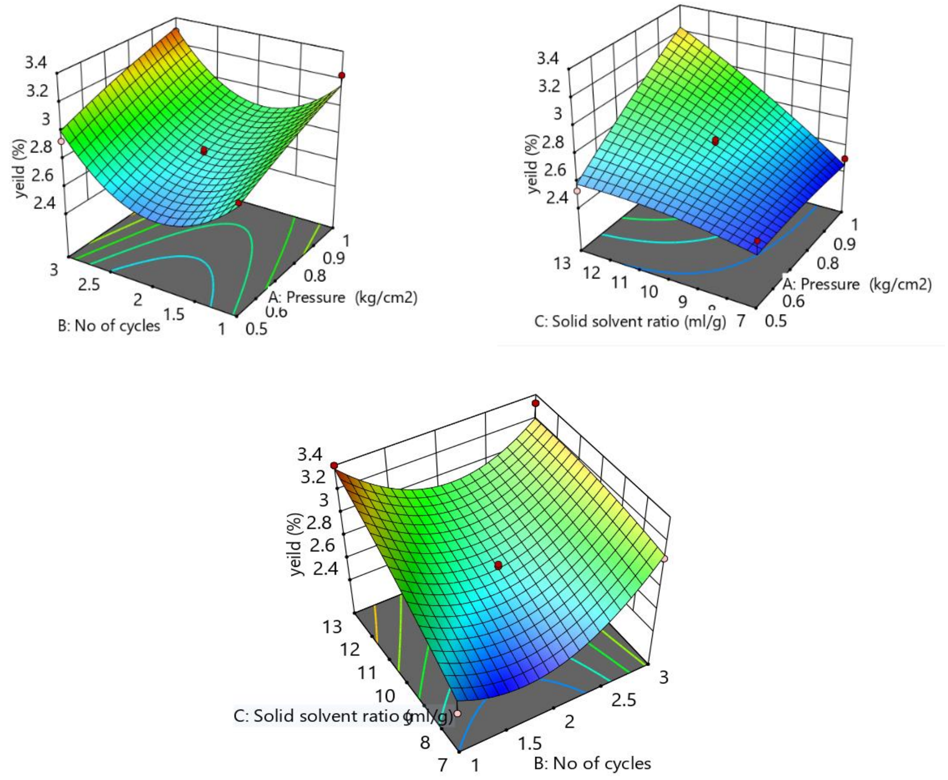


Fig. 4.3 Effect of process parameters on total yield of essential oil

4.3.2 Energy consumption

Total energy consumption of lemongrass essential oil extraction obtained in various combinations of experiments are shown in Table 4.2. Total energy consumption of cinnamon leaf essential oil extraction varied between 0.3 to 0.43 kW.

A second order non-linear regression equation was used to describe the relation between dependent and independent variables. Following regression model was obtained to predict the energy consumption for hydro-distillation of cinnamon leaf essential oil.

Total energy consumption = 0.321 – 0.0328 A - 0.0048 B - 0.0082 C - 0.0242 AB - 0.0032 AC + 0.0082 BC + 0.0416 A^2 + 0.0116 B^2 +0.0006C^2 ……(4.2)

Where A: Steam pressure (kg cm^{-2})

B: No. of cycles of DIC (N)

C: solvent-solid ratio (g/ml)

The ANOVA table for the response "Total energy consumption" is shown in Appendix A (Table A.6). From Table A.6, it may be inferred that the values of R Squared, Adj R-Squared and Pred R-Squared for the total energy consumption were 96.20, 91.33 and 47.56%, respectively.

The relationship between total energy consumption and independent variables are illustrated by plotting 3D graphs representing the response surface generated by the model (Equation 4.2). The 3D responses were shown in figure 4.4.

From figure 4.4 it could be inferred that there is no significant variation in total energy consumption with changes in process parameters. Very slight reduction in energy consumption was observed with increase in the number of cycles of DIC and solid solvent ratio.

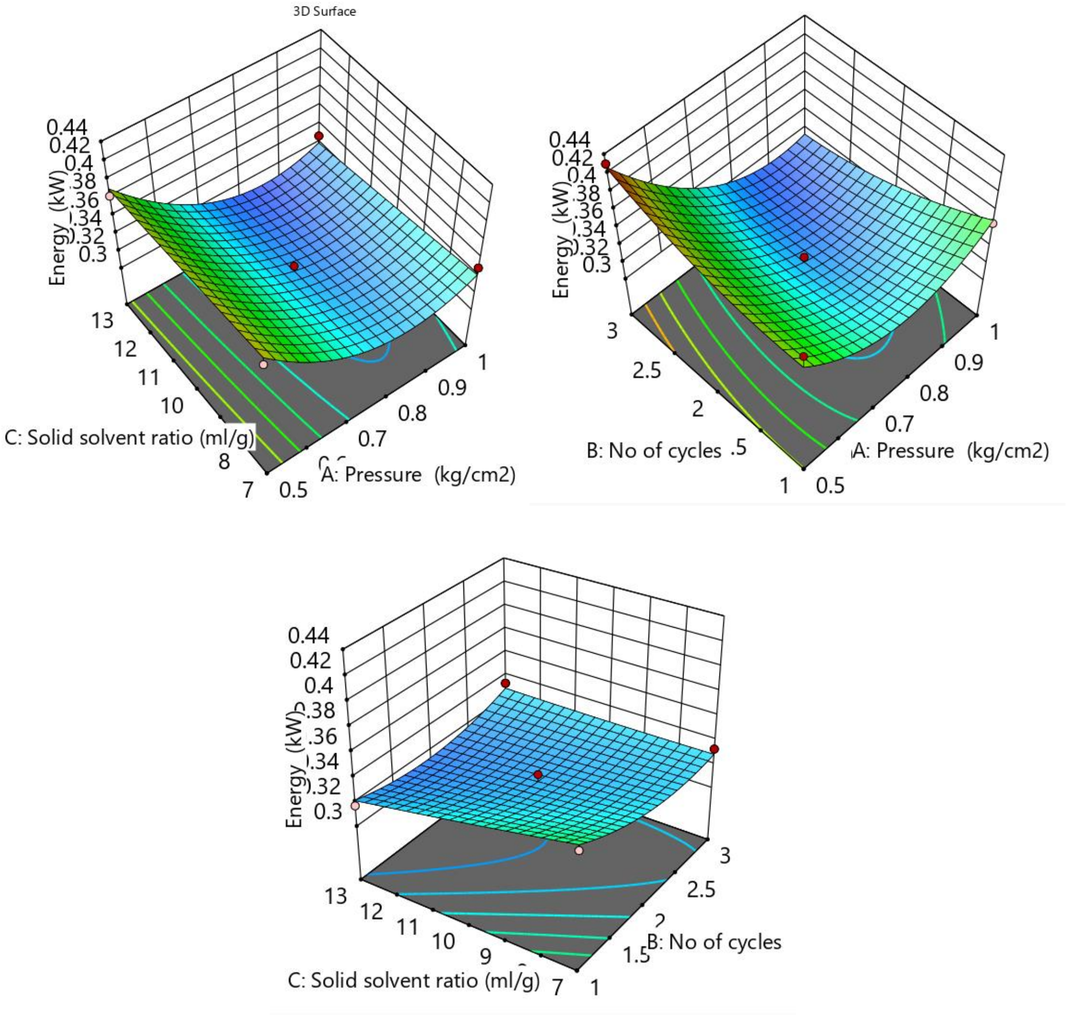


Fig. 4.4 Effect of process parameters on total energy consumption

4.4 DETERMINATION OF PHYSICAL QUALITY CHARACTERISTICS OF THE CINNAMON LEAF ESSENTIAL OIL

The physical quality characteristics of the cinnamon leaf essential oil extracted using instant controlled pressure drop assisted hydro-distillation method is listed in Table 4.2.

4.4.1 Refractive Index

The values of refractive index of cinnamon leaf essential oil obtained in various experiments are shown in Table 4.2. The values of refractive index varied from 1.361 to 1.365.

4.4.2 Specific Gravity

The specific gravity of an optimized sample of hydro-distilled cinnamon leaf essential oil was 1.077. Since the oil is denser than water, its specific gravity was greater than 1.

4.5 DETERMINATION OF CHEMICAL CONSTITUENTS

Gas Chromatography-Mass Spectroscopy technique was carried out for the analysis of volatile oil components in the cinnamon leaf essential oil extracted through instant controlled pressure drop assisted hydro-distillation.

The gas chromatograph for essential oil extracted through DIC assisted hydro distillation is shown in Figure 4.5.

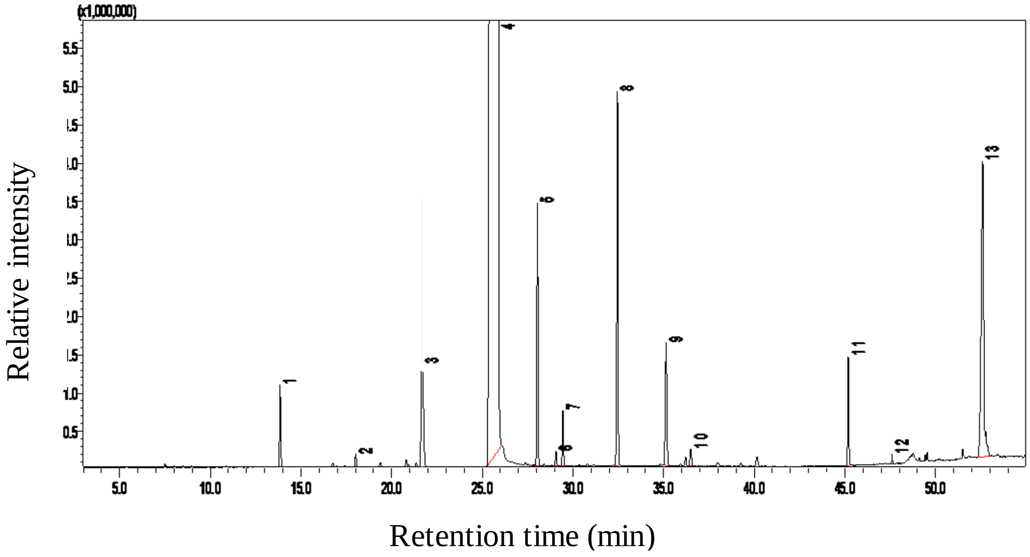


Fig. 4.5 Gas chromatograph of cinnamon leaf essential oil

From the figure 4.5, it could be clearly seen that the fourth peak in the chromatograph is the abundant component in the cinnamon leaf essential oil. The fourth peak represents the component Eugenol which is about 89.1% of the total components present in cinnamon leaves. The peak number and the respective constituents of essential oil extracted by DIC assisted hydro-distillation are shown in Table 4.3.

Table 4.3 Constituents of Essential Oil

Peak	Name of compound	Relative abundance
1	Linalool	0.45
2	L-.alpha.-Terpineol	0.07
3	2-Propenal, 3-phenyl-	1.14
4	Eugenol	89.17
5	Caryophyllene	1.44
6	Acetic acid, cinnamyl ester	0.08
7	Humulene	0.31
8	Phenol, 2-methoxy-4-(2-propenyl)-, acetate	2.19
9	Caryophyllene oxide	0.88
10	(1R,3E,7E,11R)-1,5,5,8-Tetramethyl-12-oxabicyclo[9.1.0]dodeca-3,7-diene	0.13
11	Benzyl Benzoate	0.56
12	Benzoic acid, 2-phenylethyl ester	0.04
13	2,6,10-Dodecatrien-1-ol, 3,7,11-trimethyl-	3.55

From table 4.3, it could be inferred that Eugenol was the major component identified with 89.17%. Similar results were obtained by other studies. Gas chromatographic–mass spectroscopy studies on Cinnamomum zeylanicum leaf volatile oil resulted in the identification of 19 components, which accounts for 99.4% of the total amount and the major component was eugenol with 87.3% (singh *et al.*, 2007).

The chemical constituents of Cinnomon leaf essential oil are entirely different from cinnamon bark essential oil. Major constituent present in cinnamon bark is cinnamaldehyde. But the analysed cinnamon leaf essential oil did not show any traces of cinnamaldehyde.

SUMMARY AND CONCLUSION

CHAPTER V

SUMMARY AND CONCLUSION

Essential oils (EOs) are complex aromatic liquids derived from plant parts like flowers, leaves, seeds, and roots, synthesized as secondary metabolites. They are hydrophobic and lipophilic, with a broad range of antimicrobial properties. Extraction methods are critical in determining the yield and purity of EOs. Traditional methods like hydro-distillation (HD) are widely used but have limitations such as high energy consumption and long processing times, which can degrade the quality of the oils. Modern techniques like microwave-assisted extraction (MAE) and ultrasound-assisted extraction (UAE) offer improvements but still have drawbacks.

DIC technology is an innovative method that subjects plant materials to high-pressure saturated steam, followed by an abrupt pressure drop, causing instant auto-vaporization of volatile compounds. This method enhances solvent diffusion and extraction efficiency while maintaining the quality of bioactive compounds. It is particularly effective for materials like cinnamon leaves, which have high concentrations of volatile compounds like eugenol and cinnamaldehyde.

Cinnamon, derived from the bark and leaves of Cinnamomum species, has been used in traditional medicine and culinary applications for centuries. It has significant antimicrobial, insecticidal, and medicinal properties. The essential oil from cinnamon leaves, rich in eugenol, exhibits strong antifungal and antioxidant activities, making it a potential green pesticide and antimicrobial agent.

The study aims to:

1. Optimize the parameters of the DIC method for maximum yield of essential oil from cinnamon leaves.

2. Analyze the physico-chemical characteristics of the extracted oil.

The research involves a study of essential oil extraction from cinnamon leaves using DIC-assisted hydro-distillation. Key parameters such as steam pressure, no. of times of DIC treatment and solvent sample ratio are optimized to achieve the highest yield and quality of the essential oil. The extracted oils are then analyzed for their chemical composition and physical properties.

Two drying methods, sun drying and heat pump drying, were compared to optimize the drying process. The drying kinetics of the cinnamon leaves were best described by the Page model, which showed a higher coefficient of determination (R^2) for sun-dried samples and heat pump-dried samples, indicating excellent fit and minimal error.

The study found that DIC-assisted hydro-distillation shows slight improvement in the yield and quality of essential oils compared to traditional hydro-distillation. The rapid pressure drop in the DIC process enhances the swelling of plant materials, leading to higher diffusivity and better extraction efficiency. The essential oils extracted using DIC technology showed higher concentrations of eugenol, indicating superior antifungal and antioxidant properties.

DIC-assisted hydro-distillation is a promising technique for the extraction of high-quality essential oils from cinnamon leaves. It offers several advantages over traditional methods, including higher yields, reduced energy consumption, and preservation of bioactive compounds. This method has potential applications in the food, pharmaceutical, and agricultural industries for producing essential oils with enhanced therapeutic and preservative properties.

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CHAPTER VI

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APPENDIX

APPENDIX A

Table A.1 Data of sun dried leaves

Time (min)	Initial Moisture Content (wb)	Initial Moisture Content (db)	Wt. of sample (g)	Moisture (g)	Dry matter (g)	Mt (db)	M.R
0	53.82	1.165	100	53.82	46.18	1.1654	1
30	53.82	1.165	78.4	32.22	46.18	0.69770	0.59866
60	53.82	1.165	62	15.82	46.18	0.34257	0.29394
90	53.82	1.165	52.9	6.72	46.18	0.14551	0.12486
120	53.82	1.165	47.8	1.62	46.18	0.03508	0.03010
150	53.82	1.165	47.4	1.22	46.18	0.02641	0.02266
180	53.82	1.165	47.1	0.92	46.18	0.01992	0.01709
210	53.82	1.165	47.1	0.92	46.18	0.01992	0.01709

Table A.2 Data of heat pump dried leaves

Time (min)	Initial Moisture Content (wb)	Initial Moisture Content (db)	Wt. of sample (g)	Moisture (g)	Dry matter (g)	Mt (db)	M.R
0	53.82	1.165	100	53.82	46.18	1.16544	1
30	53.82	1.165	73.5	27.32	46.18	0.591598	0.507618
60	53.82	1.165	56.7	10.52	46.18	0.227804	0.195466
90	53.82	1.165	50.9	4.72	46.18	0.102209	0.0877
120	53.82	1.165	49.9	3.72	46.18	0.080554	0.069119
150	53.82	1.165	49.6	3.42	46.18	0.074058	0.063545
180	53.82	1.165	49.3	3.12	46.18	0.067562	0.057971
210	53.82	1.165	48.9	2.72	46.18	0.0589	0.050539

Table A.3 Goodness of fit of cinnamon leaf sample sun dried

Model	Newton	Page	Henderson and Pabis	Logarithmic
SSE	0.4624	0.0007	0.4624	0.2881
R-square	0.4912	0.9992	0.4912	0.6829
DFE	7.0000	6.0000	6.0000	5.0000
Adj R^2	0.4912	0.9990	0.4064	0.5561
RMSE	0.2570	0.0111	0.2776	0.2401

Table A.4 Goodness of fit of cinnamon leaf sample heat pump dried at 55°C

Model	Newton	Page	Henderson and Pabis	Logarithmic
SSE	0.3183	0.0083	0.318	0.1662
R-square	0.6032	0.9897	0.6032	0.7929
DFE	7.0000	6.0000	6.0000	5.0000
Adj R^2	0.6032	0.9880	0.5371	0.7100
RMSE	0.2132	0.0371	0.2303	0.1823

Table A.5 The ANOVA table for the response "Essential Oil yield".

Source	Sum of squares	df	Mean square	F-value	P-value	
Model	1.44	9	0.1603	14.74	0.0009	significant
A-Pressure	0.2048	1	0.2048	18.83	0.0034	
B-No of cycles	0.0561	1	0.0561	5.16	0.0573	
C-Solid solvent ratio	0.4095	1	0.4095	37.66	0.0005	
AB	0.0000	1	0.0000	0.0000	1.0000	
AC	0.1089	1	0.1089	10.01	0.0158	
BC	0.1122	1	0.1122	10.32	0.0148	
A^2	0.0019	1	0.0019	0.1748	0.6884	
B^2	0.5495	1	0.5495	50.53	0.0002	
C^2	0.0063	1	0.0063	0.5814	0.4707	
Residual	0.0761	7	0.0109			
Lack of Fit	0.0705	3	0.0235	16.79	0.0099	significant
Pure Error	0.0056	4	0.0014			
Cor Total	1.52	16				

Table A.6 The ANOVA table for the response “Total energy consumption”.

Source	Sum of squares	df	Mean square	F-value	P-value	
Model	0.0201	9	0.0022	19.72	0.0004	significant
A-Pressure	0.0086	1	0.0086	75.65	< 0.0001	
B-No of cycles	0.0002	1	0.0002	1.59	0.2475	
C-Solid solvent ratio	0.0005	1	0.0005	4.80	0.0646	
AB	0.0024	1	0.0024	20.74	0.0026	
AC	0.0000	1	0.0000	0.3725	0.5609	
BC	0.0003	1	0.0003	2.40	0.1653	
A^2	0.0073	1	0.0073	64.32	< 0.0001	
B^2	0.0006	1	0.0006	5.02	0.0601	
C^2	1.645E-06	1	1.645E-06	0.0145	0.9075	
Residual	0.0008	7	0.0001			
Lack of Fit	0.0007	3	0.0002	7.49	0.0406	significant
Pure Error	0.0001	4	0.0000			
Cor Total	0.0209	16				

ABSTRACT

INSTANT CONTROLLED PRESSURE DROP ASSISTED EXTRACTION OF ESSENTIAL OIL FROM CINNAMON LEAVES

By

AMEERA C (2020-02-006)
NAVYA PRAVEEN K (2020-02-021)
ALEX K SANI (2020-02-026)
AKASH BALAKRISHNAN (2020-02-041)

ABSTRACT OF THESIS

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DEPARTMENT OF PROCESSING AND FOOD ENGINEERING

KELAPPAJI COLLEGE OF AGRICULTURAL
ENGINEERING AND TECHNOLOGY
TAVANUR –679573, MALAPPURAM
KERALA, INDIA
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ABSTRACT

This study aims to optimize the extraction parameters to maximize the essential oil yield from *Cinnamomum verum* leaves using the Instant Controlled Pressure Drop (DIC) pre-treatment technique followed by hydro-distillation. The study encompasses various aspects, including drying characteristics, extraction efficiency, and quality of the essential oil. Two drying methods, sun drying and heat pump drying, were compared to optimize the drying process. The experimental design involved a Box-Behnken design to evaluate the effects of three independent variables: pressure (0.5, 0.75, 1 kg/cm^2), number of cycles (1,2,3), and solid-solvent ratio (7,10,13 mL/g) on the yield and quality of essential oil. The results indicated slight effects of pressure and the number of cycles on the extraction yield. Maximum yield obtained was approximately 3.3%. Gas chromatography-mass spectrometry (GC-MS) analysis identified the main constituents of the essential oil as eugenol with 89 % of total constituents. Initial moisture content of the cinnamon leaves was recorded at 53.82% (wb). Post-drying, the final moisture content was found to be 0.92% (wb) for sun-dried samples and 2.72% (wb) for heat pump-dried samples. The drying kinetics of the cinnamon leaves were best described by the Page model, which showed a higher coefficient of determination (R^2) of 0.9992 for sun-dried samples and 0.9897 for heat pump-dried samples, indicating excellent fit and minimal error. The findings indicate that further exploration of other plant materials using the DIC pre-treatment method may enhance extraction efficiency and improve product quality.