# ALOEVERA, PECTIN, CITRIC ACID, ESSENTIAL OIL BASED FILM FOR FOOD PACKAGING

A dissertation

Submitted in partial fulfillment of the requirements for the award of the degree of

# **MASTER OF SCIENCE**

In

### **CHEMISTRY**

**Submitted by:** 

# ANAMIKA KUMARI (2K23/MSCCHE/73) and NEHA GUPTA (2K23/MSCCHE/27)

Under the supervision of

DR. DEENAN SANTHIYA



## DEPARTMENT OF APPLIED CHEMISTRY

DELHI TECHNOLOGICAL UNIVERSITY
(Formerly Delhi College of Engineering)
Bawana Road, Delhi – 110042

JUNE, 2025



## **CANDIDATE'S DECLERATION**

We, Neha Gupta (2k23/MSCCHE/27) and Anamika
Kumari(2k23/MSCCHE/73), the students of M.Sc. Chemistry,
hereby declare that the work presented in the thesis, entitled
"Aloevera, Pectin, Citric acid, Essential oil based Film for Food
Packaging", in partial fulfillment of the requirements for the
award of the Master of Science degree, submitted in the
Department of Applied Chemistry, Delhi Technological
University is an authentic record of my own work carried out
under the supervision of Dr. Deenan Santhiya.

The matter presented in the thesis has not been submitted by us for the award of any other degree of this or any other Institute.

Place: Delhi Neha Gupta (2k23/MSCCHE/27)

Date: 20/06/2025 Anamika Kumari (2k23/MSCCHE/73)



## **DELHI TECHNOLOGICAL UNIVERSITY**

(Formerly Delhi College of Engineering) Shahbad Daulatpur, Main Bawana Road, Delhi-42

## **CERTIFICATE**

I, hereby certify that the dissertation titled "Aloevera, Pectin, Citric acid, Essential oil based Film for Food Packaging", which is submitted by ANAMIKA KUMARI(2K23/MSCCHE/73) and NEHA GUPTA(2K23/MSCCHE/27) to the Department of Applied Chemistry, Delhi Technological University, Delhi in partial fulfillment of the requirement for the award of Master of Science. This dissertation represents results of original work, and studies are carried out by the students themselves and the contents of this dissertation do not form the basis for the award of any other degree to the candidates or to anybody else from this or any other University/Institution.

Place: Delhi DR.DEENAN SANTHIYA SUPERVISOR

Date: 20/06/2025

# **ACKNOWLEDGEMENT**

The success and final outcome of this project required a lot of guidance and assistance from many people and we are extremely fortunate to have got this all along the completion of this project work.

We wish to express our gratitude towards our project supervisor, Dr. Deenan Santhiya, Department of Applied Chemistry, Delhi Technological University, who provided us a golden opportunity to work under her guidance. Her scholastic guidance and sagacious suggestions helped us to complete the project on time.

We wish to thank Prof. Anil Kumar, Head of the Department of Applied Chemistry, Delhi Technological University for his constant motivation. We are thankful and fortunate enough to get constant encouragement, support and guidance from all teaching staff of Department of Applied Chemistry, which helped us in successfully completing our project work.

We are also thankful to PhD scholar Ms. Krizma khatreja for her constant support and motivation..

Finally, yet importantly, we would like to express our heartfelt thanks to our beloved family and friends who have endured our long working hours and whose motivation kept us going.

ANAMIKA KUMARI

**NEHA GUPTA** 

## **ABSTRACT**

The demand for environmentally friendly packaging solutions to replace plastic is currently on the rise. Eatable or biodegradable films made from common substances such as proteins, polysaccharides, and lipids have surfaced as reasonable substitutions for non-biodegradable bundling. Films inferred from pectin, aloe vera, citric acid, and essential oils were made employing a casting strategy. Pectin-based Film show great filmforming capacities, mechanical quality, and viable obstructions against dampness and gasses. Aloe vera gel, basically composed of water, serves as a normal antibacterial specialist. Much appreciated to its antioxidant and antimicrobial properties, aloe vera gel is an perfect candidate for improving the rack life of nourishment items. The joining of aloe vera gel emphatically influences the mechanical and physicochemical properties of the films, increasing their pliable quality. Essential oils serve a crucial function as added substances within the creation of renewable dynamic bundling with improved execution. The affect of these basic oils on the relationship between structure and properties—including physicochemical and antimicrobial characteristics—is completely investigated. The prepared Films were characterized utilizing Fourier change infrared spectroscopy (FTIR), X-ray diffraction (XRD), and field outflow filtering electron microscopy (FE-SEM). This study illustrates the potential of composite films made from pectin, aloe vera, citric acid, and essential oils as biodegradable alternatives to synthetic plastics for food packaging.

# **CONTENT**

<b>♦</b>	Candidate Declaration	ii
<b>*</b>	Supervisor Certificate	iii
<b>*</b>	Acknowledgement	iv
<b>*</b>	Abstract	v
<b>*</b>	List of Abbreviation	viii
<b>*</b>	List of Table	ix
<b>♦</b>	List of Figure	ix

#### 1. CHAPTER: INTRODUCTION AND LITERATURE REVIEW

- 1.1 Introduction of Food Packaging
- 1.2 Role of Food Packaging
- 1.3 Aloevera in food packaging film
- 1.4 Pectin in food packaging film
- 1.5 Cross-linker in food packaging film
  - 1.5.1 Citric acid as cross-linker
- 1.6 Essential oil in food packaging film
  - 1.6.1 Ginger essential oil in food packaging film
    - 2. CHAPTER: MATERIAL AND METHODS
- 2.1 Raw Materials
- 2.2 Extraction of AV Gel
- 2.3 Film formation methods

- 3. CHAPTER: CHARACTERISATION AND RESULT
- 3.1 Fourier Transform Infrared Spectroscopy(FTIR) Analysis
  - 3.1.1 Key Applications
  - 3.1.2 Benefits
  - 3.1.3 FTIR Instrumentations
- 3.2 X-Ray Diffraction (XRD) Analysis
  - 3.2.1 Intro
  - 3.2.2 Benefits of Food packaging
  - 3.2.3 Application of X-ray & Bragg's Law
- 3.3 FESEM With EDX Analysis
  - 4. CHAPTER: CONCLUSION
  - 5. CHAPTER: REFERENCE

# **List of Abbreviations:**

• AV Aloevera

• Pec Pectin

CA Citric acidEO Essential Oil

• GEO Ginger essential oil

• CLG Cross-Linking

• BFPF Biopolymer food packaging film

• EO-AP Essential oil-antibacterial packaging

• ABTS 2,2-azido-bis-3-ethylbenzothiazoline-6-sulfonic acid

• DPPH 2,2-diphenyl-1 pyridylhydrazide

# **List of Tables:**

Table 1	Comparison of conventional plastic food packaging
	materials with biopolymer- based materials.
Table 2	Major chemical compounds of Aloe Vera.
Table 3	Lists the specific microorganisms involved in its production.
Table 4	Major components of common GEO and their contents.
Table 5	Effects of GEO on properties of biodegradable food
	packaging films
Table 6	eZAF Smart Quant Results

# **List of Figures:**

FIG.1	Beneficial properties of Biodegradable film
<b>FIG.2(A)</b>	Functional properties of AV
<b>(B)</b>	Phytochemical constituents of AV
FIG.3	Various products from AV and their usages
FIG.4	Structure of pectin
FIG.5	Citric Acid structure (A) molecular and (B)Skeletal
FIG.6	A predictive cross-linking reaction of citric acid with cellulose
FIG.7	The possible action mechanisms of EOs
FIG.8	Flow chart of film preparation method
FIG.9	Methods and material of film preparation
<b>FIG.10</b>	Instrumentation of FTIR
<b>FIG.11</b>	FTIR Spectra of (a) AV (b) CA (c) EO (GEO) (d) Pec
	(e)WithEO Film and (f) without EO Film
<b>FIG.12</b>	Laue method diagram XRD
<b>FIG.13</b>	Powder method diagram XRD
<b>FIG.14</b>	XRD Spectra of (a)Pec (b)CA (c) Film withEO (d) Film without
	EO
<b>FIG.15</b>	FESEM/Morphology image of film without EO
<b>FIG.16</b>	FESEM/Morphology image of film with EO
<b>FIG.17</b>	EDX images of without EO
<b>FIG.18</b>	Sum Spectrum

## 1. CHAPTER: INTRODUCTION

#### 1.1 INTROUCTION OF FOOD PACKAGING

Food packaging plays a big role in keeping our food fresh and safe as it travels from where it's made to where it's eaten—even if that's on the other side of the world. It helps food last for days, weeks, or even months after it's produced. The primary purpose of packaging materials is to shield food from contaminants and extend its shelf life. Plastics are frequently used in food packaging because of their versatility; they are fluid, moldable, heat-sealable, easy to print, and seamlessly integrated into production processes. However, most plastic packaging is discarded within the same year, which leads to significant environmental issues because it is not biodegradable. The short lifespan, combined with the large volume of packaging material and low recyclability of plastics in many regions, results in a substantial solid waste problem. This can be especially concerning when plastics are not appropriately collected by waste management systems and end up in aquatic ecosystems causing harm. The accumulation of plastic waste in the environment is a growing concern because increased plastic consumption is linked to serious environmental and health issues. Currently, waste and recycling infrastructure is unable to keep pace with the increasing volume of endof-life plastics. The surge in disposable packaging is a major contributor to waste problems because plastic is prevalent in consumer products, including single-use plastic packaging—like the kind used for snacks, takeout, and grocery items. These are often discarded carelessly by consumer, adding to the global waste stream.

<u>Table 1</u>
Comparison of conventional plastic food packaging materials with biopolymer- based materials.

Item	Specific materials	Advantages	Disadvantage
Plastic	Polypropylene;	Good barrier properties;	Non-renewable;
	Polyethylene;	Mechanical properties;	Not biodegradable;
	Polystyrene;	Hydrophobicity;	Environmental
	Polyethylene	Economy; Convenience;	pollution; Lack of
	terephthalate	Mature technology for	antibacterial and
		industrial production.	antioxidant properties;
			Health hazards of
			microplastic migration
			and residues.
Biopolymer	Polysaccharides	Renewable;	Poor mechanical
	(chitosan, alginate,	Biodegradable;	and barrier

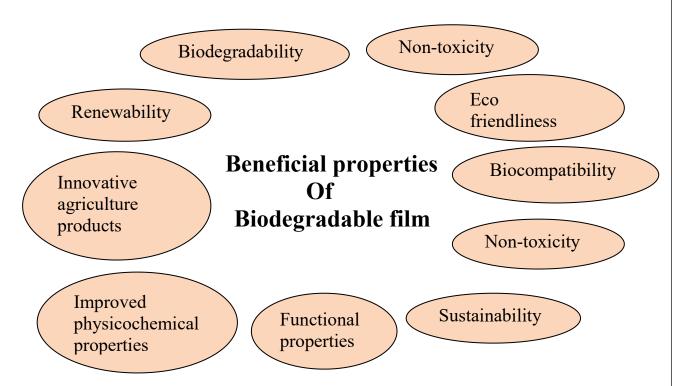
	1 1		1		. •	
Iа	h	e		con	tın	mes

Item	Specific materials	Advantages	Disadvantages
	pectin,starch etc) Proteins (gelatin, whey protein, zein, etc.) Lipids (beeswax) Biopolyesters (Polylactic acid; Polyvinyl alcohol)	Green; Safe; Antioxidant	properties; Difficul- to produce industrially.

Currently the demand for eco Friendly packaging materials to replace plastics is increasing. Edible or biodegradable packaging films prepared from natural compounds such as proteins polysaccharides and lipids have emerged as alternative do non-biodegradable packaging material. Fruit and vegetable waste has potential as a bio plastic material promoting environmental sustainability. By products of fruits and vegetables is it used in the production of biodegradable films are gaining popularity due to rich source of bio active compound environment friendly sustainable green development cheapness and producing biopolymers as an alternative to synthetic plastics polymers. Films incorporating plant extracts into polymers usually resulted in modified physiochemical, mechanical and barrier antioxidant and antimicrobials properties compared to films made of individual components. Edible films are thin, consumable layers that serve as protective barriers, limiting the movement of moisture, oxygen, and solutes in food products. These films can either coat the entire surface of a food item or be placed between different food components to maintain quality and separation (Guilbert, 1986). They can be utilized as coordinate nourishment coatings or made as standalone sheets, with extra potential to act as boundaries to gas and smell exchange (Kester and Fennema, 1986). In recent years, edible films and coatings have attracted attention due to their eco-friendly benefits compared to traditional plastic packaging. A major advantage is that these films are edible and leave no waste, contributing to environmental sustainability. Even if not consumed, they biodegrade more easily than synthetic materials since they are derived from natural, renewable sources. Furthermore, edible films can improve the sensory attributes of foods when enhanced with flavorings, colors, or sweeteners. They are particularly useful for packaging small, perishable food items that are usually not packaged individually—such as berries, nuts, and beans. These films can too be joined inside layered nourishment items to avoid dampness and solute exchange between layers, making a difference keep up surface and quality in things like pies, candies, and pizzas. Additionally, edible films can be infused with antimicrobial and antioxidant substances, making them effective in

extending shelf life. They can control the release rate of preservatives from the food's surface into its interior. Moreover, edible films can be integrated into multi-layer packaging systems as the food-contact layer, combined with non-edible materials for added strength. Although they are environmentally sustainable and produce less waste, edible films currently lack the mechanical strength and barrier effectiveness of synthetic packaging. Therefore, continued research is essential to improve their composition, production techniques, and functionality in the food industry. A biodegradable film is a type of primary packaging composed mainly of biodegradable polymers, especially polysaccharides. These films provide numerous advantages over traditional artificial packaging, specifically their capacity to hold product and enlarge the shelf lifestyles of minimally processed foods, all while being environmentally. Therefore the current advancements in biodegradable film technology, with a focus on the types of polysaccharides used in their production. It also outlines the essential properties these films must possess to function effectively. Additionally, blending polysaccharides with plasticizing agents can influence the performance of biodegradable films, either positively or negatively.

Fig.1 Beneficial properties Of Biodegradable film



Because of all this, people are paying more attention to how food is packaged. There's a growing interest in finding eco-friendly alternatives that can reduce both food waste and environmental damage. The development of biodegradable and edible films has emerged as a sustainable alternative to conventional plastic-based packaging. Several natural materials can be used to produce edible/biodegradable films and coatings, offering improved food preservation and safety. One promising solution is biodegradable edible films—packaging

made from natural materials that are safe for both food and the planet.

#### 1.2 ROLE OF FOOD PACKAGING

Food packaging plays a vital role in maintaining the quality, safety, and shelf life of food products from the point of manufacture to the moment of consumption. It acts as a protective barrier against external factors such as moisture, air, light, microorganisms, and physical damage, which can compromise the integrity and edibility of food. By preventing contamination and spoilage, packaging helps ensure food safety and reduces the risk of foodborne illnesses. Additionally, packaging supports efficient transportation and storage by providing structural stability and enabling bulk handling without damaging the product. It also serves as an important medium of communication between the manufacturer and the consumer, offering critical information such as nutritional facts, ingredients, allergen warnings, manufacturing and expiry dates, usage instructions, and branding. Modern food packaging often incorporates innovative technologies such as modified atmosphere packaging and active packaging systems, which further enhance food preservation capabilities. Beyond its functional aspects, packaging serves as a marketing tool, providing essential product information and attracting consumers through design elements. As sustainability concerns grow, the food packaging industry is increasingly focusing on developing ecofriendly materials and recyclable solutions to minimize environmental impact.

Thus, food bundling isn't just about sticks and guarantees; it also instructs, markets, and contributes to the broader objective of environmental sustainability.

#### 1.3 ALOVERA IN FOOD PACKAGING

Aloe Vera is a medicinal plant of significant therapeutic value that has been widely studied for its diverse applications in the food, pharmaceutical, and cosmetic industries. The inner gel of Aloe vera consists of soft, mucilaginous tissue made up primarily of parenchyma cells. This gel is a transparent, jelly-like substance comprising a complex mixture of bioactive compounds. It contains carbohydrates, proteins, dietary fibers, soluble sugars, essential vitamins (such as A, C, E, and B-complex), minerals (including calcium, magnesium, and zinc), amino acids, organic acids, and various phenolic compounds. Although Aloe vera gel is composed mostly of water—up to 99%—the remaining ~1% includes a potent mix of biologically active substances such as aloin, emodin (from the anthraquinone group), flavonoids, saponins, and aloe-mannan. These

compounds are largely responsible for the gel's antibacterial, antifungal, and anti-inflammatory properties.

Additionally, Aloe vera exhibits strong antioxidant activity, which can help neutralize free radicals and delay oxidation in food systems. Aloe vera gel acts as a natural preservative, preventing microbial growth and spoilage, thereby extending food product shelf life. Beyond its preservative potential, it also contributes to enhancing the nutritional value of food when used as an ingredient or coating and with its non-toxic, biodegradable, and edible properties; Aloe vera gel is an ideal candidate for sustainable food packaging and natural preservation systems.

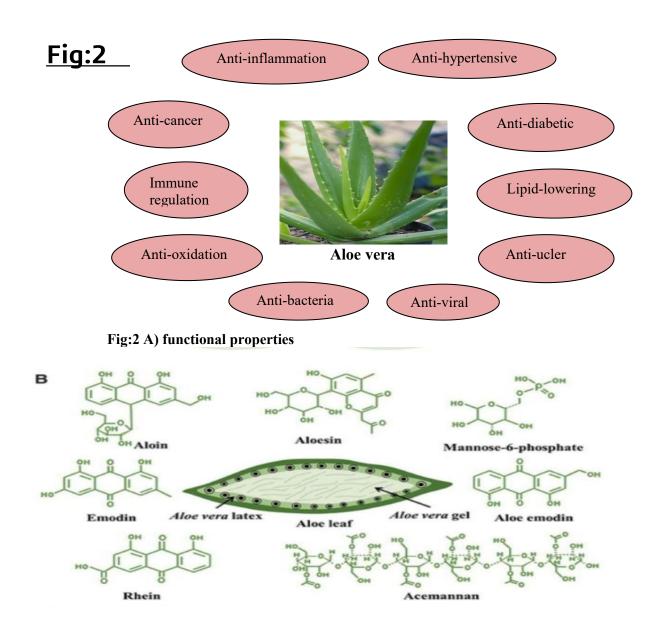


Fig: 2 B) phytochemical constituents

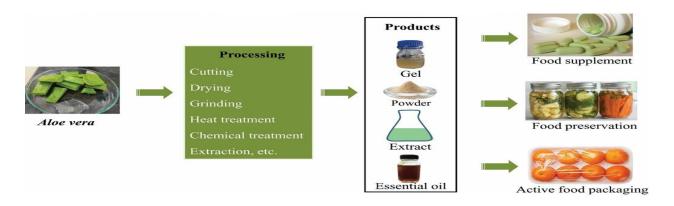


Fig. 3. Various products from AV and their usages.

# Table 2

Major chemical compounds of Aloe Vera.

Class of compounds	Chemical constituents
A .1	
Anthraquinones	Aloin A, aloin B, aloe-emodin, anthranol, isobarbaloin,
	emodin, aloetic-acid, ester of cinnamic acid
Carbohydrates	Mannose, mannan (pure and acetylated), acetylated
	glucomannan, galactogalacturan, glucogalactomannan,
	galactoglucoarabinomannan, pectic substance, galactan,
	arabinogalactan, xylan, L-rhamnose, aldopentose
Proteins	Lectins, lectin-like substance, glycoproteins
Vitamin	B-complex vitamins, vitamin A, vitamin C, vitamin E, and
	choline
Chromones	8-C-glucosyl-(2 '-O-cinnamoyl)-7-O- methylaloediol A, 8-C-
	glucosyl-7-O-methyl- (S)-aloesol, 8-C-glucosyl-(S)- aloesol,
	8-C- glucosyl-7-O-methylaloediol, 8-C-glucosyl- noreugenin,
	isorabaichromone, isoaloeresin D, and neoaloesin A
Enzymes	Alkaline phosphatase, catalase, amylase, cyclooxidase,
	cyclooxygenase, lipase, phosphoenolpyruvate carboxylase,
	oxidase, carboxypeptidase, and superoxide dismutase
Inorganic compounds	Ca, Cl, Cu, Mn, Ma, Na, P, Zn, and K
Amino acids	All essential amino acids, aspartic acid, glutamic acid,
	hydroxyproline, proline, tyrosine
Organic compounds,	Triglycerides, triterpenoid, gibberellin,
lipids, and others	$\gamma$ -linolenic acid, cholesterol, $\beta$ -sitosterol), potassium sorbate,
	salicylic acid, and arachidonic acid

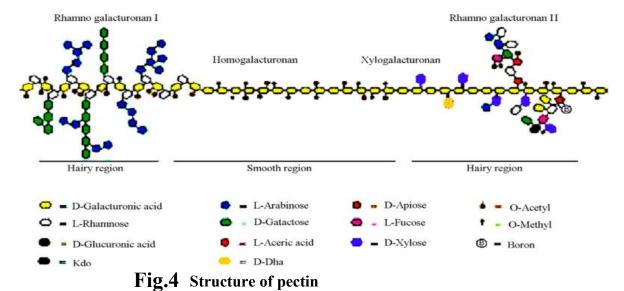
#### 1.4 PECTIN IN FOOD PACKAGING FILM

Among various biopolymers, Pectin stands out as a promising biopolymer option due to its abundance and impressive physical and functional properties. Pectin is a naturally occurring polysaccharide primarily sourced from plants, composed of units of D-galacturonic acid linked through  $\beta$ -(1-4) bonds, along with sugars like galactose and rhamnose. This complex carbohydrate is structurally diverse; its sugar units may have carboxylic groups that are partly methylated or neutralized using basic substances. When polygalacturonic acid carries numerous methyl ester groups, it is referred to as pectinic acid. If these methyl groups are absent, it is called pectic acid. Pec, in general, refers to water-soluble forms of pectinic acid with variable methylation and neutralization levels that can gel in the presence of specific amounts of sugars and acids.

Numerous studies have confirmed that pectin is one of the most prevalent polysaccharides found in the primary cell wall and middle lamella of plant tissues, Pectin is obtained through two main sources:

- 1. **Commercial sources** These include citrus peels, apple pomace, pomegranate peel, mango and lemon peels, sugar beet pulp, and potato pulp. These materials are widely used in industrial-scale pectin extraction due to their high availability and pectin content.
- 2. **Non-commercial sources** These include agricultural by-products such as cocoa husks, bark from mulberry branches, peach processing waste, sisal residues, pumpkin and banana peels, watermelon rind, and soybean hulls. These are less commonly used but represent sustainable alternatives for pectin extraction from food industry waste and underutilized biomass.

Pec are usually classified based on their methoxyl content: high methoxyl pectins (more than 50% esterified) and low methoxyl pectins (less than 50%). High methoxyl types require sugar and acidic conditions to form gels, whereas low methoxyl pectins can gel using divalent metal ions like calcium, even without added sugar. In commercial applications, high methoxyl pectins are more commonly used.



In the food industry, pec plays a vital role as a natural biopolymer used for thickening, gelling, emulsifying, and stabilizing food products. It can also be processed into edible films or coatings that act as a protective barrier around food. Pec-based materials are increasingly valued for their biodegradability. Furthermore, and pectin antioxidant properties, and when supplemented with bioactive agents, it can also provide antimicrobial benefits. These functional improvements enhance the potential of pectin-based packaging to extend food shelf life. The antioxidant capacity of pec is linked to its molecular structure, especially the presence of hydroxyl, methoxy, and carboxyl groups found in its galacturonic acid units. Additionally, lower molecular weight pectin fragments tend to have greater antioxidant activity due to more reactive end groups. Impurities such as proteins and polyphenols, often coextracted with pectin, may also contribute positively to its antioxidant effects.

Despite numerous studies on pectin's extraction and structural modifications, relatively few focus on its use in packaging materials. Recent research, however, has explored the development of pectin-based films using agricultural waste and plant extracts for enhanced food preservation and intelligent packaging applications. Some studies also highlight pectin's medical and health-related applications. However, further investigation is needed into the sources of pectin, its extraction processes, and the functional properties of the resulting material specifically for food packaging uses.

Pectin is a structurally sophisticated polysaccharide found predominantly in the cell walls of higher plants, particularly dicotyledons. It plays a crucial role in maintaining cell structure and supporting various physiological processes during plant growth and development. Found in high concentrations in fruits and vegetables, pectin typically comprises 35–40% of the primary cell wall in dicot plants. Its molecular configuration is highly variable, as it consists of linear and branched chains of hundreds to thousands of sugar units, preventing the assignment of a uniform structure.

The primary structural domains of pectin include homogalacturonan (HG), rhamnogalacturonan I (RG-I), and rhamnogalacturonan II (RG-II). These domains may carry additional side chains composed of neutral sugars such as arabinose, galactose, and xylose, forming branched structures like arabinogalactan I and II (AG-I and AG-II), as well as xylogalacturonan (XGA).

The solubility of pectin in water largely depends on factors such as its polymer chain length and the extent of its methyl esterification. While pectin and its salts formed with monovalent ions typically dissolve well in water, those associated with di- or trivalent cations are generally insoluble. When pectin is finely powdered, it tends to absorb water quickly but often

clumps together, which delays complete dissolution. Upon dissolving, pectin molecules can undergo breakdown through de-esterification and depolymerization, particularly in acidic environments and at high temperatures. Under such conditions, the reduced ionization of carboxylic acid groups leads to decreased electrostatic repulsion between molecules, thereby promoting gel formation. Heat treatment may further cause  $\beta$ -elimination, a reaction that fragments the polymer chains and weakens their gel-forming ability. Although smaller pectin fragments dissolve more easily, they exhibit limited interaction with the plant cell wall structure.

Pectin-Based Edible Coatings for Food Packaging and Preservation: Food packaging has evolved from merely containing and transporting food to enhancing shelf life and maintaining product quality. With growing demand for eco-friendly and functional packaging, biodegradable polymers like poly(lactic acid), PHAs, starch, and especially natural polysaccharides such as pectin, have gained prominence. Pectin is a popular choice for edible films and coatings because of its sustainable origins, flexible properties, and effectiveness in blocking moisture, oxygen, and oils. Low methoxyl (LM) pectin forms strong gels with calcium ions at low pH, making it effective for coatings that extend fruit shelf life. For instance, pectin films have been shown to preserve avocados and fresh-cut melons by reducing respiration and moisture loss. Custom formulations using pectin and other additives like glycerol, citric acid, or essential oils can further enhance protection against microbial spoilage and maintain sensory qualities. Furthermore, pairing pectin-based coatings with modified atmosphere packaging (MAP) enhances the quality of fresh-cut fruits, such as persimmon, by providing an additional layer of protection. Pectin-based pre-treatments also play a role in preserving the nutrient content of dried fruits, including pineapple and papaya. When used in frying, pectin-based coatings effectively limit oil absorption in foods such as potato chips and French fries, resulting in improved texture and enhanced nutritional value.

Overall Pectin-based coatings provide a sustainable solution for enhancing food safety, prolonging shelf life, and preserving food quality across different processing techniques, making them an eco-friendly option for the food industry.

#### 1.5 CROSS-LINKER IN FOOD PACKAGING FILM

Green cross-linking technology significantly improves the functionality of bio-based films and coatings, with citric acid gaining attention as a widely used natural cross-linking agent. Previous study shows that the chemical characteristics of citric acid and its incorporation into various bio-based film materials are explored, with particular focus on how it contributes to extending the shelf life of food products. The cross-linking efficiency of citric acid differs among various biopolymers—such as polysaccharides, proteins, and biopolyesters—and is affected by

parameters like citric acid concentration, temperature, and pH. In general, citric acid enhances both the structural performance and preservation potential of bio-based films and coatings.

#### 1.5.1 CITRIC ACID AS CROSS-LINKER

Citric acid is a molecule classified as a tricarboxylic acid, with a molecular weight of 210.14 g/mol. It contains three carboxylic acid functional groups, which exhibit pKa values of 3.1, 4.7, and 6.4. It is a naturally occurring compound found in trace amounts in most plants and animals, functioning as an intermediate in the tricarboxylic acid (Krebs) cycle. It was first obtained from lemon juice by Karls Scheels in England in 1874. Roughly 70% of citric corrosive is utilized within the nourishment and refreshment division, where it serves as a broadly favored acidulant since of its tart flavor and fabulous water solvency. These features have contributed to its increasing demand. In addition, its buffering ability and metal-binding properties make it useful in pharmaceutical and chemical industries, particularly as a sequestering agent in various formulations. Various microorganisms—including bacteria, fungi, and yeasts—have been identified as capable of producing citric acid. Table.2 lists the specific microorganisms involved in its production.

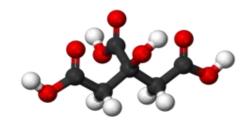
<u>Table 3</u>
Lists the specific microorganisms involved in its production

Туре	Microorganism	Commercial Use
Fungi	Aspergillus niger	Yes
Fungi	Penicillium janthinellum	No
Yeasts	Saccharomycopsis sp.	Yes
Yeasts	Candida lipolytica	No
Yeasts	Yarrowia lipolytica	No
Bacteria	Bacillus licheniformis	No
Bacteria	Corynebacterium glutamicum	No

However, for industrial-scale manufacturing, as it were Aspergillus niger and select yeasts like Saccharomycopsis species are commonly utilized.

<u>FIG.5</u>

A)



R۱

## Fig.5:Citric Acid structure (A) molecular and (B)Skeletal

Citric acid (CA) is extensively utilized in the medical, environmental, and food industries due to its non-toxic nature and cost-effectiveness as a natural active compound. In the European Union, it is approved as a food additive under the code E330 (with its sodium and potassium salts labeled as E331 and E332, respectively), while in the United States, it holds GRAS (Generally Recognized as Safe) status. This recognized safety and environmentally friendly profile make CA more suitable for use in the development of bio-based functional polymer films (BFPFs) compared to many other cross-linking agents. Additionally, the global industrial production of citric acid is well-established, and its manufacturing costs remain low. Structurally, CA contains three carboxyl groups and one hydroxyl group, enabling it to cross-link with various polymer molecules. The cross-linking (CLG) of CA with biopolymers can occur through both physical and chemical means. Physical cross-linking typically involves mixing CA with polymers at ambient temperatures, where bonding occurs through non-covalent interactions such as hydrogen bonding or ionic forces—examples include interactions with polyvinyl alcohol (PVA) and chitosan. As illustrated in Figure 1, CA can also chemically cross-link with cellulose at elevated temperatures via an esterification reaction. In this process, the carboxyl group of citric acid reacts with the hydroxyl group of cellulose. Initially, CA forms an anhydride intermediate through the removal of water, which then reacts with cellulose to create ester bonds. This intermediate can regenerate and further react with additional carboxyl groups, allowing the chemical cross-linking reaction to continue in a chain-like manner.

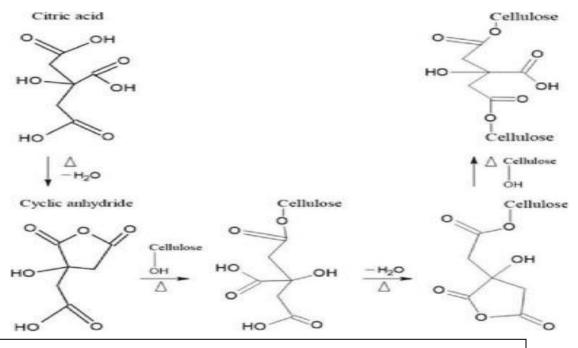


Fig.6: A predictive cross-linking reaction of citric acid with

The chemical cross-linking (CLG) reaction between citric acid (CA) and polymer materials generally requires high temperatures, typically above 100 °C. The effectiveness of this cross-linking is strongly influenced by both the temperature and the duration of the reaction. As most bio-based functional polymer films (BFPFs) are manufactured at room temperature, citric acid (CA) typically forms physical cross-links with a wide range of biopolymers. On the other hand, in cases where films are manufactured at high temperatures—such as those containing starch or cellulose—CA forms chemical cross-links under these elevated thermal conditions. Moreover, due to its hydroxyl group and acidifying nature, CA enhances the polymer matrix with additional antioxidant and antimicrobial functions, which further improves the preservation capacity of BFPFs. Its antimicrobial effect is largely due to its acidic character but is also attributed to its ability to chelate metal ions, thereby limiting the availability of essential nutrients for bacterial growth in the surrounding environment.

Recent advancements in bio-based functional polymer films (BFPFs) cross-linked with citric acid (CA) have shown significant potential in preserving fresh food. CA-based films have been effective in extending the shelf life of fresh fruits, vegetables, poultry, and seafood due to their antimicrobial, anti-fogging, and moisture-barrier properties.

- **Fruits**: Films incorporating CA help slow down deterioration in fresh produce by minimizing moisture loss, spoilage, and microbial invasion. For example, strawberries and tomatoes wrapped in CA-based films preserved their freshness, taste, and nutritional content better than those in standard packaging.
- **Vegetables**: Edible coatings with CA have been shown to delay browning and microbial spoilage in sliced vegetables such as lotus root and guava by suppressing enzyme activity and oxidative stress.
- **Poultry and Meat**: Films containing citric acid have been shown to significantly lower microbial levels in chicken and pork during refrigerated storage, thereby enhancing the safety and extending the market shelf life of these products.
- **Seafood**: CA-chitosan coatings extended the freshness of Japanese sea bass fillets, while cellulose nanofiber coatings with CA and essential oils preserved the quality of barbecue chicken.

Overall, citric acid enhances BFPF performance through cross-linking, acts as an antimicrobial and anti-browning agent, and supports improved shelf-life and sensory qualities of various perishable foods.

#### 1.6 ESSENTIAL OIL IN FOOD PACKAGING FILM

Essential oils (EOs) are extensively applied in the food industry as natural antimicrobial agents, particularly in packaging materials. Their antimicrobial effectiveness is primarily attributed to components such as aldehydes, phenols, and oxygenated terpenoids (Ju et al., 2017b; Khaneghah, Hashemi, & Limbo, 2018). One of the key features of EOs and their active constituents is their hydrophobic nature, which enables them to integrate into the lipid layers of microbial cell membranes and mitochondria. This interaction disrupts membrane structure, increasing permeability and leading to leakage of ions and other cellular contents. While minor leakage might not affect cell survival, significant loss of essential substances can result in microbial cell death (Ju et al., 2017b; Khaneghah et al., 2018). EOs do not act through a single antibacterial mechanism; rather, they often employ multiple modes of action simultaneously. Essential oils exhibit antimicrobial properties by targeting multiple cellular processes, including cell wall disruption, cell membrane damage, DNA interference, and inhibition of respiration and energy production, as illustrated in Fig.7.

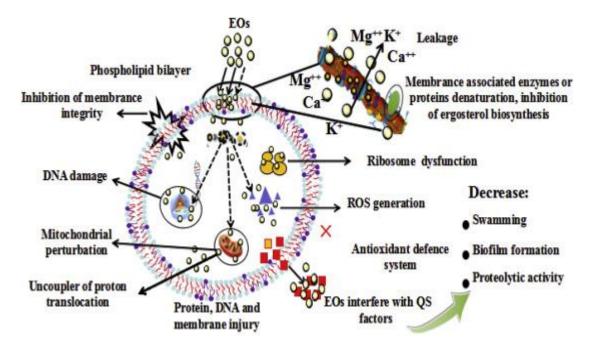


Fig.7: The possible action mechanisms of EOs.

Essential oils (EOs) derived from natural sources are promising alternatives to synthetic preservatives, offering advantages in maintaining food quality and safety while posing minimal risks to human health. Incorporating EOs into food packaging materials helps address these challenges by enhancing their antimicrobial efficacy, improving stability, and preserving overall product quality.

Despite progress in EO-based antimicrobial packaging (EO-APs), several issues remain unresolved. One key concern is that the distinct scent of

14

EOs can alter the flavor and aroma of packaged food. Additionally, research on how antimicrobial agents migrate through packaging materials is still limited, particularly studies using methods such as statistical correlation, numerical simulations, or finite element modeling. Although many EO components are recognized as safe or approved as flavor additives, some studies have reported potential irritation or toxicity. Furthermore, EO regulations vary between countries, making it important to consider legal compliance in international markets.

Moreover, the effectiveness and physical properties of EO-APs depend on multiple factors, including the EO type, the concentration of active ingredients, and the packaging material used. It's also important to establish connections between specific EO-AP applications and the types of microorganisms they are meant to control to ensure optimal performance. Advanced delivery systems for encapsulating essential oils, particularly when paired with complementary preservation methods like

#### 1.6.1 GINGER ESSENTIAL OIL (GEO) IN FOOD PACKAGING FILM

high pressure, refrigeration, ozone treatment, or modified atmosphere packaging, can significantly boost their antimicrobial effectiveness.

Ginger essential oil is an aromatic and volatile extract obtained from the rhizomes of *Zingiber officinale*. This oil constitutes approximately 0.25%–0.3% of the rhizome's content and is primarily made up of active compounds such as gingerols, aromatic alcohols, and terpenoids. It is considered the most bioactive component of ginger due to its broad-spectrum biological activities, including antioxidant, antimicrobial, and anticancer effects. Research has shown that ginger essential oil possesses potent antioxidant properties, demonstrating significant radical scavenging activity against ABTS and DPPH free radicals. The IC50 values of 0.54 mg/mL for ABTS and 10.03 mg/mL for DPPH demonstrate ginger essential oil's effectiveness in mitigating oxidative stress.

The functional properties of essential oils are directly linked to their chemical composition. Ginger essential oil (GEO) contains two main groups of active compounds: monoterpenes ((C<sub>5</sub>H<sub>8</sub>)n) and sesquiterpene hydrocarbons, along with oxygenated compounds like aldehydes, phenols, and alcohols. Table.4 given below summaries the major components of common GEO and their contents.

<u>Table.4</u>
Major components of common GEO and their contents.

S/ NO.	Chemical components	Range of conc. (%)	Formula
1	α-Zingiberene	18.0–28.0	C15H24
2	Trans-Caryophyllene	9.0–10.8	C15H24
3	Geranial	7.8–13.8	C10H16O
4	β-Sesquiphellandrene	6.5–11	C15H24

# Table 4(continued)

S/ NO.	Chemical components	Range of conc. (%)	Formula
5	Neral	5.3–10.5	C10H16O
6	Camphene	5.0-11.5	C10H16
7	Eucalyptol	5.0-5.5	C10H18O
8	β-Phellandrene	4.9–5.5	C10H16
9	α-Curcumene	4.4–11.5	C15H22
10	α-Pinene	2.1–3.0	C10H16
11	Myrcene	1.8 – 2.0	C10H16
12	Heptan-2-ol	1.0-2.0	C7H16O
13	Linalyl propionate	1.0-1.5	C13H22O2
14	Citronellol	0.9 - 1.0	C10H20O
15	Borneol	0.8 - 1.0	C10H18O
16	Zingiberenol	0.7 - 1.5	C15H26O
17	β-Elemene	0.7 - 1.0	C15H24
18	2-Undecanone	0.6 – 0.9	C11H22O
19	(E,E)-Farnesol	0.5 - 1.9	C15H26O
20	Citronellal	0.5 - 1.0	C10H18O
21	1,6-Octadien-3-ol,	0.5 - 1.0	C10H18O
	3,7- dimethyl		
22	Levomenol	0.5 – 0.9	C15H26O
23	Elemol	0.5 – 0.8	C15H26O
24	trans-β-Farnesene	0.4–1.0	C15H24
25	β-Pinene	0.4–1.0	C10H16
26	Isogeranial	0.4-0.5	C10H16O
27	Geraniol	0.43753	C10H18O
28	6-Methyl-5-hepten-	0.3 – 0.6	C8H14O
	2- one		
29	γ-Amorphene	0.3 – 0.6	C15H24
30	α-Copaene	0.3-0.5	C15H24
31	α-Phellandrene	0.2967	C10H16
32	Terpinolene	0.2 - 0.5	C10H16
33	Geranyl acetate	0.2 - 0.5	C12H20O2
34	γ-Elemene	0.2 – 0.5	C15H24
35	Aromadendrene	0.2 – 0.4	C15H24
36	Eremophilene	0.2 – 0.4	C15H24
37	α-Selinene	0.2 - 0.3	C15H24
38	γ-Eudesmol	0.1 - 0.5	C15H26O
39	α-Panasinsene	0.1–0.2	C15H24

The major components of ginger essential oil (GEO) include sesquiterpene hydrocarbons, with key compounds being  $\alpha$ -zingiberene (18.0–28.0%), geranial (7.8–13.8%), and trans-caryophyllene (9.0–10.8%). Other notable constituents are eucalyptol,  $\beta$ -phellandrene, camphene,  $\alpha$ -pinene, and

heptan-2-ol. These compounds contribute to GEO's distinct flavor, aroma, and its antibacterial and antioxidant properties.

In terms of antimicrobial properties, ginger oil demonstrates strong inhibitory action against a variety of bacterial strains, including *Staphylococcus aureus*, *Escherichia coli*, *Pseudomonas aeruginosa*, and *Acinetobacter baumannii*, along with several drug-resistant clinical isolates of *A. baumannii*.

Ginger essential oil's benefits span multiple fields, including healthcare and agriculture, showcasing its versatility. When sprayed on crops, it helps to reduce pest retention time, thereby minimizing both pest-related damage and the environmental impact of chemical pesticides.

Ginger essential oil also exhibits preservation effects, particularly in fruits like papaya, where it helps maintain freshness and microbial safety. Animal studies have shown that ginger essential oil supplementation boosts antioxidant enzyme activity and reduces lipid peroxidation in reproductive cells, with notable benefits observed in birds such as quails. Despite its benefits, ginger essential oil's volatility restricts its use in food and agricultural systems. It is prone to degradation from environmental conditions such as air exposure, temperature changes, humidity, and oxidation, which can significantly reduce its effectiveness over time.

In recent times, ginger essential oil (GEO) has been explored for its role as a natural food preservative. For example, Kasi et al. (2017) evaluated the effects of ginger extract on peanut oil and found that it significantly enhanced antioxidant activity by scavenging ABTS<sup>+</sup> radicals, reduced aflatoxin contamination risks, and improved the oil's linoleic acid ratio. These changes led to increased stability and longer shelf life of the oil.

Additionally, GEO demonstrated a favorable safety profile when applied to food. According to Ahmed et al. (2021), it showed low toxicity levels in human liver cancer cells (HepG2) and negligible toxicity toward normal lung fibroblast cells (WI-38), supporting its suitability for food applications. Despite these benefits, certain limitations restrict GEO's direct use. Ginger essential oil's volatility, limited water solubility, and interactions with food components can impact the flavor and aroma of food products. For instance, Mattje et al. (2019) reported that GEO added to burgers influenced their flavor and overall acceptability, and it was less effective than other additives like sodium isoascorbate or ginger extract in preventing fat oxidation. The rapid evaporation of ginger essential oil can compromise its antioxidant efficacy, potentially diminishing its benefits.

To overcome these challenges, GEO can be encapsulated using techniques such as emulsions or solid particle carriers. It can also be incorporated into biodegradable packaging films or coatings made from proteins or polysaccharides. These approaches help protect GEO's active components, enhance its antibacterial and antioxidant effects, and reduce unwanted flavors and odors, making it more effective and practical for use in food preservation.

A wide range of research has confirmed that incorporating ginger essential oil (GEO) significantly influences the functional characteristics of biodegradable food packaging films. Table.4 provides an overview of recent findings regarding how GEO affects properties such as film thickness, transparency, mechanical strength, moisture resistance, and both antimicrobial and antioxidant activity.

<u>Table 5</u>
Effects of GEO on properties of biodegradable food packaging films.

Source of EO	Type of polymer	Dose of GEO	Film properties
Ginger	Chitosan	0.1, 0.2,0.3 (%, v/v)	TS↓, EB↑
Cinnamon and ginger	Chitosan	0.00,0.05,0.20, 1.00 (%, v/ v)	Thickness and opacity \( \)
Cinnamon /Ginger	Chitosan- carboxymethyl cellulose	4.4,8.8,13.2/ 3.5,7.0,10.6 (%W?W)	Thickness, opacity, water contact angle and EB↑; WVP, TS and thermal
Ginger	Gelatin	0,10,20,40,80 (%,w/w)	stability↓ Thickness and EB↑; WVP and TS↓
Eugenol and ginger	Gelatin/Chitosan	0.5(%, w/w)	Opacity,WVP and EB↑; TS↓
Turmeric	Chitosan	1.5 μL/cm2	Thickness, WVP and opacity↑; TS, EB, swelling ratio, WS and MC↓
Ginger	Fish Sarcoplasmic Protein/ Chitosan	0.5, 1.0(%,v/v)	Thickness and opacity↑; WVP, TS, EB and WS↓
Ginger	Fish Sarcoplasmic Protein/ Chitosan	0, 0.5, 1, 1.5, 2, 3 (%, v/v)	
Ginger	chitosan	0, 0.1, 0.2,0.3 (%, w/v)	Thickness and EB↑; TS↓,

Table 5 continued

Source of EO	Type of polymer	Dose of GEO	Film properties
Lemon, ginger, peppermint, bergamot, sweet orange, and grapefruit	Sodium alginate and agar	0.2 (%, w/v)	Thickness,water contact angle and EB\(\gamma\); TS and thermal stability\(\psi\)
Ginger	Chitosan (Ch) montmorillonite (MMT)	0.1, 0.3, 0.5 (%, v/v)	Thickness, and WVP and EB↑; TS↓
Ginger and Rosema	,	0.5, 1 (%, w/w)	•
Ginger	Gelatin	0,0.25,0.5,0.75 , 1 (%, w/ v)	Thickness, WVP and EB↑; TS↓
Ginger	Chitosan	0, 0.1, 0.2, 0.3 (%, v/w)	Water contact angle and thermal stability↓
Green tea/ Ginger	NFC /Starch	1 (%, w/v)	Thickness, opacity, water contact angle and TS↑; WS,MC EB and WVP↓
Ginger	Mung bean star cellulose nanocrystals	ch 0,11,22,33 (%, v/w)	Thickness, opacity, water contact angle and EB↑; TS↓
Ginger	Agar- sodium alginate (A	1, 2, 3, 4 (%, v/v)	Oxygen permeability, WVP, MC, WS and swelling ratio↓
Ginger	arboxymethyl cellulose (CMC polyvinyl alcol (PVOH)		Thickness, opacity, water contact angle, WVP and EB↑; Thermal Stability, WS And TS↓
Ginger	Gelatin/Chitosar	1.5 (%, v/v	· ·

Because most biopolymer materials used in these films are hydrophilic, incorporating the hydrophobic GEO requires emulsification. Therefore, researchers often introduce GEO in the form of nanoemulsions or spicy emulsions, enabling it to be effectively enclosed within both the emulsion and the biopolymer matrix (Cai & Wang, 2021). Studies show that GEO-loaded solid nanoparticles, which provide dual encapsulation within biodegradable materials, potentially enhancing stability.

### 2. CHAPTER: MATERIAL AND METHODS

#### 2.1 RAW MATERIALS

For this experiment, we used Aloe Vera, pectin, citric acid, ginger essential oil (Ginger essential oil).

Pec, CA, EO(GEO) were provided by the physical laboratory (Department-Applied Chemistry, DTU). All chemicals employed were of reagent grade or superior purity.

AV gel was converted into liquid form of it. Pec and CA was taken from laboratory which was in powdered form. And GEO was also taken from laboratory.

#### 2.2 EXTRACTION OF AV

Aloe vera gel was provided by the fresh aloe vera plant leaves from terrace garden. Leaves were collected randomly from plant which showed no symptoms of disease or physical damage. They were adequately washed with running tap water, peeled and chopped into small pieces and the skin was stripped off to obtain gel.. After that it was grinded in a mixer grinder to obtain a thick paste which was then strained with a strainer. As a result, we get a gel into liquid form of it which was filtered to remove the remaining residual plant material.

#### 2.3 FILM FORMATION METHOD

The film blends of aloe vera, pectin, citric acid and ginger essential oil were prepared by casting method using a magnetic stirrer. There were many films made among which it was aloe vera with ginger essential oil and another one was aloe vera without ginger essential oil. The process of film formation was step by step given below

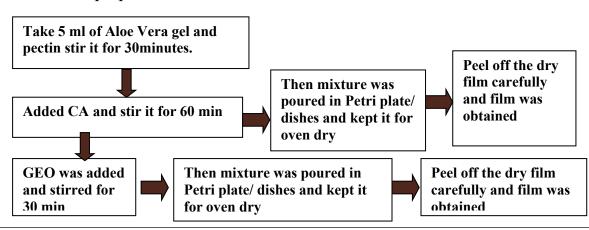
Firstly we poured 30ml of aloe vera gel into the beaker, and then we add 0.9gm of pectin, stir it for 30minutes.

After that 0.3gm of citric acid was added and stirred thoroughly for 60minutes then mixture was poured in Petri plate/ dishes and kept it for oven dry. After that Peel off the dry film carefully and film was obtained.

One of the film was prepared with combination of AV, Pec, and CA and another one was prepared adding 6 drops of GEO followed by steps given above for film preparation.

FIG.8

Flow chart of film preparation method



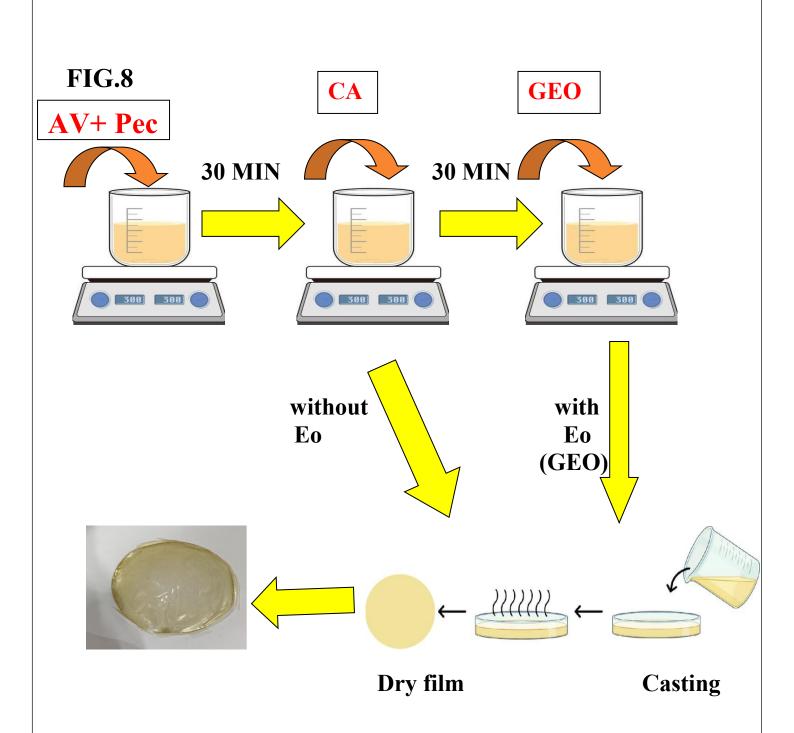


Fig.9 Methods and material of film preparation

### 3. CHAPTER: CHARACTERISATION AND RESULT

### 3.1 FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR) ANALYSIS

FTIR(Fourier Transform Infrared) spectroscopy is a valuable tool for analyzing the molecular structure and composition of food packaging films. This technique provides insights into the chemical properties of the material, ensuring it meets regulatory standards and is safe for food contact.

# 3.1.1 Key Applications:

- Material Identification: FTIR helps identify the type of polymer or material used in the packaging film.
- Chemical Composition Analysis: It provides information on the chemical bonds and functional groups present in the material, which is essential for understanding its properties and potential interactions with food.
- Contamination Detection: FTIR can detect contaminants or additives in the packaging material that might affect food safety or quality.
- Quality Control: By analyzing the FTIR spectrum, manufacturers can verify the material's composition and ensure it meets regulatory standards, such as FDA compliance.

#### 3.1.2 Benefits:

- Ensures Food Safety: FTIR analysis helps ensure the packaging material is safe for food contact and doesn't contain harmful contaminants.
- Verifies Material Composition: It confirms the material's composition, which is essential for quality control and regulatory compliance.
- Detects Potential Issues: FTIR can detect potential issues with the material, such as degradation or contamination, which can affect its performance and safety.

# 3.1.3 <u>FTIR Instrumentation</u>

An FTIR spectrometer consists of four key components:

- 1. Radiation Source: Infrared radiation is emitted across a wide range of wavelengths by a broad-spectrum light source, with source selection dependent on the target spectral region:
  - Ceramic emitters for mid-infrared
  - Mercury arc lamps for far-infrared
  - Halogen lamps for near-infrared
- 2. Interferometer: The heart of the FTIR spectrometer, typically based on the

Michelson design. It comprises:

- A beamsplitter
- A fixed mirror
- A moving mirror
  The interferometer modulates the IR beam, producing an interference pattern that's directed through the sample.
- 3. Sample Stage: The modulated beam interacts with the sample, either through transmittance or reflectance mode. The sample absorbs specific infrared frequencies, creating a unique spectral fingerprint.
- 4. Detection System: The detector measures the light after sample interaction, converting it to an electrical signal, with options including:
  - DLATGS for routine measurements
  - Cryogenically cooled detectors for high sensitivity
  - Silicon photodiodes for near-infrared detection
  - Silicon bolometer for far-infrared detection

FIG.10 Instrumentation of FTIR

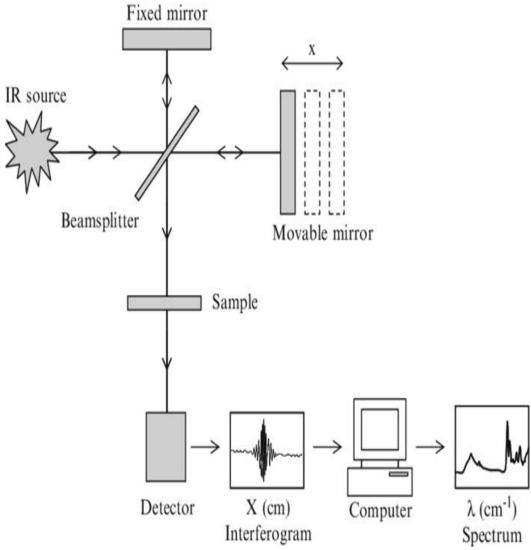
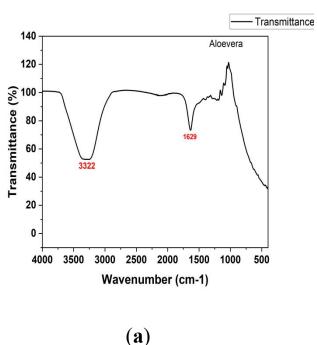
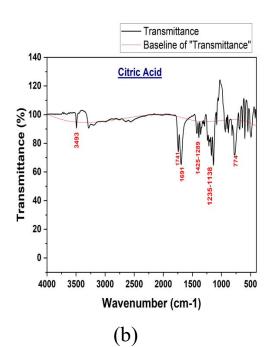


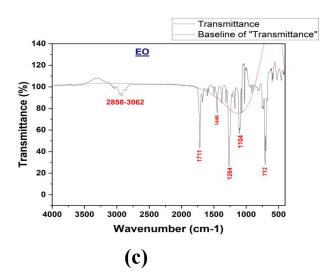
Image source: https://www.researchgate.net/figure/Schematic-sketch-of-the-essential-features-of-a-Fourier-transform-infrared-FTIR fig1 225065938

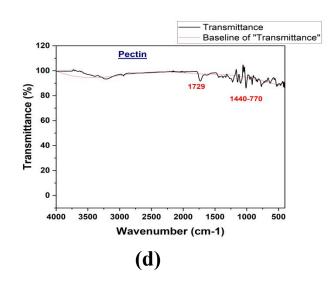
FTIR spectrums (Perkin Elmer FTIR spectrometer with ATR & Specular reflectance) of the prepared films were analyzed using FTIR spectra and recorded from wavenumbers 400-4000 cm-1. Sample were in the form of Aqueous (GEO, AV gel), Non-aqueous (CA, Pec), and film. The spectra were used to investigate component interactions within the films.

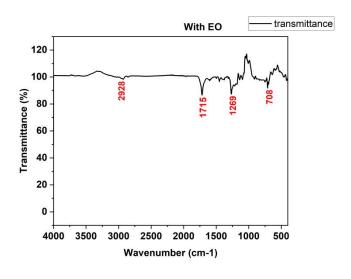
**FIG.10** 

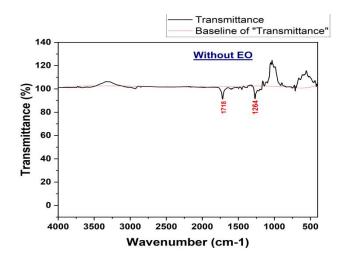












(e) (f)
Fig. 11 FTIR Spectra of (a) AV (b) CA (c) EO (GEO) (d) Pec (e) WithEO Film and (f) without EO Film

## 3.2 X-RAY DIFFRACTION (XRD) ANALYSIS

- 3.2.1 X-Ray Diffraction (XRD) is a technique used to analyze the crystalline structure of materials. In the context of food packaging films, XRD helps:
  - Determine Crystallinity: The degree of crystallinity, as measured by XRD, impacts the material's:
    - o Barrier performance (e.g., moisture, gas permeability)
    - Mechanical properties (strength and durability)
    - o Optical characteristics (e.g., clarity, haze)
  - Identify Materials: XRD can identify the type of polymer or material used in the packaging film.
  - Analyze Crystal Structure: XRD provides information on the material's crystal structure, including:
    - Crystal size and orientation
    - Lattice parameters
    - Defects or impurities
  - Ensure Quality and Performance: By analyzing the XRD pattern, manufacturers can:
    - o Verify material composition and structure
    - o Detect potential issues or defects
    - o Optimize material properties for specific applications

# 3.2.2 Benefits for Food Packaging

XRD analysis of food packaging films ensures:

• Material Safety: Verifies the material's composition and structure, ensuring it's safe for food contact.

- Performance Optimization: Allows tailoring material properties for applications like barrier films or flexible packaging or Facilitates material optimization for specific packaging needs.
- Quality Control: Enables detection of material defects, ensuring product reliability and safety.

## 3.2.3 Application of X-ray Diffraction and Bragg's Law

The phenomenon of X-ray diffraction plays a crucial role in exploring the internal structure of solids, particularly in the field of crystallography and X-ray spectroscopy. One of the most fundamental principles used in these studies is Bragg's Law, which provides a mathematical condition for constructive interference of X-rays reflected from different crystal planes.

To successfully apply Bragg's Law  $(2d\sin\theta=n\lambda)$  for determining crystal structures, it is essential to appropriately match the wavelength  $(\lambda)$  of the incident X-rays with the angle of incidence  $(\theta)$ . In practical experiments, this matching is achieved by either varying the wavelength of the X-rays or adjusting the orientation of the crystal, thereby altering the value of  $\theta$ .

To investigate the structure of crystals using this principle, three primary experimental techniques are commonly employed:

- Laue Method
- Rotating Crystal Method
- Powder Method
- 1. The Laue method is a foundational X-ray diffraction technique used to analyze crystal structures.
- O Principle and Experimental Setup:
  In this method, a beam of polychromatic (white) X-rays—with wavelengths typically ranging from 0.2 Å to 2 Å—is directed onto a small single crystal (usually about 1 mm³ in size), which is mounted on a goniometer. This instrument allows the crystal to be rotated, changing its orientation relative to the incoming X-ray beam. The X-ray beam hits the crystal surface perpendicularly to the plane of the crystal. As the rays pass through the crystal, they encounter multiple sets of crystal planes (Bragg planes), each with a unique spacing d and orientation. Constructive interference happens at specific angle θ formed with the incident beam and the crystal lattice, when the Bragg condition is fulfilled. That result in enhanced intensity at specific angles creates a distinct diffraction pattern.
- Observation of Diffraction Pattern:
   A photographic plate placed behind the crystal records result the diffraction pattern. The pattern typically consists of a symmetrically arranged set of spots, reflecting the symmetry elements of the crystal structure.

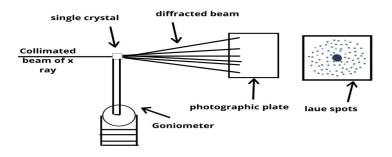


FIG.12 Laue method diagram XRD

### 2. Powder Crystal Method

The Powder Crystal Method, also referred to as the Debye-Scherrer technique, offers a simpler alternative to single-crystal techniques by eliminating the need for precise crystal orientation. Instead of aligning a single crystal along a specific axis, the material is finely ground into a powder, ensuring that the microscopic crystals (crystallites) are randomly oriented. A small powdered sample is mounted in a non-diffracting tube or bound with an inert material and hit with a narrow X-ray beam. With many randomly oriented crystallites, various crystal planes meet the Bragg condition ( $2d\sin\theta = n\lambda$ ), producing diffraction from many directions. These diffracted beams generate a cone-shaped pattern, with the cone's apex at the sample and its axis aligned with the incident beam. This diffraction pattern is captured using a Debye-Scherrer camera, which houses a photographic film wrapped inside a cylindrical chamber around the sample. After development, the film displays partial circular rings, which correspond to the different diffracting planes in the powder. It's particularly effective for identifying crystalline substances and studying materials with complex structures.

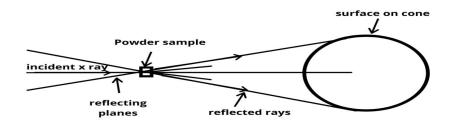
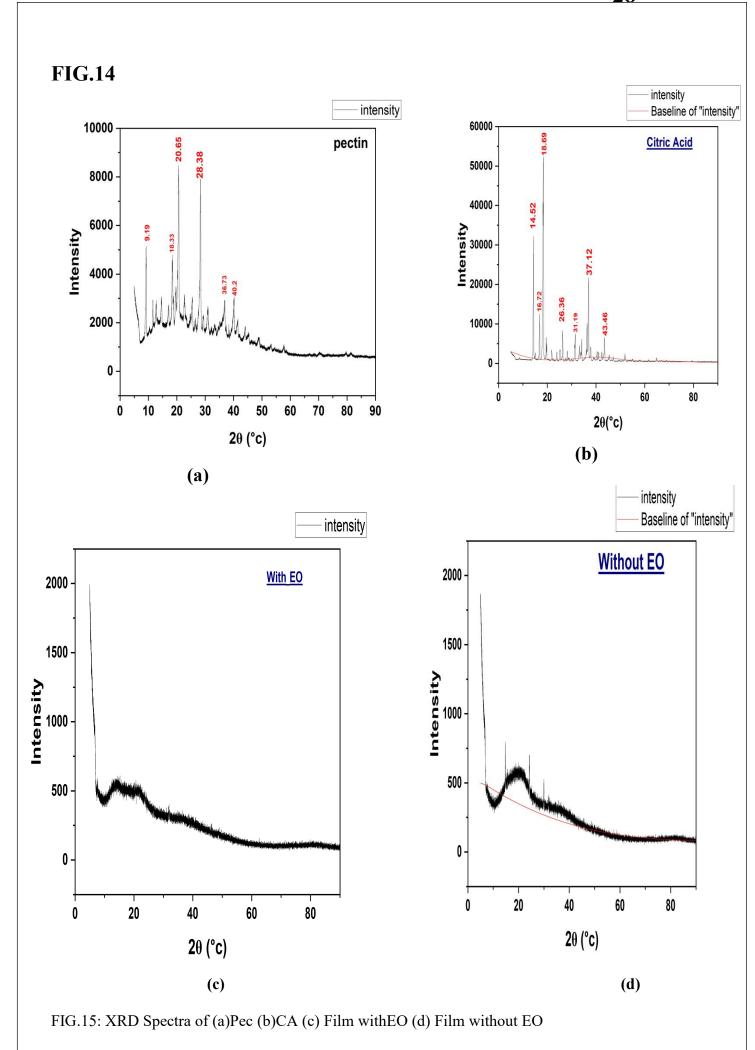


FIG.13 Powder method diagram of XRD

A diffractometer (Bruker High resolution X-ray diffractometer) with X-rays was used at to evaluate the XRD of the films and powders with a scanning speed of  $0.2^{\circ}$ / min range was fixed at  $2\theta$  between 0 and  $90^{\circ}$ .



**29** 

# 3.3 FESEM WITH EDX (Energy-Dispersive X-ray spectroscopy)

FESEM (Field Emission Scanning Electron Microscopy) and EDX (Energy-Dispersive X-ray spectroscopy) are valuable tools for characterizing food packaging films.

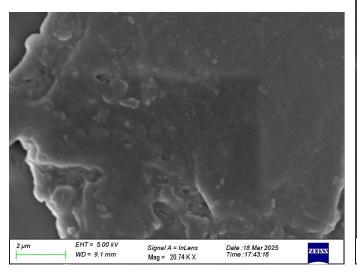
### • FESEM:

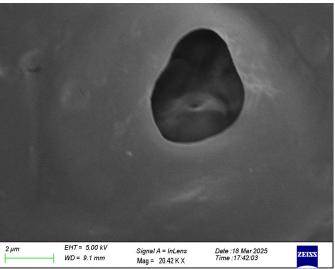
- 1. High-resolution imaging: FESEM provides detailed surface morphology images, revealing film characteristics like texture, roughness, and particle dispersion.
- 2. Film structure analysis: FESEM helps understand the film's internal structure, including layering, porosity, and defects.
- 3. Surface uniformity: FESEM assesses the film's surface uniformity, which can impact its barrier properties, printability, and overall performance.

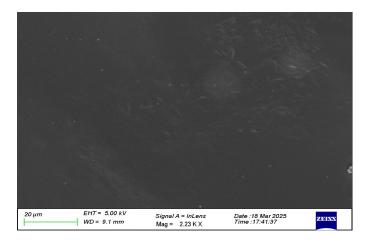
#### • EDX:

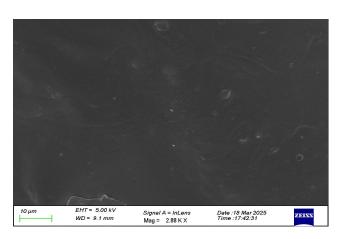
- 1. Elemental composition analysis: EDX analyzes the film's elemental makeup, detecting and measuring elements including additives, fillers, and impurities.
- 2. Elemental mapping: EDX mapping shows how elements are distributed throughout the film, shedding light on their arrangement and potential effects on film properties.
- 3. Material identification: EDX can help identify the type of materials used in the film, including polymers, metals, and inorganic fillers.
  - Benefits of FESEM-EDX for food packaging films:
- 1. Optimizing film formulation: By understanding the film's microstructure and composition, manufacturers can optimize the formulation to achieve desired properties.
- 2. Troubleshooting: FESEM-EDX can help identify defects, contaminants, or inconsistencies that may impact film performance.
- 3. Quality control: Regular FESEM-EDX analysis can ensure consistency and quality in film production.
- 4. Development of new materials: By providing detailed microstructural and compositional information, FESEM-EDX enables the development of innovative food packaging materials with tailored properties, such as improved barrier performance, mechanical strength, or biodegradability.
- By integrating FESEM and EDX, we can get a complete picture of food packaging films, facilitating the creation of high-performance packaging solutions that are both safe and environmentally friendly.

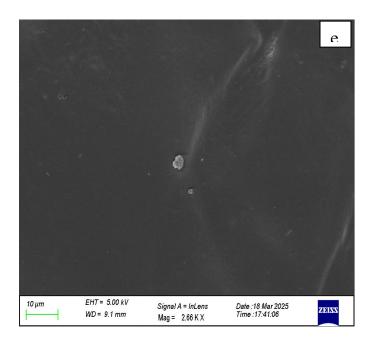
# **FIG.15**











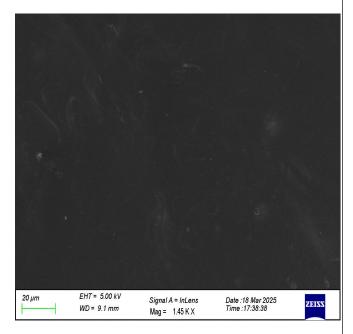
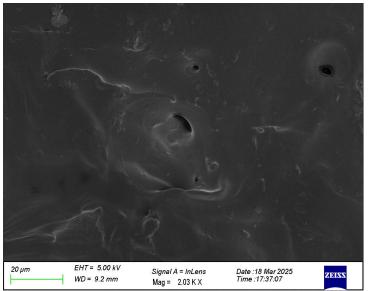
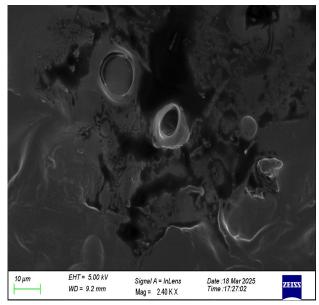
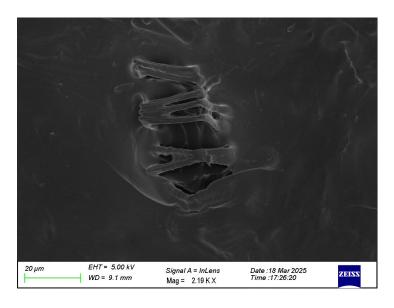


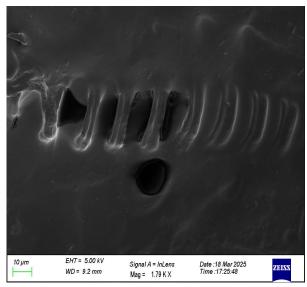
FIG.15: FESEM/Morphology image of film Without EO

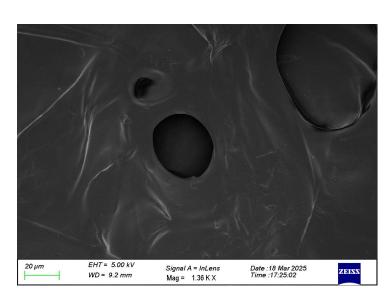
# **FIG.16**

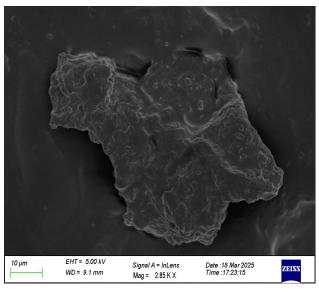












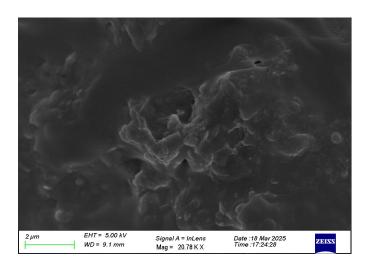
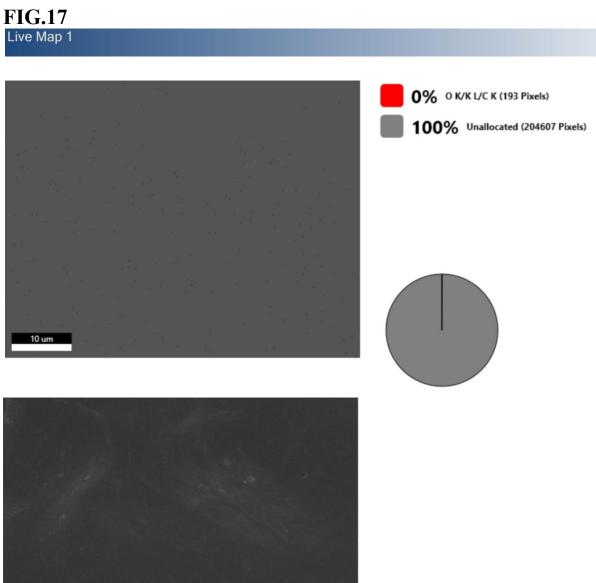


FIG.16: FESEM/Morphology image of film With EO



# ElementOverlay 24% K L 11% CK 7% CaL 3% N K 44% O K 2% MnL 2% FeL 4% NaK 2% PK K L\_ROI (5) C K\_ROI (4) N K\_ROI (3) CaL\_ROI (3)

34



FIG.17: EDX images of Without EO

**FIG.18** 

kV: 5 Mag:4450 Takeoff: 36 Live Time(s): 81.9 Amp Time(μs): 7.68 Resolution:(eV) 128

## Sum Spectrum

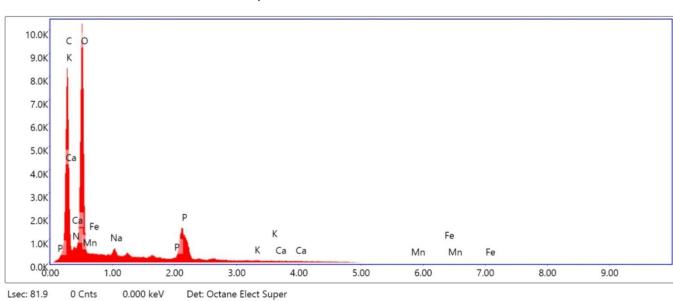


Table .6

## **eZAF Smart Quant Results**

Element	Weight %	Atomic %	Net Int.	Kratio	Z	Α	F
KL	0.00	0.00	0.00	0.0000	0.8022	0.9463	1.0000
СК	34.08	44.70	479.00	0.2780	1.0828	0.7533	1.0000
CaL	7.25	2.85	4.50	0.0406	0.8139	0.6885	1.0000
NK	0.10	0.12	1.00	0.0006	1.0435	0.5722	1.0000
ОК	46.77	46.04	758.00	0.3559	1.0102	0.7532	1.0000
MnL	0.00	0.00	0.00	0.0000	0.7054	0.7589	1.0000
FeL	0.00	0.00	0.00	0.0000	0.7152	0.8257	1.0000
NaK	1.67	1.14	17.50	0.0133	0.8924	0.8934	1.0014
PK	10.13	5.15	49.40	0.0841	0.8326	0.9929	1.0047

### 4. CHAPTER: CONCLUSION

The growing concern over non-biodegradable plastics in food packaging has driven the search for sustainable alternatives. This research project focused on developing biodegradable, edible films composed of Aloe vera gel, pectin, citric acid, and ginger essential oil (GEO). Each component was selected for its unique functional properties. Aloe vera gel served as a base polymer, providing antioxidant, antibacterial, and hydrating properties. Pectin, a natural polysaccharide, contributed mechanical strength and moisture barrier properties. Citric acid served as a natural cross-linking agent, promoting structural integrity through pectin esterification. The incorporation of GEO not only improved the bioactivity of the films but also extended the potential shelf life of wrapped food products.

The films were formulated using the casting method and comprehensively characterized using techniques like Fourier Transform Infrared (FTIR) Spectroscopy and X-Ray Diffraction (XRD) analysis. FTIR confirmed the chemical interactions among film components, revealing successful bonding and integration of active ingredients. Incorporating GEO and citric acid led to changes in crystallinity, as shown by XRD, affecting the film's mechanical, barrier, and optical properties. The experimental results substantiated the hypothesis that these natural components can be successfully integrated to create stable and functional biodegradable films suitable for food packaging purposes. These bio-based films align with environmental sustainability goals and offer added value in terms of food preservation, safety, and quality maintenance due to their inherent antimicrobial and antioxidant properties. Notably, the incorporation of GEO significantly enhanced flexibility, moisture resistance, and bioactivity.

This investigation provides a robust foundation for the development of biobased active packaging systems, underscoring the viability of these composite materials as substitutes for traditional plastic packaging solutions. However, further research is needed to improve mechanical properties, scalability, and compliance with food safety regulations. Future studies should also explore other natural extracts and essential oils for synergy and assess shelf life performance in real food storage conditions.

This study demonstrates that the synergistic combination of Aloe vera, pectin, citric acid, and ginger essential oil offers a viable and sustainable alternative to petroleum-based plastic films for food packaging purposes. These novel films have the potential to contribute meaningfully to environmental conservation while enhancing the safety, shelf life, and overall quality of packaged food products.

### 5. CHAPTER: REFERENCE

- 1. https://www.sciencedirect.com/science/article/abs/pii/S030881462404302
- 2. https://www.mdpi.com/2310-2861/8/8/505
- 3. https://www.sciencedirect.com/science/article/abs/pii/S014181302505747
- 4. https://www.sciencedirect.com/topics/engineering/film-casting-method
- 5. https://pmc.ncbi.nlm.nih.gov/articles/PMC10379830
- 6. https://www.sciencedirect.com/science/article/abs/pii/S0268005X230108 58
- 7. https://www.sciencedirect.com/science/article/abs/pii/S235255412300341
- 8. https://www.sciencedirect.com/topics/agricultural-and-biological-sciences/fourier-transform-infrared-spectroscopy
- 9. https://www.sciencedirect.com/science/article/abs/pii/S0268005X22006646
- 10.https://www.sciencedirect.com/science/article/pii/S2666893925001744
- 11.https://www.sciencedirect.com/science/article/pii/S2590157525000173
- 12.https://www.mdpi.com/2073-4360/17/9/1257
- 13.https://www.sciencedirect.com/science/article/pii/S2772753X25000528
- 14.<u>https://www.fda.gov/food/food-ingredients-packaging/food-chemical-safety</u>
- 15. https://pubs.rsc.org/fi/content/articlehtml/2025/lc/d4lc00637b
- 16. Ahmed, M., Auras, R., & Ullah, M. W. (2021). Ginger essential oil as a natural antioxidant and antimicrobial agent in biodegradable films for food packaging applications. *Food Packaging and Shelf Life*, 27, 100611. https://doi.org/10.1016/j.fpsl.2020.100611
- 17. Cai, L., & Wang, Y. (2021). Encapsulation of ginger essential oil in biodegradable nanoparticles for active food packaging. *International Journal of Biological Macromolecules*, 172, 330–339. https://doi.org/10.1016/j.ijbiomac.2021.01.011
- 18. Guilbert, S. (1986). Technology and applications of edible protective films. In M. Mathlouthi (Ed.), *Food packaging and preservation* (pp. 371–394). Springer. https://doi.org/10.1007/978-1-4613-2175-0 15
- 19. Ju, J., Chen, X., Xie, Y., Yu, H., Guo, Y., Cheng, Y., & Xu, X. (2017). Application of essential oils in food packaging: A review. *Food Chemistry*, 237, 1147–1156. https://doi.org/10.1016/j.foodchem.2017.05.044
- 20. Kasi, R., Ravindran, K., & Krishnakumar, B. (2017). Antioxidant and antimicrobial effects of ginger extract in peanut oil preservation. *Journal of Food Science and Technology*, 54(2), 341–349. https://doi.org/10.1007/s13197-016-2453-0
- 21. Khaneghah, A. M., Hashemi, S. M. B., & Limbo, S. (2018). Antimicrobial effectiveness of essential oils and their application in food packaging: A review. *Trends in Food Science* & *Technology*, 71, 132–138. https://doi.org/10.1016/j.tifs.2017.10.025
- 22. Kester, J. J., & Fennema, O. R. (1986). Edible films and coatings: A review. *Food Technology*, 40(12), 47–59.

- 23. Santhiya, D., & Kumar, A. (2023). Development and evaluation of biodegradable food packaging films using polysaccharides and essential oils. *Journal of Polymer and Environmental Research*, 31, 421–433.
- 24. https://www.mdpi.com/2304-8158/12/17/3288
- 25.https://www.sciencedirect.com/science/article/pii/S2590157524008411
- 26.https://www.chem.uci.edu/~lawm/263%25204.pdf
- 27. https://www.sciencedirect.com/topics/materials-science/x-ray-powder-diffraction
- 28. https://www.researchgate.net/publication/354866014\_Preparation\_and\_C haracterization\_of\_Ginger\_Essential\_Oil\_Microcapsule\_Composite\_Film\_s
- 29. FTIR: Principle, Instrumentation, Applications, Advantages,
- 30.X-ray diffraction and its emerging applications in the food industry PubMed
- 31. https://physicswave.com/x-ray-diffraction-analysis-principle-instrument-and-applications
- 32. Essential oils as additives in active starch-based food packaging films: A review ScienceDirect
- 33. https://drive.google.com/file/d/1dOLZ33xVxYhl8zGq88RxzZ7yfyr2vvUl/view
- 34.https://www.mdpi.com/2673-4176/3/3/29
- 35. https://www.sciencedirect.com/science/article/abs/pii/S014181302301142
- 36.https://www.intechopen.com/chapters/79990
- 37. <a href="https://www.sciencedirect.com/science/article/abs/pii/S030881462202926">https://www.sciencedirect.com/science/article/abs/pii/S030881462202926</a>
- 38.https://www.sciencedirect.com/science/article/pii/S0023643824013719
- 39.https://www.sciencedirect.com/journal/food-packaging-and-shelf-life
- 40.https://www.sciencedirect.com/science/article/pii/S2214993721001081
- 41.https://www.sciencedirect.com/science/article/pii/B978012811449000008
- 42.https://www.sciencedirect.com/science/article/abs/pii/S092134492200004
- 43.https://www.sciencedirect.com/science/article/abs/pii/S092134492030380 3
- 44.https://www.sciencedirect.com/science/article/pii/S0921344920303803
- 45.https://www.mdpi.com/2076-3417/15/3/1462
- 46.https://www.sciencedirect.com/science/article/pii/S2238785421009637
- 47. https://scholar.google.com/scholar%3Fq%3DPectin-based%2Bfilms%2Bexhibit%2Bexcellent%2Bfilm-forming%2Babilities,%2Bmechanical%2Bstrength,%2Band%2Beffective %2Bbarriers%2Bagainst%2Bmoisture%2Band%2Bgases.%26hl%3Den%26as\_sdt%3D0%26as\_vis%3D1%26oi%3Dscholart
- 48.https://pmc.ncbi.nlm.nih.gov/articles/PMC2763764
- 49.https://pubmed.ncbi.nlm.nih.gov/29377147
- 50. https://www.sciencedirect.com/science/article/abs/pii/S235255412400138

- 51.https://pmc.ncbi.nlm.nih.gov/articles/PMC5622393
- 52. <a href="https://bioresources.cnr.ncsu.edu/resources/characterization-and-identification-of-lacquer-films-from-the-qin-and-han-dynasties">https://bioresources.cnr.ncsu.edu/resources/characterization-and-identification-of-lacquer-films-from-the-qin-and-han-dynasties</a>
- 53.https://nishkaresearch.com/analytical-techniques/microscopy/fesem
- 54.https://www.sciencedirect.com/topics/chemistry/energy-dispersive-spectroscopy
- 55.https://www.bruker.com/en/landingpages/bna/technology/what-is-eds.html
- 56.https://pubs.acs.org/doi/10.1021/acsomega.3c03763
- 57. https://www.researchgate.net/publication/389000990