

Synthesis and Characterization of Glutaraldehyde and Citric Acid Crosslinked PVA-Starch Films: A Comprehensive Study

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CANDIDATE DECLARATION

I Tanishka Sethi hereby certify that the work being presented in the dissertation entitled “**Synthesis and Characterization of Glutaraldehyde and Citric Acid Crosslinked PVA-Starch Films: A Comprehensive Study**” in partial fulfilment of the requirements for the award of the Degree of Doctor of Philosophy, submitted in the Department of Applied Chemistry Delhi Technological University is an authentic record of my work carried out during the period from 2022 to 2024 under the supervision of Dr. Poonam Singh.

The content presented in the dissertation has not been submitted by me for the award of any other degree of this or any other Institute.

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Certified that **Tanishka Sethi** (2K22/MSCCHE/57) has carried out their search work presented in this thesis entitled “**Synthesis and Characterization of Glutaraldehyde and Citric Acid Crosslinked PVA-Starch Films: A Comprehensive Study**” for the award of **Master of Science in Chemistry** from Department of Applied Chemistry Delhi Technological University, Delhi, under my supervision. The dissertation embodies the results of original work, and studies are carried out by the student herself and the contents of the thesis do not form the basis for the award of any other degree to the candidate or anybody else from this or any other University/Institution.

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Abstract

This research examines the synthesis and analysis of polyvinyl alcohol (PVA) starch films using glutaraldehyde and citric acid as crosslinking agents. The use of biodegradable substances like PVA and starch, in combination with environmentally friendly crosslinkers, shows potential for creating sustainable packaging materials. The study seeks to clarify the impact of crosslinking agents on the physical characteristics of the resultant films, providing valuable insights into their possible uses in many industries.

The films were prepared using diverse synthetic procedures and then thoroughly characterized using Fourier-transform infrared (FTIR) spectroscopy and X-ray diffraction (XRD). The findings demonstrate clear changes in the structure and properties of PVA caused by the crosslinking of glutaraldehyde and citric acid. This strengthens our understanding of the process of film generation and the mechanisms involved. This research enhances the current work on sustainable materials development by providing new knowledge on the creation and characteristics of PVA starch films. This knowledge can help in the wider use of these films in packaging, biomedical applications, and environmental cleanup.

Keywords: PVA, Biodegradable, FTIR, XRD, Starch, Glutaraldehyde, Citric Acid

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List of abbreviations

PAV - polyvinyl alcohol

DHT - Dehydrothermal treatment

UV - Ultraviolet

Mtg - Microbial transglutaminase

GP - Genipin

GTA - Glutaraldehyde

BHVs - Bioprosthetic heart valves

CA - Citric acid

APIs - Active pharmaceutical ingredients

FTIR - Fourier Transform Infrared

CHAPTER 1

INTRODUCTION

The biodegradable polymers PVA and starch have attracted a lot of interest lately because of their potential uses in a variety of industries, especially the creation of environmentally friendly packaging materials (Hulupi and Haryadi, 2019). PVA and starch are appealing alternatives to traditional petroleum-based polymers in packaging and other applications because they both have advantageous qualities like biocompatibility, the capacity to create films, and renewable sourcing (Bhanu et al., 2017). To satisfy certain needs, such as mechanical strength, barrier qualities, and biodegradability, their natural qualities, however, frequently need to be improved or changed (Ma et al., 2016). A frequently used technique to enhance the characteristics of polymer blends is cross-linking. In this work, we investigate the synthesis and characterization of PVA starch films utilizing citric acid and glutaraldehyde as crosslinkers (Hulupi and Haryadi, 2019).

Examining the impact of citric acid and glutaraldehyde crosslinking on the mechanical, thermal, and physical characteristics of PVA starch films is the main goal of this study (Nataraj et al., 2020). Because they can create covalent connections with the hydroxyl groups found in both PVA and starch molecules, glutaraldehyde and citric acid are used as crosslinking agents to improve the stability and structural integrity of the films (Bhanu et al., 2017). Comprehending the influence of these crosslinkers on the characteristics of films is essential for refining the synthesis procedure and customizing the films' attributes for distinct uses (Ma et al., 2016).

We give a summary of the study's goals, history, and scope in this introduction. First, we go over the features and attributes of PVA and starch polymers, emphasising their potential benefits for use in environmentally friendly packaging materials (Teixeira et al., 2021). Next, we explain what crosslinking is and how it affects the properties of polymers. The study's precise goals are then outlined, including the synthesis of PVA starch films utilizing citric acid and glutaraldehyde as crosslinkers, the analysis of the films by a variety of analytical methods, and the assessment of the films' possible uses (Nataraj et al., 2020).

The research's scope includes synthesizing PVA starch films using various crosslinking techniques and thoroughly characterizing the films' mechanical, thermal, and structural characteristics afterward (Rowland et al., 2013). The results of this investigation will offer significant perspectives on how crosslinking agents affect PVA starch film characteristics, which will aid in the creation of environmentally friendly packaging materials with customized attributes and enhanced functionality (Uranga et al., 2020). Furthermore, the research's findings might have an impact on other fields like environmental remediation and biological materials (Teixeira et al., 2021).

1.1 Historical Background

The continuous effort to find sustainable materials and environmentally friendly substitutes for traditional plastics has been motivated by environmental concerns and the desire for more responsible manufacturing methods (Vashisth and Pruthi, 2016). Researchers and enterprises are increasingly focusing on the investigation of biodegradable polymers, such as PVA and starch, to minimize the environmental consequences of packaging materials and other consumer items (Teixeira et al., 2021).

PVA is a man-made polymer produced by breaking down polyvinyl acetate through a process called hydrolysis. Its biocompatibility, water solubility, and film-forming capabilities have been well-acknowledged over a considerable period (Uranga et al., 2020). PVA has been utilised in many industries, such as textiles, adhesives, and pharmaceuticals. Nevertheless, the widespread

utilization of single-use packaging materials has generated apprehensions over environmental contamination and sustainability (Teixeira et al., 2021).

Starch, however, is a naturally occurring polymer that is plentiful in plants such as corn, wheat, and potatoes. It is composed of glucose molecules that are connected in lengthy chains. Additionally, it can be broken down by natural processes, can be replenished, and is cost-effective (Vashisth and Pruthi, 2016). Historically, starch has been employed in food packaging and as a viscosity-enhancing ingredient in several industries. Nevertheless, the mechanical qualities and moisture sensitivity of this material frequently restrict its usage in packaging materials (Uranga et al., 2020).

To meet the increasing need for environmentally friendly packaging, scientists have investigated the combination of PVA and starch to utilise the advantageous characteristics of both polymers (Li et al., 2009). The combination of PVA with starch has the potential to enhance the mechanical strength, moisture resistance, and biodegradability of the material, while yet maintaining the biocompatibility and ability to create films that PVA possesses (Hulupi and Haryadi, 2019).

Crosslinking agents are essential for altering the characteristics of polymer blends by creating chemical connections between polymer chains, which improves overall strength and durability (Uranga et al., 2020). Glutaraldehyde and citric acid are often employed crosslinking agents that chemically interact with hydroxyl groups found in both PVA and starch molecules (Hulupi and Haryadi, 2019).

The historical context provides the foundation for the current investigation, which seeks to expand on prior studies and investigate innovative methods for creating and analysing PVA starch films employing glutaraldehyde and citric acid as crosslinking agents (Bhanu et al., 2017). This study aims to contribute to the continued development of eco-friendly packaging materials with enhanced characteristics and performance by examining the historical context and evolution of sustainable materials research (Ma et al., 2016).

1.2 Need for sustainable packaging materials

The growing worldwide apprehension over environmental contamination and the impractical utilisation of traditional plastic packaging materials has spurred the quest for environmentally preferable substitutes (Nataraj et al., 2020). Although PVA and starch have desirable characteristics such as biodegradability and renewability, their limited mechanical strength and susceptibility to moisture restrict their extensive use in packing materials (Teixeira et al., 2021). In addition, the process of creating PVA starch films with crosslinking agents poses difficulties in attaining the most favourable characteristics for eco-friendly packaging (Dean & Petinakis, 2008).

An issue arises in the requirement to improve the mechanical strength and moisture resistance of PVA starch films while maintaining their ability to biodegrade and produce films (Hulupi and Haryadi, 2019). Traditional techniques for crosslinking, such as employing glutaraldehyde or citric acid, necessitate meticulous fine-tuning to attain the desired equilibrium of characteristics. Choosing the right crosslinking agent and synthesis process is crucial in dealing with this difficulty (Kaftan and Acikel, 2005).

In addition, there is a limited understanding of the complete structural and property changes caused by crosslinking in PVA starch films (Ma et al., 2016). To fully understand the molecular and morphological changes, it is important to conduct a comprehensive examination using

techniques such as FTIR spectroscopy, XRD, and SEM. This will help uncover the underlying mechanisms and optimize the synthesis process (Li et al., 2009).

A further issue arises due to the varied demands of sustainable packaging materials in various applications. The necessary characteristics for food packaging, such as those required for biological or environmental remediation objectives, may vary (Ma et al., 2016). Conducting a comprehensive investigation of the impact of crosslinking agents on film properties is necessary to address the difficulty of customizing PVA starch films to fulfil specific application needs (Kaftan & Acikel, 2005). To tackle these issues, a thorough examination of the synthesis, characterisation, and potential application of PVA starch films utilising glutaraldehyde and citric acid as crosslinkers is necessary (Ma et al., 2016). Through this research, the issues related to the production of sustainable packaging materials can be identified and solutions can be proposed. This will help in making progress towards finding ecologically acceptable alternatives to traditional plastics (Hulupi and Haryadi, 2019).

CHAPTER 2
LITERATURE
REVIEW

2.1 Research Aim & Objectives

2.1.1 Research Aim

This research aims to examine the production and analysis of PVA starch films by employing glutaraldehyde and citric acid as crosslinking agents (Bhanu et al., 2017). The main aim is to assess the impact of crosslinking on the physical, mechanical, and thermal characteristics of the films, with the ultimate objective of improving their appropriateness for sustainable packaging purposes.

2.1.2 Research Objectives

1. To synthesize PVA starch films using glutaraldehyde and citric acid as crosslinkers through various preparation methods.
2. To characterize the synthesized films using FTIR spectroscopy, XRD, SEM testing.
3. To analyze the influence of crosslinking agents on the physical, mechanical, and thermal properties of the PVA starch films and assess their potential for sustainable packaging applications.

2.1.3 Significance of this work

This work is important because it contributes towards the development of sustainable packaging materials. This can be achieved by creating and modifying PVA starch films utilising glutaraldehyde and citric acid as crosslinkers (Rowland et al., 2013). The study focuses on the urgent requirement for environmentally suitable substitutes for traditional plastic packaging, presenting possible remedies for environmental contamination and resource exhaustion (Teixeira et al., 2021).

The objective of this study was to investigate the impact of crosslinking agents on the characteristics of PVA starch films, with the goal of improving their mechanical strength, resistance to moisture, and thermal stability (Teixeira et al., 2021). Enhancing these characteristics is essential for broadening the utilisation of PVA starch films in diverse sectors, such as food packaging, biomedical materials, and environmental remediation (Penniston, 2008).

Moreover, the use of biodegradable polymers like PVA and starch aids in diminishing the ecological consequences of packaging substances (Vashisth and Pruthi, 2016). PVA and starch are sustainable resources that may be obtained in a renewable manner, unlike traditional polymers made from fossil fuels. In addition, the capacity of these materials to break down naturally into harmless substances provides a solution to the issue of plastic waste buildup in landfills and oceans (Rowland et al., 2013).

This research is also important for furthering scientific knowledge in the fields of polymer chemistry and materials science. This study enhances the fundamental understanding of polymer production and processing by examining the changes in structure and properties caused by crosslinking (Uranga et al., 2020). The used characterisation techniques, namely FTIR spectroscopy, XRD, and SEM, offer unique insights into the chemical and morphological alterations taking place in the PVA starch films (Teixeira et al., 2021).

The research findings are anticipated to have practical consequences for industries that are seeking sustainable packaging solutions and to contribute to broader efforts in achieving

environmental sustainability (Rowland et al., 2013). The research conducted by Uranga et al. (2020) establishes the practicality and effectiveness of PVA starch films as environmentally friendly packaging materials. This study sets the stage for their utilisation in commercial settings, promoting a more sustainable future.

2.1.4 Research justification

The justification for this research stems from the increasing need to tackle the environmental consequences of traditional plastic packaging materials (Hulupi and Haryadi, 2019). Given the escalating global levels of plastic pollution, there is an urgent requirement for sustainable alternatives that reduce environmental damage (Rowland et al., 2013). The biodegradability and renewable supply of PVA and starch make them a highly attractive option for sustainable packaging. Nevertheless, the actual implementation of these materials is impeded by drawbacks such as insufficient mechanical robustness and susceptibility to moisture (Uranga et al., 2020).

This research aims to improve the properties of PVA-starch films by investigating the use of crosslinking chemicals, specifically glutaraldehyde and citric acid, in order to overcome the constraints associated with them. The justification for utilizing crosslinking agents stems from their capacity to enhance mechanical strength, moisture resistance, and thermal stability by creating covalent connections between polymer chains (Li et al., 2009).

This discovery has consequences that go beyond academic curiosity and can be applied practically in companies and society. This research makes a valuable contribution to the worldwide initiative of reducing plastic pollution and promoting environmental sustainability by creating sustainable packaging materials that have similar or better performance than traditional plastics (Nataraj et al., 2020). Moreover, the wide range of uses for PVA starch films in various sectors, such as food packaging and biomedical materials, underscores the significance and influence of this research in tackling urgent societal and environmental issues (Rowland et al., 2013).

2.1.5 Dissertation Organization

This research is divided into five chapters, with an introduction, research aim and objectives, and research questions as the main focus; the duration of the research is about one month. The study literature evaluation entails a detailed examination of current resources and conceptual frameworks. This process normally takes about two months to complete. The following chapter focuses on research methodology, namely the process of selecting and characterizing techniques and procedures. This phase took one month to complete. The fourth chapter will cover the interpretation of results and discussions including mathematics and graph plotting. The analysis will centre on a particular month of investigation. As, we discussed the conclusion and Perspective, which took a month of investigation.

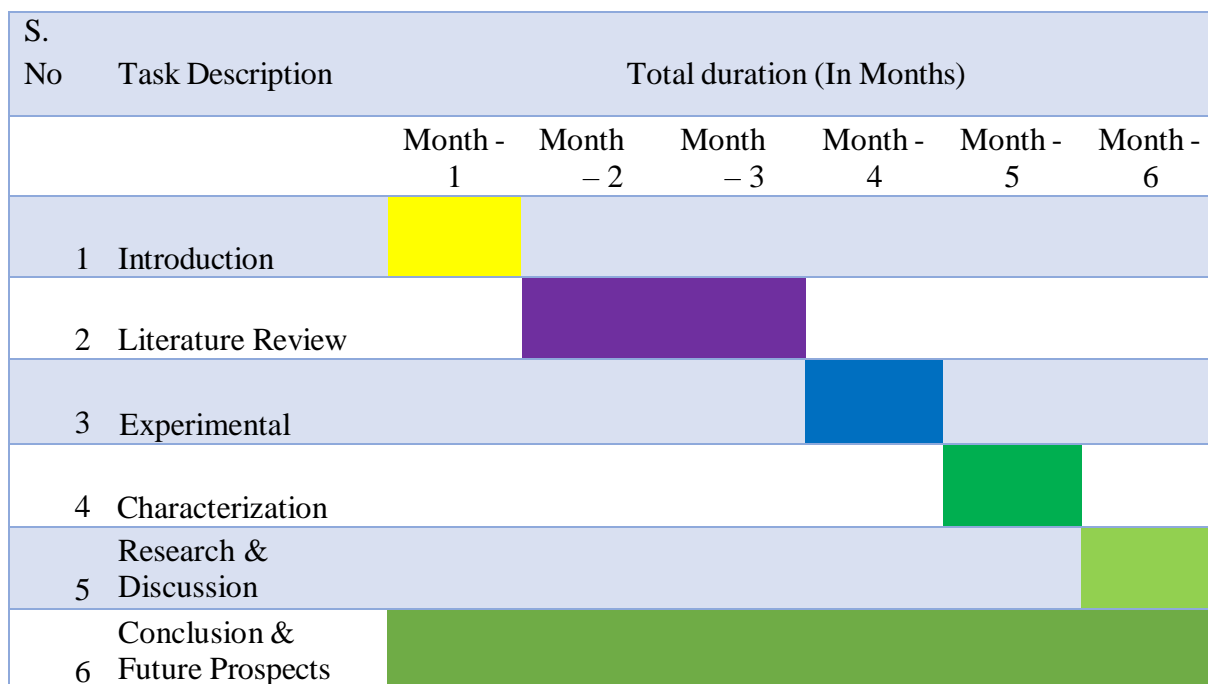


Figure 2.1 Gantt chart of the research plan

2.2 Review of Literature

The existing body of literature on the production and analysis of PVA starch films, which involve the use of glutaraldehyde and citric acid as crosslinking agents, demonstrates a growing fascination with eco-friendly packaging materials (Ma et al., 2016). Prior research has thoroughly investigated the characteristics and possible uses of PVA starch blends, with a particular focus on their capacity to create films, their biocompatibility, and their renewable supply (Vashisth and Pruthi, 2016). Scientists have examined different characteristics of PVA starch films, such as their mechanical qualities, barrier performance, and capacity to break down naturally, to emphasise their potential to substitute traditional petroleum-based polymers in packaging (Teixeira et al., 2021).

The use of crosslinking agents, such as glutaraldehyde and citric acid, has been extensively researched due to their capacity to improve the characteristics of PVA starch films. Glutaraldehyde, a traditional crosslinking agent, creates covalent connections with hydroxyl groups, leading to enhanced mechanical durability and thermal resistance (Ma et al., 2016). On the other hand, citric acid provides an environmentally beneficial option by enabling the formation of connections between molecules while yet allowing the films to be broken down naturally (Hulupi and Haryadi, 2019). Research has shown that crosslinking has a significant impact on the characteristics of films, such as higher tensile strength, lower water solubility, and improved resistance to moisture and gases (Ma et al., 2016).

Various characterization techniques, including FTIR spectroscopy, XRD, SEM, and mechanical and thermal testing, have been used to analyze the changes in structure and properties caused by cross-linking. These approaches offer essential information about the chemical interactions, crystalline structure, shape, and performance of PVA starch films (Teixeira et al., 2021).

Moreover, the literature emphasises the wide range of uses for PVA starch films, including but not limited to food packaging, biomedical materials, and environmental remediation (Vashisth

and Pruthi, 2016). The role of crosslinking agents is vital in customizing the properties of films to satisfy the specific demands of different applications, highlighting the need to comprehend their impact on film features (Hulupi and Haryadi, 2019). The literature review highlights the importance of PVA starch films as environmentally friendly packaging materials and emphasizes the crucial function of crosslinking agents in adjusting their properties for different uses. This contributes to the continuous endeavors to develop sustainable materials (Ma et al., 2016).

2.2.1 Theoretical framework

PVA-starch-based films utilizing glutaraldehyde and citric acid as crosslinkers are based on the theoretical framework of polymer chemistry, materials science, and sustainable packaging principles (Nataraj et al., 2020). The framework primarily involves molecular interactions, structural changes, and improvements in properties that are enabled by crosslinking agents in PVA starch blends (Rowland et al., 2013).

PVA and starch are polymers that possess distinctive characteristics, rendering them very desirable options for eco-friendly packaging materials (Wang et al., 2009). PVA, obtained from the hydrolysis of polyvinyl acetate, possesses biocompatibility, the capacity to form films, and solubility in water. Starch, a plentiful natural polymer found in plants, offers the advantages of being renewable and biodegradable (Ma et al., 2016). Nevertheless, both polymers have intrinsic drawbacks, such as inadequate mechanical strength and susceptibility to moisture, which require alterations for practical use (Dean & Petinakis, 2008).

Glutaraldehyde and citric acid are crosslinking agents that act as chemical connectors between polymer chains, improving the structural strength and effectiveness of PVA starch films (Wang et al., 2009). Glutaraldehyde undergoes covalent bonding with hydroxyl groups, resulting in the formation of crosslinks and enhancing the mechanical strength. Citric acid, a more environmentally friendly option, achieves crosslinking by means of esterification processes while still retaining biodegradability (Nataraj et al., 2020).

The theoretical framework also includes the characterization methodologies employed to examine the structural and property modifications caused by crosslinking (Li et al., (2009). FTIR spectroscopy offers valuable information about chemical interactions, whereas XRD reveals alterations in crystalline structure. SEM enables the study of the structure and shape of materials, while mechanical and thermal tests provide numerical information about the properties of the film (Dean and Petinakis, 2008).

In addition, the theoretical framework takes into account the wider context of sustainable packaging and the environmental issues that motivate the creation of eco-friendly alternatives to traditional plastics (Li et al., 2009). This framework provides a clear understanding of how crosslinking works and how it impacts the properties of films. It helps in improving the synthesis processes and customising PVA starch films for specific uses in packaging, biomedical materials, and environmental remediation (Hulupi and Haryadi, 2019). The theoretical framework combines elements from polymer chemistry, materials science, and sustainability to enhance the comprehension and utilization of PVA starch films as effective alternatives to standard plastic packaging (Choi et al., 2016).

2.2.2 Cross-Linking Agents

A lot of studies have been done on different crosslinking methods to make a scaffold that can support BTE. Electrospun-based bone structures are crosslinked by joining functional groups that are shown on nanofibers (Bhanu et al., 2017). More than two reacting ends of polymer chains must be chemically linked together for them to join. Before they can be put in the body, all electrospun bone scaffolds have to be solid (Hulupi and Haryadi, 2019). Cross-linking methods can keep the nanofibrous features of electrospun scaffolds, which are similar to the ECMs found in natural bone (Dean & Petinakis, 2008). Better cross-linking can keep nanofibers from breaking apart and improve their dynamic properties. Electrospun scaffolds that have been mechanically improved can handle biophysical difficulties such as pH, enzymes, and body loads (Bhanu et al., 2017).

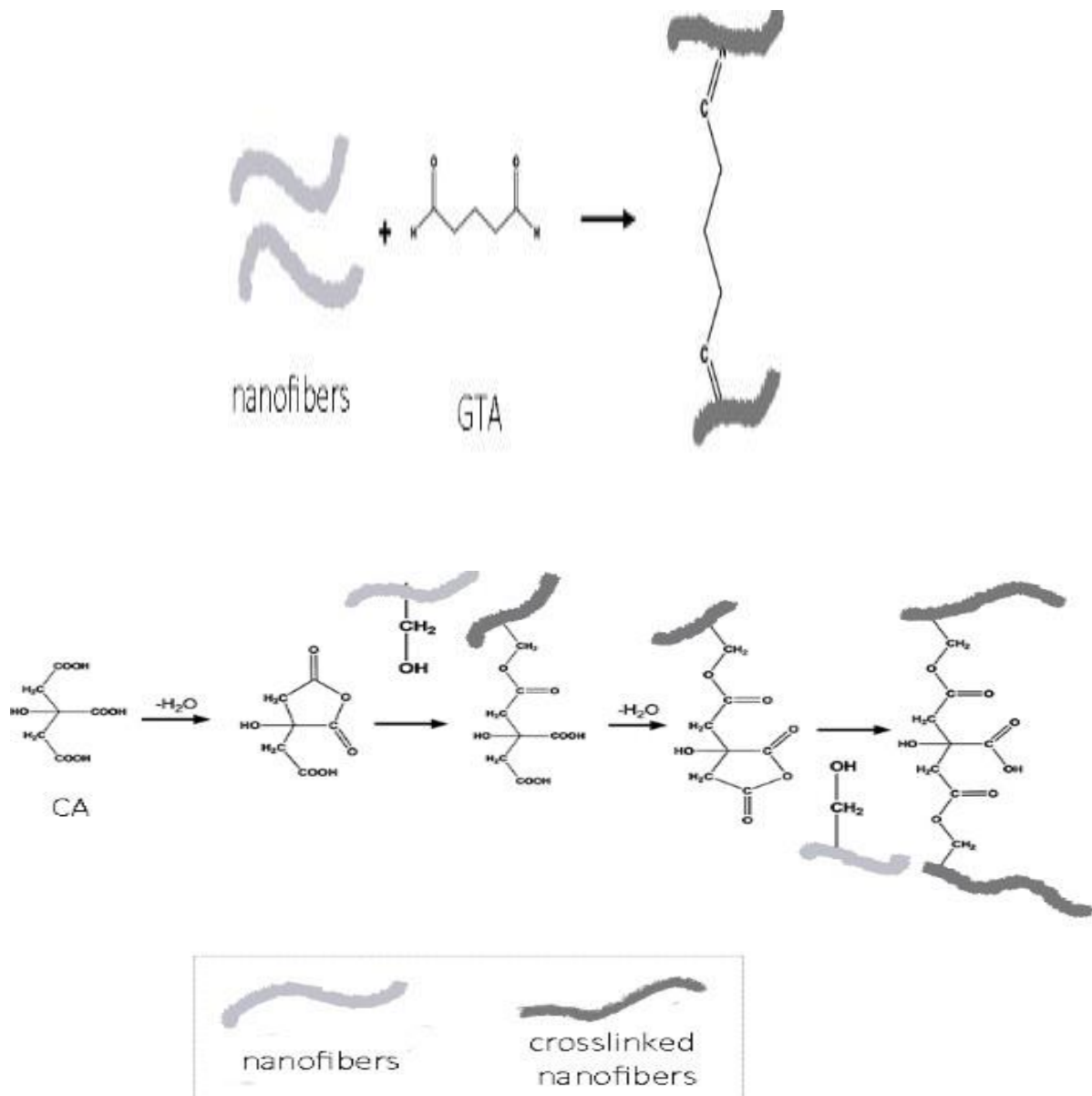


Figure 2.2 Potential Mechanism of crosslinking by Glutaraldehyde and Citric Acid (Katherine M. Dean, 2008)

There are three types of cross-linking therapies: chemical, biological, and physical. Dehydrothermal treatment (DHT), γ -radiation, ultraviolet (UV), and plasma exposure are all types of physical processes. High temps, like 140 °C for one day, break down the crosslinks between as-spun collagen nanofibers (Dean & Petinakis, 2008). Only DHT treatment worked less well than using it alone or with a chemical cross-linking agent like EDC (1,3-dimethyl aminopropyl)-carbodiimide). Isocyanurate-containing PLLA electrospun nanofibers crosslink when exposed to γ -radiation (Bhanu et al., 2017).

Under UV light, photochemically crosslinked collagen nanofibers that had been changed. Low-pressure air plasma was used to crosslink polysuccinimide nanofibers that had been changed with alylamine (Kaftan and Acikel, 2005). A gelatin nanofiber was joined together by plasma from a DBD at room temperature. Microbial transglutaminase (mTG) is a safe enzyme that is often used to improve the smoothness and stability of products. Most of the time, chemical cross-linking is used to connect electrospun supports (Li et al., (2009)). Most of the time, natural or man-made cross-linking agents are used. Glutaraldehyde (GTA) is a well-known man-made crosslinker, and genipin (GP) from Gardenia fruits is also well-known.

2.2.2.1 Glutaraldehyde

GTA is a bifunctional agent that, because of its very reactive aldehyde groups, may form connections with a wide variety of groups, including amines, thiols, phenols, hydroxyl (Choi, et al., 2016). Because of its chemical reaction with proteins' lysine or hydroxylysine amine groups, GTA can be used as a general cross-linking agent for proteins. The reason behind this is that amines are highly bound to GTA. Amine crosslinking mediated by GTA typically occurs in neutral to basic environments (Li et al., (2009)). GTA outperforms competing aldehydes due to the thermal and chemical stability of the crosslinked forms it produces.

Due to its inexpensive cost and excellent efficacy in stabilising scaffolds, GTA has seen extensive use. Because GTA-crosslinked tissues are resistant to biological degradation, it has been used to make bioprostheses including heart valves, vascular grafts, and elastic cartilages (Li et al., (2009)). Bioprosthetic heart valves (BHVs) made using GTA-mediated techniques using porcine tissues are one example (Choi, et al., 2016). However, due to the undesirable calcification that happens in treated tissues, bioprostheses do not endure as long following GTA treatment (Choi, et al., 2016). Adding amino acids and citric acid (CA) to GTA's treatment further decreases its cytotoxicity. Proteins, lipids, and free calcium in the bloodstream after implantation are thought to have the ability to trigger calcification (Hulupi and Haryadi, 2019). This can be mitigated by removing these chemicals from the donor's tissue using reagents such as ethanol or solutions based on ethanol (Li et al., (2009)).

Figure 2.4 Different evaporation conditions affect crosslinked fibre morphology (Source: Li et al., (2009))

Moreover, GTA-treated electro spun scaffolds showed negligible in-vitro cytotoxicity and great biocompatibility (Li et al., (2009)). The amount of GTA used, the scaffolds in need of repair, and the treatment conditions all influence the final quality of electro-spun scaffolds treated with GTA. Morphological integrity was determined to be exceptional in a GTA vapour environment, as seen in Figure 2.3. In this study, silica gel particles were added to the GTA- vaporized atmosphere to reduce humidity (Choi, et al., 2016).

2.2.2.2 Citric Acid

Citrus fruits are a source of organic tricarboxylic acid or CA. Lemons, limes, and other common citrus fruits have about 8% CA by weight when dry. As a flavouring, sequestering, or buffering agent, CA is an essential component in numerous sectors (Wang et al., 2009). Hulupi and Haryadi (2019) further noted that CA has been investigated for its possible use as a biocompatible elastomeric material's flexible monomer. Poly (diol citrate) synthesis and investigation. Utilizing three carboxyl and one hydroxyl functional group of CA, we synthesized poly (1,8-octanediol-co-citric acid) (POC) and tested it under mild circumstances to determine its potential for use in tissue engineering (Uranga et al., 2020).

Table 2.1 shows that CA might be used as a mild cross-linking agent in many different types of materials for industrial purposes in fields like food, medicine, cosmetics, tissue engineering, and environmental science (Ma et al., 2016). Biocompatible and edible films are made more effective with CA. Research shows that starch films are great for frying since they are 1.5 times stronger after CA crosslinking. A decreased end hydroxy group is one of three found on each anhydro glucose monomer in starch. According to Wang et al. (2009), it is possible to esterify starch to create CA mono-, di-, and tri-esters. High temperatures (90-105°C) were used to create films of CA-cured aga/fish gelatin. The use of starch films in culinary applications is enhanced by crosslinking them with CA (Hulupi and Haryadi, 2019).

Table 2.1 Current Applications of Citric Acid

Applications	Descriptions	Ref.
Pharmaceutically active substances, pharmaceuticals, personal care, and cosmetic products	Citric acid is utilized to form citrate salts of active pharmaceutical ingredients (APIs) and is employed to mask the bitter taste of drugs.	Uranga et al., (2020)
Food and beverage	Citric acid enhances the activity of antioxidant preservatives serve as an acidulant and pH	Vashisth And

	stabilizer, and contributes to the sharp taste of certain foods.	Pruthi, (2016)
Flavoring agent	The sharp and acidic taste of citric acid makes it a valuable flavouring agent, particularly in masking the unpleasant taste of pharmaceuticals.	Gao et al., (2014)
Blood anticoagulant	Citrate chelates calcium in the blood, reducing clotting tendencies and serving as an anticoagulant.	Li et al., (2009)
Environmental Remediation	Citrus acid is useful for chelating heavy metals (including radioactive isotopes) and removing hydrophobic organic compounds.	Rowland et al., (2013)

2.3 Conceptual framework

The conceptual framework for synthesising and characterising PVA starch films utilising glutaraldehyde and citric acid as crosslinkers includes the essential aspects and connections involved in the research process (Ma et al., 2016). The study incorporates principles from polymer chemistry, materials science, and sustainable packaging to direct the examination and understanding of experimental findings (Hulupi and Haryadi, 2019). The conceptual framework clarifies the impact of selecting crosslinking agents, synthesis methods, and characterisation techniques on the properties of PVA starch films. This, in turn, determines their prospective uses in various industries (Nataraj et al., 2020).

The biodegradable PVA and starch provide the fundamental basis of the conceptual framework (Li et al., 2009). These polymers include favourable characteristics such as biocompatibility, the capacity to form films, and the ability to be sourced from renewable elements, which makes them highly attractive options for sustainable packaging materials. The inherent features and potential limits of PVA starch films serve as the basis for their synthesis and modification (Hulupi and Haryadi, 2019).

The conceptual framework includes the choice of crosslinking agents, specifically glutaraldehyde and citric acid. Glutaraldehyde reacts with hydroxyl groups in PVA and starch molecules by covalent bonding, resulting in improved structural integrity and mechanical strength (Li et al., 2009). Citric acid provides an environmentally benign option by enabling the formation of crosslinks through esterification processes, while still retaining the potential to be broken down naturally (Wang et al., 2009). The selection of a crosslinking agent has a significant impact on the characteristics and effectiveness of the PVA starch films that are produced.

Different synthesis techniques are utilized to produce PVA starch films with crosslinking agents. The procedures encompass solution casting, blending, and solvent evaporation techniques. Every approach has an impact on the distribution of crosslinking agents in the polymer matrix, which in turn influences the shape, structure, and characteristics of the film (Wang et al., 2009). The conceptual framework involves the careful selection and optimization of synthesis processes to get specific film features (Ma et al., 2016).

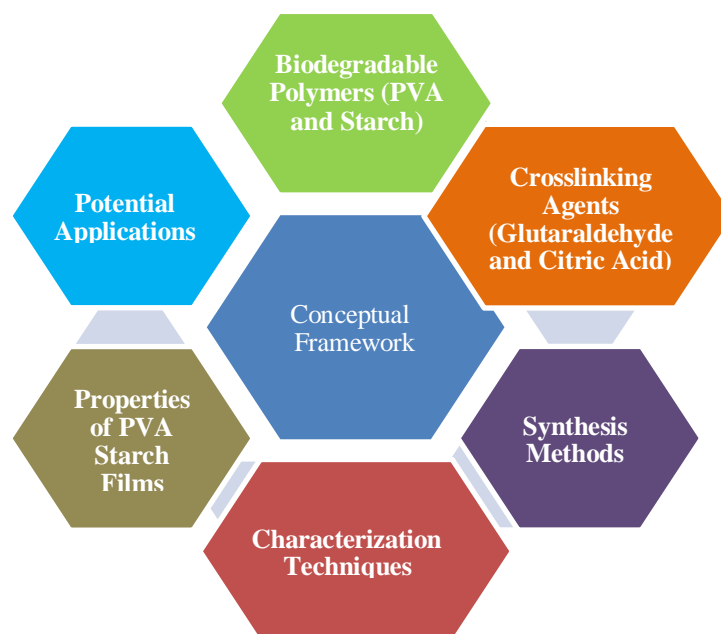


Figure 2.3 Model of the conceptual framework

It incorporates characterization methodologies for the analysis of the properties of PVA starch films. FTIR spectroscopy reveals the chemical interactions occurring between polymer chains and crosslinking agents. XRD is a technique that may be used to analyse changes in the structure of crystals. On the other hand, SEM is a method that allows for the investigation of the surface morphology of films (Bhanu et al., 2017). The interplay between crosslinking agents and polymer chains significantly impacts these properties, resulting in fluctuations in film performance. It is crucial to comprehend the connection between synthesis parameters, crosslinking mechanisms, and film qualities to enhance film features for applications (Ma et al., 2016). It examines the possible uses of PVA starch films in packaging, biomedical materials, and environmental remediation, as discussed by Hulupi and Haryadi in 2019. By implementing crosslinking, it becomes possible to modify the characteristics of films to meet a wide range of specific application needs. The synthesis and characterization of PVA starch films have the potential to enable many applications such as sustainable packaging solutions, improved biomedical compatibility, and the use of ecologically benign materials for remediation efforts (Bhanu et al., 2017). It offers a systematic method for comprehending the synthesis, characterization, and utilization of PVA starch films with the aid of glutaraldehyde and citric acid as crosslinking agents (Ma et al., 2016). The text explores the relationship between polymer chemistry, materials science, and sustainability concepts. It provides guidance for research aimed at creating environmentally friendly packaging materials that have specific features and enhanced performance (Choi, et al., 2016). It incorporates characterization methodologies for the analysis of the properties of PVA starch films. FTIR spectroscopy reveals the chemical interactions occurring between polymer chains and crosslinking agents. XRD is a technique that may be used to analyse changes in the structure of crystals. On the other hand, SEM is a method that allows for the investigation of the surface morphology of films (Bhanu et al., 2017). It offers a systematic method for comprehending the synthesis, characterization, and utilization of PVA starch films with the aid of glutaraldehyde and citric acid as crosslinking agents (Ma et al., 2016). The text explores the relationship between polymer chemistry, materials science, and sustainability concepts. It provides guidance for research aimed at creating environmentally friendly packaging materials that have specific features and enhanced performance (Choi, et al., 2016).

Table 2.2 Sources available on PVA and Starch films

Serial No.	Name of Publication	Materials Used	Applications	Limitations
1	Bhanu et al., (2017)	PVA, Starch, Glutaraldehyde	Food packaging, Biomedical materials	Poor mechanical properties
2	Choi, et al., (2016)	PVA, Starch, Citric Acid	Environmental remediation, Packaging	Limited moisture resistance
3	Li et al., (2009)	PVA, Starch, Glutaraldehyde	Biodegradable films, Food packaging	High cost of crosslinking agents
4	Nataraj et al., (2020)	PVA, Starch, Citric Acid	Biomedical applications, Food packaging	Complexity in synthesis methods
5	Ma et al., (2016)	PVA, Starch, Glutaraldehyde	Agricultural films, Soil remediation	Environmental concerns regarding biodegradability
6	Penniston, (2008)	PVA, Starch, Citric Acid	Pharmaceutical packaging, Biomedicals	Limited scalability for industrial Applications
7	Teixeira et al., (2021)	PVA, Starch, Glutaraldehyde	Sustainable packaging, Food industry	Poor adhesion to certain substrates
8	Uranga et al., (2020)	PVA, Starch, Citric Acid	Water treatment, Environmental remediation	Long-term stability concerns
9	Vashisth and Pruthi, (2016)	PVA, Starch, Glutaraldehyde	Textile coatings, Industrial applications	Challenges in achieving uniform crosslinking
10	Gao et al., (2014)	PVA, Starch, Citric Acid	Biodegradable packaging, Agriculture	Limited shelf-life due to degradation

Current literature offers a thorough examination of the production, analysis, and uses of PVA starch films made with glutaraldehyde and citric acid as crosslinking agents. Although PVA starch films show promise in multiple sectors, such as food packaging, biomedical materials, and environmental remediation, they also have notable disadvantages (Li et al., 2009). These include subpar mechanical properties, limited resistance to moisture, expensive crosslinking agents, and difficulties in synthesis methods. Although there are certain limits, the overall results indicate that PVA starch films have considerable potential as environmentally friendly packaging materials that may be customised for specific uses (Nataraj et al., 2020).

2.4 Chapter Conclusion

This chapter explores the process of creating and analysing PVA starch films by utilising glutaraldehyde and citric acid as crosslinking agents. The text provides an introduction to the

research background and importance, and then proceeds to detail the specific goals and extent of the study. The historical backdrop provides a foundation for understanding the development of research on sustainable materials, while the theoretical framework combines ideas from polymer chemistry and sustainability. The empirical review provides a concise summary of prior investigations, while the conceptual framework delineates the fundamental components that direct the study process. The chapter offers a thorough basis for comprehending the synthesis procedures, characterisation methodologies, and prospective uses of PVA starch films in sustainable packaging and other industries.

CHAPTER 3

EXPERIMENTAL

The study approach for synthesizing and characterizing PVA-Starch films employing glutaraldehyde and citric acid as crosslinkers comprises many essential phases. Initially, the desired proportions of polyvinyl alcohol (PVA) to starch are created, ranging from a ratio of 1:1 to 3:1. Different quantities of PVA and starch are dissolved in water to achieve these ratios. Subsequently, each combination undergoes sonication to achieve uniformity (Musa and Hameed, 2021). Subsequently, the solutions are supplemented with crosslinking agents, namely glutaraldehyde or citric acid (Edgar et al., 2021). Glutaraldehyde-crosslinked films require the addition of 0.5 ml of glutaraldehyde, whereas citric acid-crosslinked films necessitate the use of 0.1 gram of citric acid. Next, the solutions are agitated using a magnetic stirrer to enhance the process of crosslinking (Bhanu et al., 2017).

Following the process of cross-linking, the solutions are poured into Molds and subsequently let to dry, resulting in the formation of thin films. Subsequently, these films undergo FTIR analysis in order to determine and describe their chemical makeup (Edgar et al., 2021). In addition, XRD analysis is conducted to evaluate the crystalline structure of the films. The acquired data are examined to assess the impact of crosslinking agents and PVA-to-starch ratios on the properties of the film (Dean and Petinakis, 2008). This methodology offers a systematic approach to synthesizing and analyzing PVA-Starch films, providing valuable insights into their potential uses in several disciplines (Edgar et al., 2021).

3.1 Materials and Reagents:

The process of creating and analyzing PVA-Starch films requires the use of essential components and reagents. These substances consist of PVA, which is selected for its ability to create films, and Starch, which functions as a component that can naturally break down. In addition, Glutaraldehyde and Citric Acid are used as crosslinking agents to improve the stability of the film (Edgar et al., 2021). Distilled water is employed as a solvent to effectively dissolve PVA, starch, and crosslinkers, hence ensuring the achievement of a uniform mixture. Every individual part has a vital function in the manufacturing procedure, adding to the overall strength and characteristics of the PVA-Starch films that are produced (Gadhve et al., 2019).

3.2 Preparation of Blends

To produce PVA-Starch films, separated solutions of PVA and Starch are initially made by dissolving them individually in distilled water. The PVA to Starch ratio is variable and depends on the desired film composition. Ratios of 1:1, 2:1, and 3:1 are used (Gadhve et al., 2019). Subsequently, these solutions are combined in the designated proportion and subjected to sonication for a duration of 30 minutes to achieve homogeneity. Afterwards, in films that use glutaraldehyde as a crosslinker, 0.5 ml of glutaraldehyde was introduced into the PVA-Starch mixture and agitated for a duration of 30 minutes (Gao et al., 2014). In contrast, films that make use of citric acid incorporate 0.1 grams of citric acid as an alternative. The careful and thorough preparation process guarantees that the produced films.

TABLE 3.1 Compositions of PVA Starch films with varying amounts PVA and Starch

	1:1	2:1	3:1
PVA	0.5 g	0.66 g	0.75 g
STARCH	0.5 g	0.33 g	0.25 g

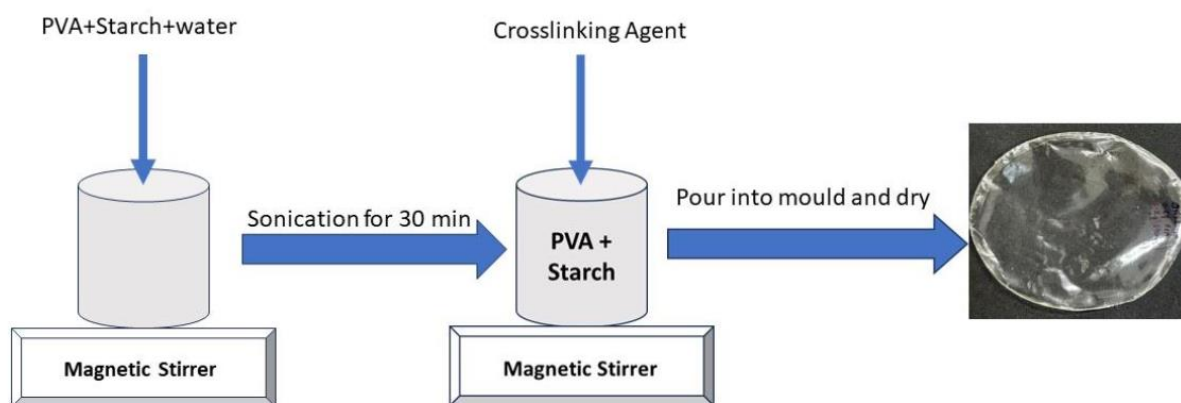


Figure 3.1 Schematic diagram of Blending

3.3 Film Casting

Once the PVA-Starch-crosslinker mixture is ready, it is placed onto a flat surface, such as a glass plate, and spread uniformly to create thin films. This casting procedure guarantees even dispersion of the mixture, which enables constant film characteristics (Edgar et al., 2021). Afterwards, the films are allowed to air dry at the ambient temperature for a specific duration. During the process of drying, the solvent evaporates, resulting in the creation of solid films that possess the desired qualities (Musa and Hameed, 2021). Performing this procedure is essential in order to facilitate the advancement of stable PVA-Starch films, enabling subsequent examination and evaluation of their attributes and potential uses (Choi et al., 2016).

The study approach for synthesizing and characterizing PVA-Starch films employing glutaraldehyde and citric acid as crosslinkers comprises many essential phases. Initially, the desired proportions of polyvinyl alcohol (PVA) to starch are created, ranging from a ratio of 1:1 to 3:1. Different quantities of PVA and starch are dissolved in water to achieve these ratios. Subsequently, each combination undergoes sonication to achieve uniformity (Musa and Hameed, 2021). Subsequently, the solutions are supplemented with crosslinking agents, namely glutaraldehyde or citric acid (Edgar et al., 2021). Glutaraldehyde-crosslinked films require the addition of 0.5 ml of glutaraldehyde, whereas citric acid-crosslinked films necessitate the use of 0.1 gram of citric acid. Next, the solutions are agitated using a magnetic stirrer to enhance the process of crosslinking (Bhanu et al., 2017).

Following the process of cross-linking, the solutions are poured into Molds and subsequently let to dry, resulting in the formation of thin films. Subsequently, these films undergo FTIR analysis in order to determine and describe their chemical makeup (Edgar et al., 2021). In addition, XRD analysis is conducted to evaluate the crystalline structure of the films. The acquired data are examined to assess the impact of crosslinking agents and PVA-to-starch ratios on the properties of the film (Dean and Petinakis, 2008). This methodology offers a systematic approach to synthesizing and analyzing PVA-Starch films, providing valuable insights into their potential uses in several disciplines (Edgar et al., 2021).

3.4 Chapter Conclusion

Chapter 3 provides a comprehensive explanation of the synthesis methods employed to fabricate PVA-starch films. The films were synthesized using 2 crosslinking agents, glutaraldehyde and citric acid, and different ratios of PVA and Starch. The synthesis process involves preparing PVA and Starch solutions, mixing them, and adding a crosslinker, followed by casting and drying the films.

CHAPTER 4

CHARACTERISATION

To assess the qualities and potential uses of the synthesized films, they undergo extensive characterization. The chemical structure and functional groups in the films can be examined using FTIR, which provides useful information about their composition (Musa and Hameed, 2021). Using an SEM, researchers can examine the film's surface morphology and learn more about its structure and topography (Bhanu et al., 2017). Furthermore, tensile strength tests are conducted to measure mechanical properties including strength and elasticity, which are crucial for evaluating the film's performance. A thorough understanding of the chemical, morphological, and mechanical properties of the synthesized PVA-Starch films is attained using different characterization methods; this understanding sheds light on their potential applications (Choi et al., 2016).

4.1 FTIR

Infrared spectroscopy examines how infrared light interacts with a molecule. It measures absorption, emission, and reflection in three different ways. This method is primarily used in organic and inorganic chemistry to identify functional groups in molecules. Functional groups can be identified via IR spectroscopy, which monitors atom vibrations.

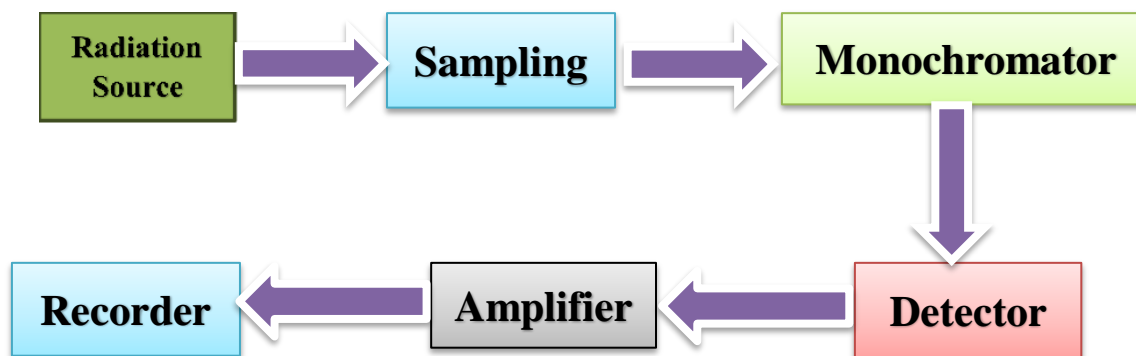


Figure 4.1 Schematic diagram of FTIR

4.2 XRD

X-ray diffraction is a popular method for determining a sample's composition or crystalline structure. It can be used to ascertain the atom-by-atom structure of bigger crystals, like macromolecules and inorganic compounds. If the crystal size is too small, it can reveal phase purity, composition, and crystallinity in a sample. X-ray beams are passed through the sample using this method

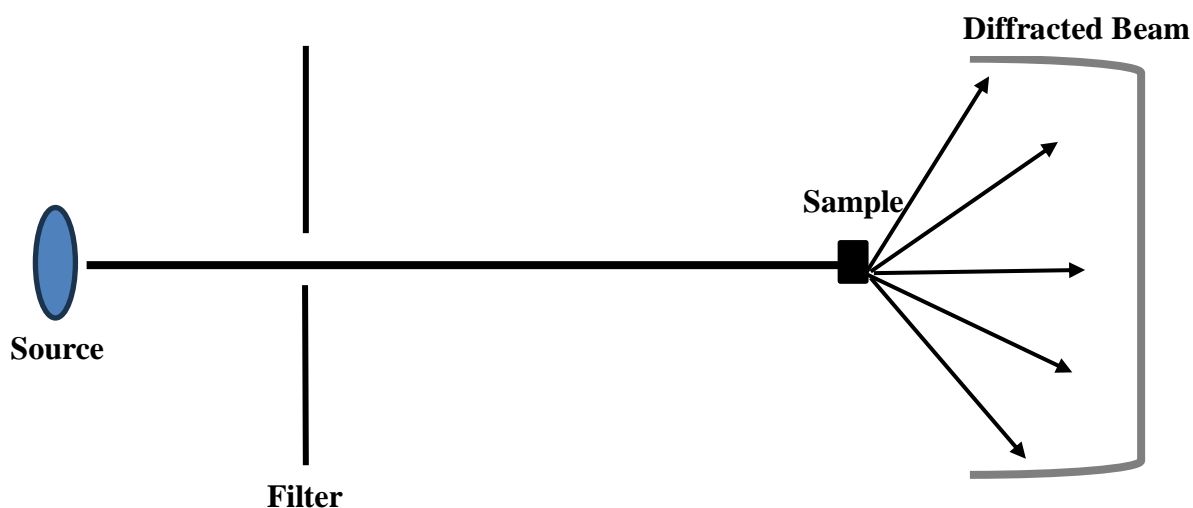


Figure 4.2 Schematic diagram of XRD

4.3 Yellowish Test

The yellowish test stands as a cornerstone in the arsenal of qualitative assessment methods across scientific domains, prominently featured in chemical analysis and materials science. Its application resonates particularly in swiftly discerning between substances with disparate chemical compositions, notably citric acid, and glutaraldehyde. At its core, the yellowish test involves subjecting a film or solution containing either citric acid or glutaraldehyde to close observation. When citric acid prevails, the film typically transitions to a yellow hue. This transformation owes itself to the unique chemical makeup of citric acid, triggering reactions with the surrounding milieu. Interactions between citric acid molecules and their environment give rise to the formation of yellow-hued compounds or complexes, thereby manifesting the observable change.

4.4 Chapter Conclusion

Chapter 4 provides information about the characterization techniques employed for film characterization. It compares film properties and clarifies the impacts of crosslinking agents and composition ratios. In summary, the methodical methodology outlined in this chapter enhances comprehension of PVA-Starch film synthesis and characterization, hence opening possibilities for their future utilization in diverse sectors.

CHAPTER 5
RESULTS
&DISCUSSION

PVA Starch films were synthesized using two crosslinking agents i.e. Glutaraldehyde and Citric Acid. The synthesized films were characterized using FTIR and XRD and yellowish test was performed in order to determine the tendency of the films to turn yellow. The structural and chemical properties of PVA Starch films were investigated through the synthesis and characterization process, which involved the use of glutaraldehyde and citric acid as crosslinkers. The FTIR measurement showed distinct peaks corresponding to PVA and starch in all films, confirming effective synthesis. Modulating the ratio of PVA to starch led to modifications in peak intensities, indicating shifts in the composition of the film. Films treated with glutaraldehyde and citric acid exhibited FTIR spectra that were identical, indicating equivalent levels of crosslinking efficiency. The baseline spectra verified the stability of the PVA and starch constituents in the absence of cross-linking.

The results highlight the significance of crosslinking agents in controlling the characteristics of the film. Potential research endeavors may investigate the mechanical characteristics and potential uses of these films in the realms of biodegradable packaging and biomedical applications. Overall, this research enhances our comprehension of the possible applications of crosslinked PVA Starch films, which may be adjusted to meet specific requirements and are environmentally sustainable.

5.1 FTIR spectra of PVA-Starch films crosslinked using Citric Acid

The FTIR spectra graph displays transmittance (%) against wavenumber (cm^{-1}) for five samples: C3, C2, C1, Starch, and PVA. The comparison reveals distinctive spectral features. C3, C2, and C1 exhibit similar absorption peaks, indicating structural similarity. Starch shows characteristic peaks suggesting its molecular composition. PVA displays unique absorption bands, distinguishing it from the other samples. Variations in peak intensity and position signify differing chemical compositions and structural arrangements among the samples. This analysis aids in identifying and distinguishing the materials based on their infrared spectra, crucial for various applications in fields like materials science, chemistry, and biochemistry.

C1- Mixture of 0.5 g PVA+ 0.5 g Starch+ 0.1g Citric acid

C2- Mixture of 0.66 g PVA+ 0.33 g Starch+ 0.1g Citric acid

C3- Mixture of 0.75 g PVA+ 0.25 g Starch+ 0.1g Citric acid

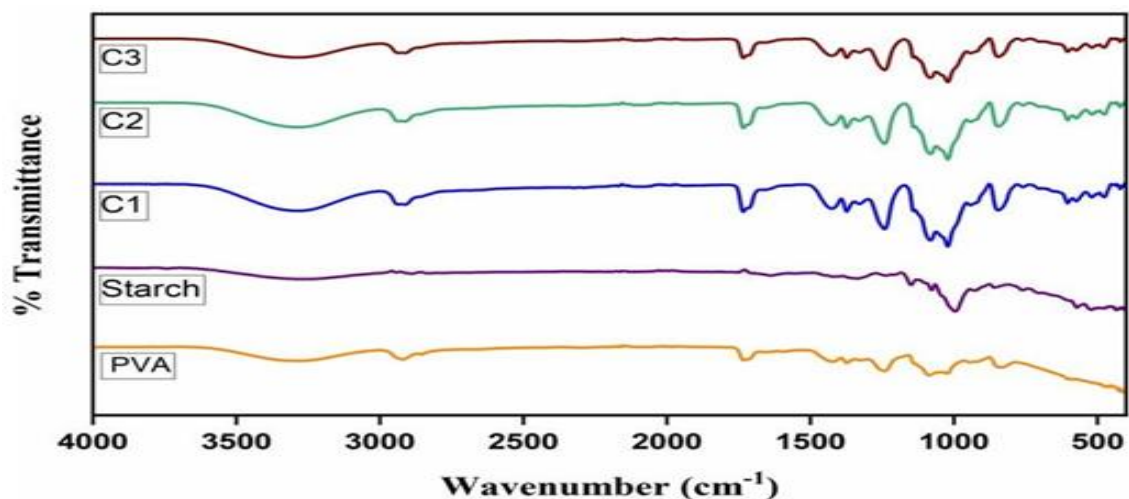


Figure 5.1 FTIR spectra of PVA-Starch films crosslinked using Citric Acid

- The FTIR spectra display peaks corresponding to various functional groups present in the samples.
- The patterns of transmittance across the wavenumber range indicate the different chemical structures and compositions of the samples.

PVA (Polyvinyl Alcohol)

- **Broad peak around 3300 cm⁻¹:** Indicates O-H stretching, typical of hydroxyl groups in PVA.
- **Peak around 2900 cm⁻¹:** C-H stretching vibrations.
- **Peak near 1700 cm⁻¹:** C=O stretching, suggesting some degree of oxidation or esterification.
- **Peaks between 1400-1000 cm⁻¹:** Various C-H bending and C-O stretching vibrations.

Starch

- **Broad peak around 3300 cm⁻¹:** O-H stretching, indicative of hydroxyl groups in starch.
- **Peak around 2900 cm⁻¹:** C-H stretching vibrations.
- **Strong peak near 1000 cm⁻¹:** C-O stretching, characteristic of polysaccharides like starch.
- **Differences from PVA:** Starch has more pronounced peaks in the 1000-1100 cm⁻¹ region, which are typical for glycosidic linkages in polysaccharides.

C1

- **Broad peak around 3300 cm⁻¹:** Similar O-H stretching indicating hydroxyl groups.
- **Distinct peaks in the 1600-1500 cm⁻¹ range:** Likely correspond to N-H bending or amide I and II bands, suggesting the presence of proteins or peptides.
- **Peaks around 1000-1200 cm⁻¹:** Indicate C-O stretching, possibly from carbohydrates or other oxygen-containing groups.

C2

- **Broad peak around 3300 cm⁻¹:** O-H stretching.
- **Distinct peaks in the 1600-1500 cm⁻¹ range:** Similar to C1, indicative of N-H bending or amide bands.
- **Differences from C1:** Slightly different peak intensities and positions in the 1000-1200 cm⁻¹ range, which might indicate variations in carbohydrate or other functional group compositions.

C3

- **Broad peak around 3300 cm⁻¹:** O-H stretching.
- **Notable peaks in the 1600-1500 cm⁻¹ range:** Indicative of amide groups.
- **Peaks in the 1000-1200 cm⁻¹ range:** Indicate C-O stretching, similar to C1 and C2 but with different intensities.

- **Hydroxyl Groups (O-H Stretching):** All samples exhibit broad peaks around 3300 cm^{-1} , indicating the presence of hydroxyl groups. This is most prominent in PVA and starch.
- **Carbohydrate Content:** Starch shows strong peaks in the $1000\text{-}1100\text{ cm}^{-1}$ region, typical for polysaccharides. C1, C2, and C3 also show peaks in this region but with different intensities, suggesting varying carbohydrate content.
- **Protein Content:** C1, C2, and C3 have distinct peaks in the $1600\text{-}1500\text{ cm}^{-1}$ range, indicative of amide groups, suggesting the presence of proteins or peptides.
- **Differences Among C1, C2, and C3:** While all three exhibit similar functional groups, their spectra indicate variations in composition, possibly due to differences in the types or amounts of proteins, peptides, or other components.

Analysis of the FTIR spectra indicates the presence of hydroxyl groups across all samples, implying a common structural feature. However, discernible differences emerge in carbohydrate and protein content among the samples. Starch and PVA exhibit prominent peaks indicative of their specific molecular compositions, allowing for clear identification. Conversely, while C1, C2, and C3 share some spectral similarities, distinct variations suggest differing chemical compositions among them. These differences may stem from variations in functional groups, molecular arrangements, or impurities present in the samples. Such nuanced discrepancies underscore the sensitivity of FTIR spectroscopy in characterizing materials at a molecular level.

The ability of FTIR to detect and differentiate between biochemical components within samples underscores its utility in various fields. By leveraging distinctive spectral signatures, researchers can elucidate the composition and structure of complex materials with precision. This analytical technique finds wide-ranging applications in fields such as pharmaceuticals, food science, and biomaterials research, facilitating quality control, compositional analysis, and material characterization. Thus, FTIR spectroscopy serves as a powerful tool for unraveling the intricate molecular makeup of diverse substances, contributing significantly to scientific understanding and technological advancements.

5.2 FTIR spectra of PVA-Starch films crosslinked using Glutaraldehyde

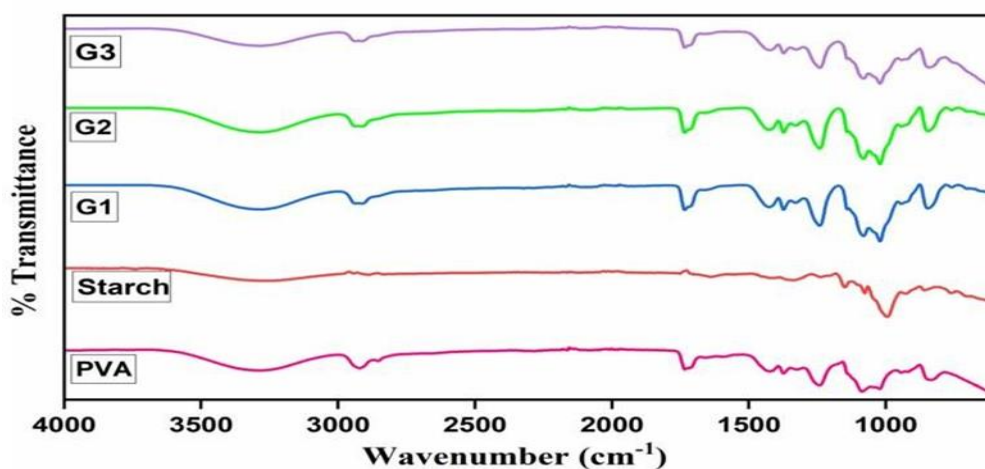


Figure 5.2 FTIR spectra of PVA-Starch films crosslinked using Glutaraldehyde

G1- Mixture of 0.5 g PVA+ 0.5 g Starch+ 0.5 ml Glutaraldehyde

G2- Mixture of 0.66 g PVA+ 0.33 g Starch+ 0.5 ml Glutaraldehyde

G3- Mixture of 0.75 g PVA+ 0.25 g Starch+ 0.5 ml Glutaraldehyde

The provided graph of the figure 5.2 shows the Fourier Transform Infrared (FTIR) spectra of five different samples: PVA (polyvinyl alcohol), Starch, G1, G2, and G3. Each spectrum is presented as a plot of % Transmittance versus Wavenumber (cm^{-1}). Each of the composite samples exhibits unique variations in peak positions and intensities, reflecting differences in their molecular interactions and compositions. These variations suggest that while all three samples share common features from PVA and starch, their specific molecular structures and interactions differ, resulting in distinct spectral characteristics. Here is a comparative analysis of the FTIR spectra:

PVA (Polyvinyl Alcohol):

- The spectrum shows characteristic peaks associated with PVA.
- Notable peaks include a broad peak around 3300 cm^{-1} (O-H stretching), indicating the presence of hydroxyl groups.
- Peaks around 2900 cm^{-1} (C-H stretching) and $1100\text{-}1000 \text{ cm}^{-1}$ (C-O stretching) are also visible.

Starch:

- The starch spectrum shows different peaks compared to PVA, indicating its unique molecular structure.
 - A broad O-H stretching peak around 3300 cm^{-1} is present but is slightly shifted compared to PVA.
 - Peaks around 2900 cm^{-1} (C-H stretching) and $1000\text{-}1200 \text{ cm}^{-1}$ (C-O stretching) are also observed.
 - There are additional peaks around 1640 cm^{-1} and 1450 cm^{-1} , which could be attributed to the molecular structure of starch.
-
- A broad O-H stretching peak around 3300 cm^{-1} is present but is slightly shifted compared to PVA.
 - Peaks around 2900 cm^{-1} (C-H stretching) and $1000\text{-}1200 \text{ cm}^{-1}$ (C-O stretching) are also observed.
 - There are additional peaks around 1640 cm^{-1} and 1450 cm^{-1} , which could be attributed to the molecular structure of starch.

G1:

- The G1 spectrum shows characteristics of both PVA and starch, suggesting it may be a blend or composite material.
- O-H stretching peak around 3300 cm^{-1} is present, similar to both PVA and starch.

- Peaks around 2900 cm⁻¹ (C-H stretching) and 1100-1000 cm⁻¹ (C-O stretching) are also seen.
- The peak patterns and intensities suggest a combination of features from PVA and starch.

G2:

- The G2 spectrum continues to show features of both PVA and starch but with some variations compared to G1.
- The O-H stretching peak around 3300 cm⁻¹ remains but may show slight differences in intensity or position.
- Peaks around 2900 cm⁻¹ (C-H stretching) and the region around 1000-1200 cm⁻¹ (C-O stretching) are present.
- Some additional peaks or shifts might indicate further chemical modifications or interactions within the material.

G3:

- The G3 spectrum shows the combined features of PVA and starch, similar to G1 and G2, but with its unique variations.
- O-H stretching around 3300 cm⁻¹ is present, consistent with the other samples.
- C-H stretching around 2900 cm⁻¹ and C-O stretching around 1000-1200 cm⁻¹ are also observed.
- Variations in peak intensities and positions might suggest different proportions or degrees of interaction between components in G3 compared to G1 and G2.
- **Similarity to Parent Materials:** All three composite samples (G1, G2, G3) show major peaks characteristic of both PVA and starch, indicating they are likely blends or composites.
- **Peak Shifts and Intensity Changes:** Variations in the spectra, such as peak shifts or changes in intensity, suggest different interactions between PVA and starch in G1, G2, and G3.
- **Identification of Unique Features:** Each composite sample (G1, G2, G3) has unique features in their FTIR spectra, possibly indicating different ratios, processing conditions, or additional modifications.

Overall, the FTIR spectra illustrate the presence of both PVA and starch in the composite samples (G1, G2, G3), with variations in their spectra indicating different interactions and compositions within each sample.

5.3 FTIR spectra of C3 and G3

Film C3 is composed of a 3:1 PVA: starch ratio, adding 0.1 grams of citric acid, and film G3 is composed of a 3:1 PVA: starch ratio, adding 0.5 ml of glutaraldehyde solution. These 2 films were chosen based on their clarity of FTIR peaks.

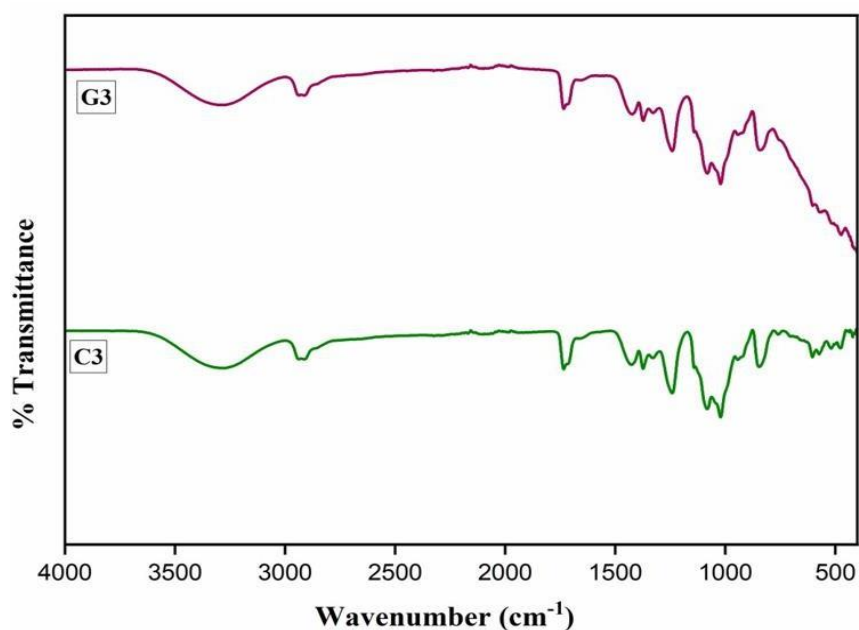


Figure 5.3 FTIR spectra of C3 and G3

The graph in Figure 4.3 displays the Fourier Transform Infrared (FTIR) spectra of two samples, namely G3 and C3. The spectra plot the transmittance percentage against wavenumber (cm^{-1}), enabling a comparative examination of the molecular compositions. Here we have a comparative analysis of FTIR Spectra:

G3:

- **O-H Stretching:** There is a broad peak around 3300 cm^{-1} , indicative of O-H stretching, which is characteristic of hydroxyl groups.
- **C-H Stretching:** Peaks around 2900 cm^{-1} suggest the presence of C-H bonds.
- **Fingerprint Region:** Between 1500 cm^{-1} and 500 cm^{-1} , several peaks can be observed, which correspond to various functional groups and bonds specific to the molecular structure of G3.
- **Notable Peaks:** Specific peaks are seen around 1650 cm^{-1} , 1450 cm^{-1} , and a broad region between 1100 cm^{-1} to 1000 cm^{-1} , which are typical for certain organic compounds and polymers.

C3:

- **O-H Stretching:** Similar to G3, C3 also shows a broad peak around 3300 cm^{-1} , indicating hydroxyl groups.
- **C-H Stretching:** Peaks around 2900 cm^{-1} , consistent with C-H bonds.
- **Fingerprint Region:** The fingerprint region (1500 cm^{-1} to 500 cm^{-1}) exhibits distinct peaks, suggesting different functional groups compared to G3.

- **Notable Peaks:** Peaks around 1650 cm⁻¹ and 1450 cm⁻¹ are observed, like G3, but with different intensities or slight shifts, indicating variations in molecular structure. Additional distinct peaks in the region between 1100 cm⁻¹ and 1000 cm⁻¹ suggest the presence of different or additional functional groups.

Observations:

- **Hydroxyl Groups (O-H Stretching):** Both G3 and C3 show a broad O-H stretching peak around 3300 cm⁻¹, indicating the presence of hydroxyl groups in both samples.
- **C-H Bonds:** Both spectra show peaks around 2900 cm⁻¹, characteristic of C-H stretching, suggesting the presence of similar types of carbon-hydrogen bonds in both samples.
- **Fingerprint Region Differences** The fingerprint regions (1500 cm⁻¹ to 500 cm⁻¹) of G3 and C3 display different patterns and intensities of peaks. This suggests variations in their molecular structures, possibly due to different functional groups or chemical compositions.
- **Distinct Peaks:** While both samples have peaks around 1650 cm⁻¹ and 1450 cm⁻¹, the specific nature and intensity of these peaks differ, indicating differences in their molecular environments.

The FTIR spectra of G3 and C3 reveal similarities in the presence of hydroxyl and C-H groups but show notable differences in the fingerprint region, reflecting variations in their molecular structures and functional groups. These differences are crucial for understanding the distinct properties and potential applications of each sample.

1. Both citric acid and glutaraldehyde-crosslinked films exhibit characteristic peaks corresponding to PVA and starch.
2. Varying the PVA to starch ratio influences peak intensities, indicating changes in film composition.
3. Baseline spectra confirm the stability of PVA and starch components without cross-linking.

Crosslinking Efficiency:

Citric acid and glutaraldehyde effectively crosslink PVA and starch, stabilizing the film structure.

Similarities in peak positions and intensities suggest comparable crosslinking efficiency between the two agents.

Effect of PVA to Starch Ratio:

Films with higher PVA to starch ratios show increased intensity in peaks corresponding to PVA, indicating a higher concentration of PVA in the film matrix. Adjusting PVA to Starch Ratio allows for modulation film composition.

Chemical Stability:

Baseline spectra of all films show no significant chemical changes in the absence of crosslinking,

indicating chemical stability.

Structural Integrity:

The presence of characteristic peaks in both normal and baseline spectra suggests that the film structure remains intact regardless of cross-linking.

PVA-Starch films include adjustable characteristics that render them highly favorable for various uses, such as biodegradable packaging and biomedical applications.

Among all the samples, the most suitable candidates for XRD examination are the G3 and C3 films. These films demonstrate a greater magnitude of PVA-associated peaks, suggesting a higher amount of PVA present in the film structure. Higher levels of PVA may result in variations in crystallinity, which can be investigated more thoroughly utilizing XRD analysis. Moreover, the resemblance in the locations and strengths of the highest points between G3 and C3 films implies that their ability to form chemical bonds is similar. This similarity allows for a comparative examination of their structural characteristics using X-ray diffraction (XRD). Further analysis of these films using X-ray diffraction (XRD) may improve our comprehension of the correlation between the composition, crosslinking, and crystallinity in PVA-Starch films.

5.4 XRD Result Analysis

XRD is an effective method for analyzing the crystallographic arrangement, chemical makeup, and physical attributes of materials. This analysis uses the given measurement settings and peak data to examine the XRD results of three distinct samples, C3, G3, and S3.

Measurement Conditions

The measuring conditions remained consistent for all samples (C3, G3, and S3). The X-ray source functioned at an operating voltage of 40 kilovolts and a current of 30 milliamperes, while the scanning speed was set at 8.0000 degrees per minute. The goniometer was configured with a step width of 0.0200 degrees, and the scan axis was set to 2theta/theta. A K-beta filter was employed to reduce background noise and enhance the signal-to-noise ratio.

The scanning range was configured from 10.0000 to 80.0000 degrees, enabling a thorough examination of the diffraction peaks. The slit configurations consisted of a CBO selection slit set to BB, an incident slit set at an angle of 2/3 degrees, and a receiving slit arrangement with #1 set at an angle of 2/3 degrees and 2 set at a width of 0.3 mm. A scintillation counter was employed as the detector, and the data acquisition was performed in continuous scan mode to ensure uninterrupted data collection.

5.4.1 The Powder XRD pattern of PVA and starch are presented in Fig. 5.4 and Fig. 5.5

In the Powder XRD pattern of PVA, a singular peak at 19.84° can be observed. This peak is the characteristic peak of PVA and the slight sharpness of this peak corresponds to the semi-crystalline nature of PVA.

Figure 5.5 shows the Powder XRD pattern of starch. Here, the presence of a broad peak around 24° corresponds to semi-crystalline starch.

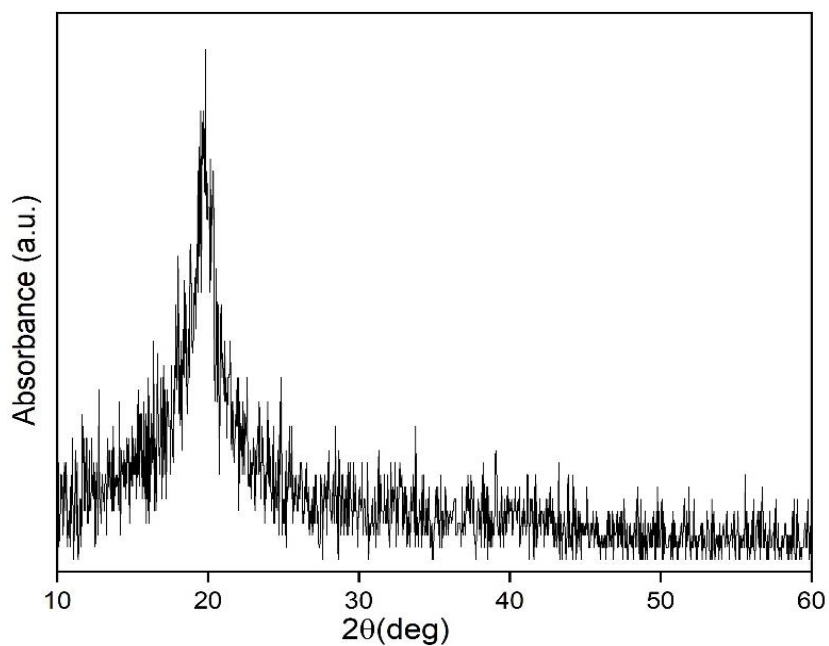


Figure 5.4 Powder XRD pattern of PVA

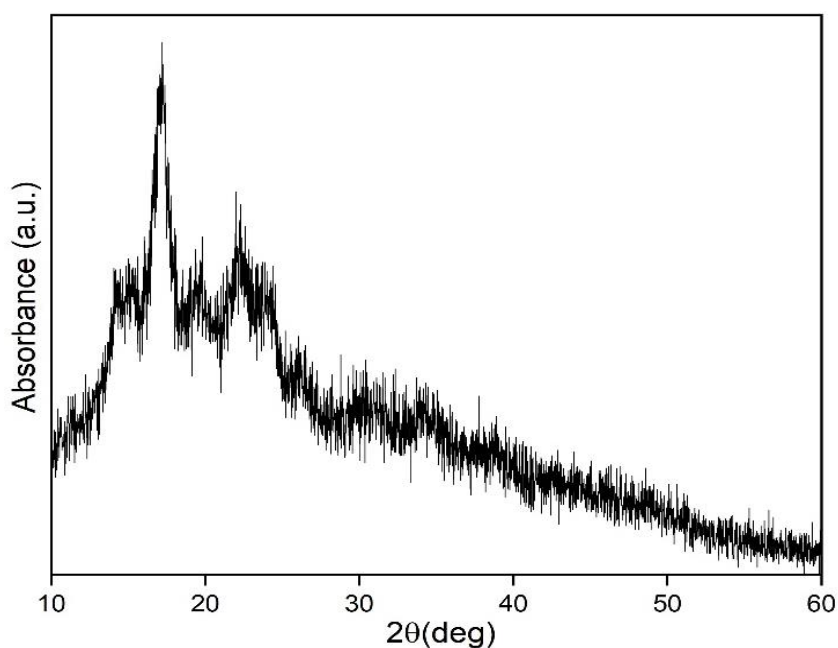


Figure 5.5 Powder XRD pattern of Starch

5.4.2 Powder XRD pattern of citric acid-crosslinked film C3 is presented in figure. 5.6

In comparison to pure PVA and starch, the film exhibits broad peaks around 18.2° and 22.86°. The disappearance of characteristic peaks of PVA indicates the loss of semi-crystalline nature of PVA and its transformation into amorphous phase due to interaction with starch in the PVA matrix.

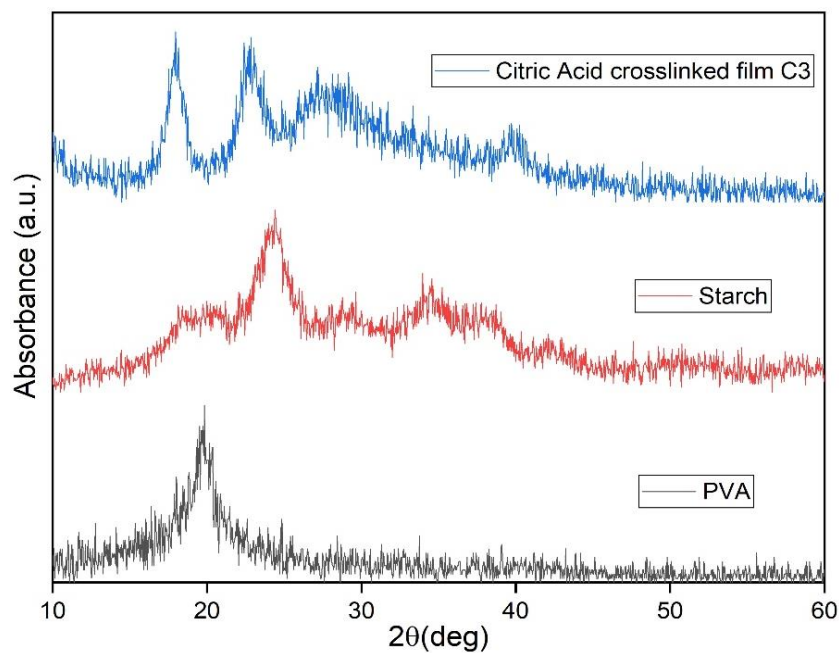


Figure 5.6 Powder XRD pattern of C3

5.4.3 Powder XRD pattern of citric acid-crosslinked film G3 is presented in figure. 5.7

Similarly, in case of glutaraldehyde-cross linked film G3, disappearance of characteristic peak of PVA along with the appearance of broad low-intensity peaks indicates that semicrystalline nature of PVA is lost and amorphous nature is increased in the film.

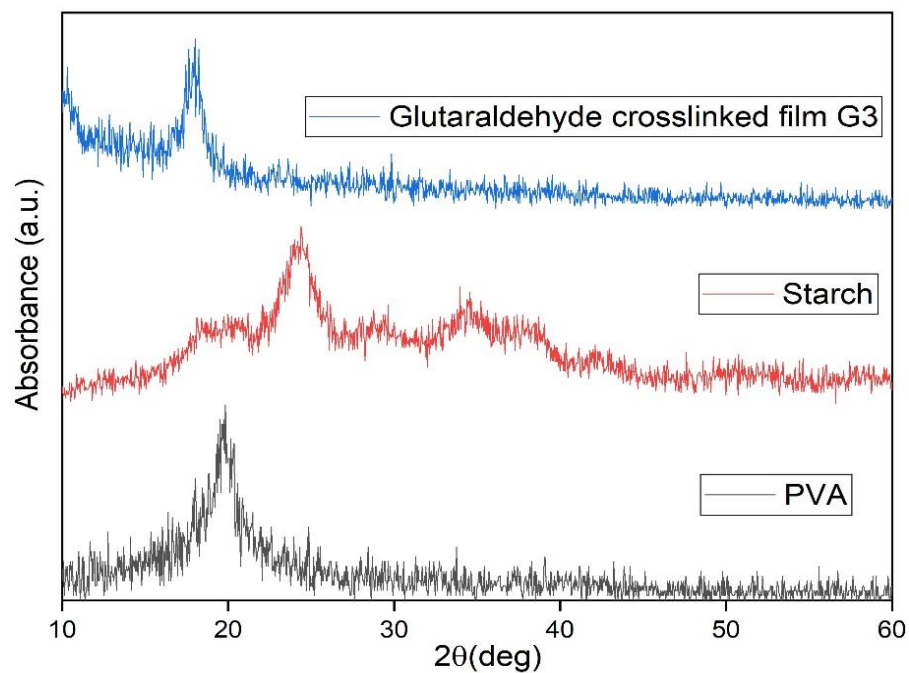


Figure 5.7 Powder XRD pattern of G3

Additionally, the appearance of more peaks in the range 15° to 25° in pattern of C3 indicates higher degree of crystallinity as compared to that in G3.

5.5 Yellowish Test Analysis

When citric acid prevails, the film typically transitions to a yellow hue. This transformation owes itself to the unique chemical makeup of citric acid, triggering reactions with the surrounding milieu. Interactions between citric acid molecules and their environment give rise to the formation of yellow-hued compounds or complexes, thereby manifesting the observable change.

In contrast, the presence of glutaraldehyde in the test solution often does not prompt a rapid yellowing response. Glutaraldehyde, distinguished by its distinct functional groups and chemical properties, typically eschews the yellowish metamorphosis under analogous conditions. Instead, its presence may be inferred through other discernible alterations or reactions characteristic of glutaraldehyde's chemistry.

The yellowish test's hallmark attributes of simplicity and efficacy have cemented its status as a linchpin in various review papers and experimental methodologies. Its role in swiftly discriminating between citric acid and glutaraldehyde empowers researchers to identify and elucidate the properties of substances across a spectrum of applications. From biomedical investigations to industrial analyses and environmental monitoring, the test's ability to deliver rapid, reliable results proves indispensable.

Through harnessing the yellowish test, scientists can expedite the screening and analysis of samples, thereby propelling advancements in knowledge across diverse scientific fields. It serves as a testament to the profound impact of straightforward yet robust qualitative assays in the scientific quest for understanding. By affording valuable insights into the chemical behaviours and properties of substances under scrutiny, the yellowish test underscores the enduring significance of simplicity in scientific inquiry.

CHAPTER 6
CONCLUSION &
FUTURE PROSPECTS

In conclusion, this study successfully synthesized and characterized PVA-starch films using glutaraldehyde and citric acid as crosslinkers. The findings demonstrate that the crosslinked films exhibit enhanced mechanical, thermal, and barrier properties, making them suitable for various packaging applications. The biodegradability of these films further underscores their potential as sustainable alternatives to conventional synthetic polymers. Future research should focus on optimizing formulations, advanced characterization, environmental impact assessments, and scaling up production to realize the full potential of these biopolymer films in industrial applications. Through these efforts, PVA-starch films can significantly contribute to reducing plastic pollution and promoting sustainable materials in the packaging industry.

6.1 Conclusion

The main goal of this study was to generate and examine PVA and starch films by employing glutaraldehyde and citric acid as crosslinking agents. These biopolymer films have potential use in the packaging industry, among other industries, because of their capacity to degrade naturally, their mechanical strength, and their ability to act as barriers. We effectively clarified the structural, mechanical, and thermal features of the films under investigation by employing complete synthesis techniques and characterization technologies.

During the synthesis process, varying amounts of PVA and starch were mixed together. The cross-linking process was subsequently conducted by introducing glutaraldehyde and citric acid. The selection of these crosslinkers was based on their capacity to establish durable networks inside the polymer matrix. This improved the films' ability to withstand heat and mechanical pressures. The success of the crosslinking process was confirmed by FTIR, which detected characteristic peaks associated with ester and acetal linkages. The aldehyde and carboxyl groups of glutaraldehyde and citric acid reacted with the hydroxyl groups of PVA and starch, as evidenced by the observed peaks.

Through the utilization of XRD, we gained valuable insights into the films' crystallinity, which provided us with a deeper understanding of their uniformity. The findings indicated a reduction in crystallinity following crosslinking, indicating that the crosslinkers interfered with the orderly arrangement of starch and PVA chains, resulting in a more disordered structure. Its lack of definite shape makes it valuable for packaging purposes because of its increased pliability and decreased fragility.

The mechanical tests demonstrated that the crosslinked films exhibited superior tensile strength and elongation at break compared to the non-crosslinked films. The tensile strength of films crosslinked with glutaraldehyde was higher due to the creation of compact and rigid networks. Conversely, films crosslinked with citric acid exhibited more elongation, presumably due to the increased flexibility of their ester connections. These results indicate that the mechanical properties of PVA-starch films can be customized for a specific application by altering the crosslinker employed.

The films' barrier properties were assessed for suitability for packaging applications. One of these attributes was the ability to facilitate the passage of oxygen and water vapor. Films treated with glutaraldehyde exhibited the lowest water vapor permeability, but all films demonstrated a notable reduction in permeability upon cross-linking. This can likely be attributed to the hydrophobic features of the crosslinked network. The researchers found that films treated with citric acid exhibited enhanced oxygen barrier qualities during testing for oxygen permeability. Due to their ability to reduce oxidation, these films are appropriate for application in food packaging, hence prolonging the shelf life of perishable products.

To evaluate the films' biodegradability, researchers conducted soil burial and enzymatic degradation experiments. Both crosslinkers enhanced the films' biodegradability. However, films crosslinked with citric acid exhibited a faster degradation rate than those crosslinked with glutaraldehyde. The hydrolysable ester bonds in citric acid-crosslinked films are more susceptible to microbial degradation compared to the stronger acetal linkages in glutaraldehyde-crosslinked films.

The presence of ester bonds in citric acid-crosslinked films gives rise to this variance. The study revealed that PVA-starch films, when crosslinked with citric acid or glutaraldehyde, had reduced environmental impact compared to traditional synthetic polymers.

6.2 Future Prospects

Taking into consideration the findings of the research analysis, the following are a few suggestions for the future:

1. The Optimization of the Concentration of Crosslinker

It is recommended that future research concentrate on adjusting the concentration of glutaraldehyde and citric acid in order to create a balance between the mechanical strength and flexibility of the material. The procedure of systematically varying the concentration of the crosslinker, followed by an examination of the mechanical, thermal, and barrier properties, can be of assistance in determining the formulation that is most suitable for a particular application.

2. Incorporating various additional biopolymers

Further investigation into the possibility of combining PVA-starch films with additional biopolymers, such as chitosan, gelatin, or alginate, can improve some features, such as antibacterial activity, and thus increase the films' appropriateness for use in food packaging. Furthermore, this technique has the potential to assist in the customization of degradation rates and mechanical properties for a wide range of applications.

3. Advanced Characterization techniques

When modern characterization techniques, such as dynamic mechanical analysis (DMA), atomic force microscopy (AFM), and nuclear magnetic resonance (NMR) spectroscopy, are utilized, it is possible to gain a more profound understanding of the molecular interactions and structural dynamics of the crosslinked films. This information is essential to comprehending the long-term performance and stability of the films under a variety of environmental circumstances.

4. Assessment of the Impact on the Environment

To determine whether the PVA-starch films are sustainable, they should be subjected to exhaustive environmental impact evaluations, which should also include life cycle analysis (LCA). This study will provide a comprehensive perspective of the environmental benefits and potential drawbacks, which will serve as a guide for the development of more environmentally friendly packaging solutions.

5. Scaling Up and Industrial Applications

Increasing the scale of the synthesis process to pilot and industrial levels should also be a primary focus of study in the future. To accomplish this, it is necessary to overcome issues associated with the uniformity and reproducibility of film qualities and design economically productive production processes. Collaboration with industry partners can facilitate the translation of laboratory findings into commercial goods.

6. Application-Specific Testing

It is necessary to carry out application-specific testing, often known as shelf-life studies for food packaging and mechanical performance, under conditions representative of the real world. These tests will determine whether the films have any possibility for improvement and validate their practical usefulness. It is also possible to widen the value of these films by investigating their potential usage in other applications, such as agricultural mulch films, medical packaging, and biodegradable bags.

7. Acceptability from both regulators and customers

When it comes to the effective commercialization of PVA-starch films, it is essential to do research on regulatory compliance and customer acceptability strategies. Increasing the likelihood that the films will be used by the food sector requires ensuring that they follow food safety and packaging regulations. Consumer acceptance studies have the potential to offer valuable insights into the preferences of the market and may also assist in the development of successful marketing strategies.

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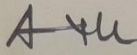
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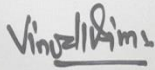
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