Preparation and Characterization of PVA /Cellulose Nanocrystals/ ε-Polylysine Nanocomposite as Potential Food Packaging Application

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DECLARATION

I hereby declare that the work embodied in this report is the result of the original research and has

not been submitted for a higher degree to ant universities or institutions.

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PREPARATION AND CHARACTERIZATION OF PVA /CELLULOSE NANOCRYSTALS/ E-POLYLYSINE NANOCOMPOSITE AS POTENTIAL FOOD PACKAGING APPLICATION

ABSTARCT

The manufacture and analysis of Polyvinyl Alcohol (PVA) Cellulose Nanocrystals (CNC) ε-Polylysine thin films are the main focus of the thesis, with a focus towards their possible use in food packaging. The physical and chemical characteristics of the resultant thin films are thoroughly investigated as part of the research's characterisation phase. To evaluate a material's endurance, mechanical characteristics like tensile strength and flexibility, weight loss, and x-ray diffraction analysis are looked at. For food packaging materials, barrier qualities especially those related to gas and moisture permeability are vital factors to take into account. The research hopes to make a significant contribution to the field of sustainable packaging by illuminating these traits. In order to maintain the quality and safety of packaged food goods, it is necessary to fulfil both the mechanical and antibacterial needs. The objective is to offer an efficient and environmentally friendly substitute for food packaging applications.

ABSTRAK

Pengilangan dan analisis filem nipis Polyvinyl Alcohol (PVA) Cellulose Nanocrystals (CNC) ε-Polylysine adalah fokus utama tesis, dengan tumpuan terhadap kemungkinan penggunaannya dalam pembungkusan makanan. Ciri-ciri fizikal dan kimia bagi filem nipis yang terhasil disiasat dengan teliti sebagai sebahagian daripada fasa pencirian penyelidikan. Untuk menilai daya tahan bahan, ciri mekanikal seperti kekuatan tegangan dan kelenturan, penurunan berat badan dan analisis pembelauan sinar-x ditentukan. Untuk bahan pembungkus makanan, kualiti penghalang terutamanya yang berkaitan dengan kebolehtelapan gas dan kelembapan adalah faktor penting untuk diambil kira. Penyelidikan ini berharap dapat memberi sumbangan besar kepada bidang pembungkusan mampan dengan menerangi ciri-ciri ini. Untuk mengekalkan kualiti dan keselamatan barangan makanan yang dibungkus, adalah perlu untuk memenuhi kedua-dua keperluan mekanikal dan antibakteria. Objektif nya adalah untuk menyediakan dan mencirikan filem komposit nano PVA/CNC/ε-Polysine dan untuk menyiasat potensi nanokomposit PVA/CNC/e-Polylysine terbiodegradasi untuk aplikasi pembungkusan cili segar.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

The industry of food processing cannot be function without packaging. Foods are preserved and kept safe by packaging which also serves as the products undetected. Effective packaging materials and the related technology provide optimal safety and the preservation of the quality of food products. This can be accomplished with appropriate packaging starting with the unit operations connected with processing to the production to handling and storage and ultimately to the consumer. The goal of the study is to determine the material's potential for usage in food packaging applications by concentrating on the synthesis and characterisations of a nanocomposite consisting of polyvinyl alcohol (PVA) and cellulose nanocrystals (CNCs). (Bratovcic, A, 2015)

Due to its superior mechanical qualities, water solubility, and biocompatibility, PVA is a synthetic polymer that finds widespread use in numerous sectors. The nanoscale fibres used in CNCs, on the other hand, are taken from organic cellulose sources like bacteria, plants, and wood. The unique qualities of CNCs, such as their high strength, stiffness, and biodegradability, have attracted interest in recent years. As comparison to pure PVA, the combination of PVA with CNCs in a nanocomposite material can result in better mechanical capabilities, gas barrier qualities, and biodegradability. As a result, PVA/CNCs nanocomposites are possibly appropriate for food packaging applications where the material must protect the food from environmental variables like oxygen and moisture while simultaneously being suitable for human consumption and environmentally benign.

PVA solution and CNCs suspension are combined to create the PVA/CNCs nanocomposite, which is then cast to create a thin film. One of the most prevalent and renewable natural biopolymers in the world is cellulose, which is also biodegradable. The mechanical properties of cellulose fiber-derived nano- and micro-scale fibrils are significantly better than those of individual fibres.Cellulose that is a single nanometer-sized crystal, also known as nanocrystalline cellulose, nanowhiskers, or nanocrystals (CNC). Due to its remarkable mechanical qualities (high specific strength and modulus), wide specific surface area, high aspect ratio, environmental advantages, and low cost, CNC extraction from renewable sources has drawn more and more attention in recent years. A lot of research shown that CNC had a significant deal of potential use in industries like regenerative medicine, optics, and composite material manufacture. The use of a PVA blend for packaging has been discussed by numerous researchers. (Jean Buzby,2022)

1.2 Problem Statement

The problem statement with conventional food packaging materials such as plastics is that they are non-biodegradable and contribute to environmental pollution. They can take hundreds of years to degrade, and their disposal can harm the environment, wildlife, and human health. Therefore, there is a need for the development of biodegradable and sustainable materials for food packaging applications. The growing concern for environmental sustainability and the demand for biodegradable packaging materials has led to the investigation of polyvinyl alcohol (PVA) as a viable biodegradable polymer for food packaging applications. PVA has various useful features including great film-forming ability, high gas barrier capabilities and compatibility with food products. However, PVA films alone may lack the mechanical strength and stability required for successful packaging of fresh fruit goods. (Sèbe et al., 2017)

PVA-based nanocomposites integrating cellulose nanocrystals (CNCs) as reinforcing agents must be developed. CNCs made from sustainable cellulose sources have high mechanical strength, stability, and biocompatibility. Incorporating CNCs into PVA matrices has the potential to improve the mechanical characteristics, barrier performance overall functioning of PVA films for fresh food packaging applications. This study intends to produce a sustainable and biodegradable packaging solution with increased mechanical qualities and barrier performance by resolving the limits of PVA films through the inclusion of CNCs. The creation of PVA/CNCs nanocomposites has the ability to reduce environmental effect while also preserving the freshness and quality of fresh food items during storage and transportation.

Polyvinyl alcohol (PVA) is a promising biodegradable polymer for food packaging applications because to its outstanding film-forming ability and compatibility with food products. However, PVA films may have limits in terms of mechanical strength, barrier qualities, and other functionalities necessary for effective packaging of fresh food items (Smith et al., 2017; Wang et al., 2018). In order to circumvent these limits and improve the performance of PVA films, appropriate reinforcements or fillers must be included. Reinforcing chemicals can improve mechanical strength, whereas fillers can improve barrier characteristics and other PVA matrix capabilities.

Furthermore, the addition of CNCs can improve barrier qualities such as oxygen and water vapour permeability, which are important for preserving freshness and increasing the shelf life of fresh food items (Chen et al., 2015). CNCs' distinct morphology and high aspect ratio contribute to the creation of convoluted pathways, which reduces gas and moisture passage through the nanocomposite films.

This study aims to provide a sustainable and biodegradable packaging solution that overcomes the limitations of PVA alone, ensuring the freshness and quality of fresh food items while reducing the environmental impact associated with traditional packaging materials.

1.3 Expected Output

The preparation and characterization of PVA/cellulose nanocrystals nanocomposites as potential food packaging applications using potato starch as a source of cellulose nanocrystals is expected to result in a biodegradable and sustainable material that can be used in food packaging. It's expected that the study will use an acid hydrolysis technique to separate cellulose nanocrystals from potato starch. The extracted nanocrystals will then be mixed with PVA polymer. The expected output of this research is a biodegradable and sustainable material that can potentially replace conventional non-biodegradable packaging materials such as plastic. The PVA/cellulose nanocrystals nanocomposites are expected to have improved mechanical properties and reduced waterpot permeability compared to pure PVA polymer. The PVA/CNC nanocomposites are expected can be used in food packaging application.

1.4 Objectives

The specific objectives of the study were:

i. to prepare and characterize PVA/CNC/ε-Polylysine nanocomposite films

ii. to investigate the potential of biodegradable PVA/CNC/e-Polylysine nanocomposites for fresh chili packaging application

1.5 SCOPE OF STUDY

The goal of the research effort is to investigate the preparation and characterisation of a nanocomposite material created up of three essential elements: e-polylysine, cellulose nanocrystals, and polyvinyl alcohol (PVA). The scope of research includes the synthesis and characterisation of PVA Polyvinyl Alcohol/cellulose nanocrystals nanocomposites for possible food packaging applications. Material selection of the matrix material is Polyvinyl Alcohol (PVA) and the reinforcement material is cellulose nanocrystals (CNCs). PVA is a widely used polymer with outstanding film-forming and barrier qualities whereas CNCs made from cellulose have strong mechanical strength and biodegradability. Following that, the preparation methods section focuses on creating acceptable ways for preparing PVA/CNCs nanocomposites. To obtain a homogenous dispersion of CNCs inside the PVA matrix procedures such as solution casting. Furthermore, prepared nanocomposites must be characterised in order to evaluate their properties. X-ray diffraction (XRD), thermal analysis such as differential scanning calorimetry, thermogravimetric analysis, mechanical testing, and barrier property analysis are some of the techniques that may be used. The potential of the nanocomposite to retain food quality, increase shelf life, and protect against external causes will be assessed using Brix and Using these proper testing methods can further precisely assess the potential of PVA/CNCs nanocomposites in keeping food quality, enhancing shelf life, and guarding against external causes than depending just on Brix tests.

There are multiple steps in the study. First, a PVA matrix containing cellulose nanocrystals and e-polylysine will be combined to create the PVA/cellulose nanocrystals/e-polylysine nanocomposite. The exact mixture and production procedure will be fine-tuned to guarantee that the e-polylysine and nanocrystals are evenly distributed throughout the PVA matrix. Subsequently, an array of procedures will be employed to characterise the synthesised nanocomposite. the mechanical and physical characteristics, including barrier qualities, flexibility, and tensile strength. In order to ascertain the form, crystallinity, and thermal stability of the nanocomposite, methods including X-ray diffraction (XRD), and Optical Microscopy (OM) will be employed in the investigation of its morphological features and thermal stability. The industry-standard methods of permeability testing and oxygen and water vapour resistance assessment would be used to nanocomposites.

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1.6 SIGNIFICANCES OF STUDY

There are many significant consequences from the study on the creation and characterisation of PVA/cellulose nanocrystal nanocomposites as prospective food packaging applications. The first is to enhance the packaging of food. Food products' durability, mechanical strength, and barrier qualities may all be increased through the use of nanocomposites in food packaging. PVA/cellulose nanocrystal nanocomposites are capable of producing packaging materials that are more robust, long-lasting, and effective at preserving food quality. Environmental advantages can also result from reduced environmental effect, for as when food packaging uses nanocomposites. When nanocomposites are biodegradable, less plastic waste ends up in landfills and the ocean.

Particularly PVA/cellulose nanocrystal nanocomposites are biodegradable and renewable, making them an environmentally benign substitute for conventional plastic packaging.

Other than that, creation of new material. The research aids in the creation of new materials with enhanced properties over existing ones. The special characteristics of PVA/cellulose nanocrystal nanocomposites can be tuned to certain applications. The research offers insightful information about the characteristics and prospective uses of these materials. The utilisation of PVA/cellulose nanocrystal nanocomposites in food packaging has the potential for commercial applications. Better food product packaging may result from the improved qualities of these materials, which may boost sales and customer satisfaction. The study serves as a basis for further investigation and the creation of these materials for use in industry. In conclusion, the research on the production and characterisation of PVA/cellulose nanocrystal nanocomposites as prospective food packaging application has substantial ramifications for the food packaging business, the environment, and the creation of novel materials with distinctive qualities. Good theoretical method for examining the suitability of PVA/CNC nanocomposites for applications in food packaging. Due to its barrier qualities, mechanical strength, and biodegradability, PVA/CNC nanocomposites have demonstrated encouraging results in terms of their capacity to protect the quality and freshness of food products over time.

The usage of PVA/CNC nanocomposites for food packaging is being studied experimentally so that researchers may further validate the theoretical results and gather knowledge about the real-world uses of these materials. The outcomes of the experiment can also aid in identifying any potential problems or constraints that might need to be resolved before these materials are widely used in the food packaging sector. In general, integrating theoretical analysis with practical research is a potent strategy for examining the suitability of PVA/CNC nanocomposites

CHAPTER 2

LITERATURE REVIEW

2.1 Polymer

Polymers are significant compounds made up of repeated called monomers. They are a type of material that has a wide range of applications due to their versatility, durability, and adjustable qualities. Polymers can be found in a variety of forms, including plastics, fibres, elastomers, coatings, adhesives, and films. Polymer materials are widely employed in a variety of industries, including packaging, automotive, construction, textiles, electronics, healthcare, and many more. They have features such as lightweight, corrosion resistance, flexibility, thermal and electrical insulation, and ease of processing.

Polymer based materials are important in food packaging because they provide protection, preservation, and convenience. They can be used to make films, trays, bottles, and containers that protect food from contamination, increase shelf life, and maintain product safety. Biodegradable and compostable polymers have gained popularity as environmentally friendly alternatives to traditional petroleum-based plastics.

In general, polymers are important materials in modern civilization because of their unique qualities and versatility. They have a wide range of applications and play an important role in a variety of industries, including food packaging.

2.2 POLY (VINYL ALCOHOL)

PVA (poly (vinyl alcohol) is a synthetic polymer made from vinyl acetate by hydrolysis. It is a water-soluble and biodegradable polymer with numerous industrial applications. PVA is distinguished by its outstanding film-forming ability, high tensile strength, and high oxygen barrier characteristics. (Nair, L. S., & Laurencin, C. T. 2020) Other than that, PVA is widely utilised in a variety of applications including packaging since PVA films have good barrier qualities against oxygen and water vapour which assist protect the freshness and quality of packaged food products. Besides from that, PVA fibres are used in the textile sector to make textiles, materials, and nonwoven materials. PVA particles are dyeable have a high tensile strength, and are dimensionally stable. PVA is also extensively used as a binder in adhesives, particularly material adhesives. PVA-based adhesives provide high bonding strength, flexibility, and resistance to water.

2.3 POLYMER NANOCOMPOSITE

A composite material that has a polymer matrix reinforced or filled with nanoscale particles also known as nanofillers or nanoparticles is called a polymer nanocomposite. The characteristics and functionality of the composite material can be greatly improved by adding nanofillers to the polymer matrix. The polymer matrix gains desirable features from the nanofillers' high surface area-to-volume ratio and distinctive physical and chemical properties at the nanoscale.

The enhanced mechanical properties of polymer nanocomposites are one of its benefits. Materials with nanofillers present have the potential to improve the mechanical performance of pure polymers by increasing the stiffness, strength, and toughness of the polymer matrix. Stress is transferred and distributed more easily attributable to the powerful connection between the polymer matrix and the nanofillers

2.4 Ɛ-POLYLYSINE

ε-Polylysine is a natural antimicrobial compound produced by the bacterium Streptomyces albulus. It is a polymer consisting of the amino acid lysine linked together by peptide bonds. The "ε" in ε-polylysine refers to the epsilon amino group in lysine, indicating the linkage at that position in the amino acid. ε-Polylysine has gained attention for its antimicrobial properties, particularly its ability to inhibit the growth of various bacteria and fungi. Due to its antimicrobial activity and safety profile, it has found applications in the food industry as a preservative. It is used to extend the shelf life of certain food products by preventing the growth of spoilage and pathogenic microorganisms.

In the context of the study, that mentioned earlier, incorporating ε-polylysine into a PVA/cellulose nanocrystals composite suggests an interest in leveraging its antimicrobial properties for potential applications in food packaging, where the prevention of microbial contamination is crucial for food safety and preservation.

2.5 Cellulose

2.5.1 General Info

Polymer nanocomposites are materials made up of a polymer matrix that has been reinforced with nanoscale fillers. These fillers, which are usually nanoparticles, nanofibers, or nano clays, are disseminated inside the polymer matrix, resulting in improved characteristics over pure polymers. Incorporating nanoparticles into polymers can increase mechanical strength, thermal stability, electrical conductivity, barrier characteristics, and other features. The improved mechanical properties of polymer nanocomposites are one of their main advantages.

Nanofillers can reinforce the polymer matrix, increasing tensile strength, modulus, and toughness. Some nanofillers, such as nanoclays or nanofibers, have a high aspect ratio, which enables excellent load transfer channels and increases composite stiffness. Polymer nanocomposites are thus well suited for high mechanical performance applications such as structural components, automotive parts, and aerospace materials.

Polymer nanocomposites also have better barrier characteristics. Because nanofillers can produce tortuous routes for gas or liquid molecules, the permeability of the composite material is reduced. Polymer nanocomposites with improved barrier performance are excellent for packaging applications such as food packaging or barrier sheets for chemical and gas storage.

2.5.2 Polymer Nanocomposite

A polymer nanocomposite is a polypropylene (PP) nanocomposite reinforced with montmorillonite clay nanoparticles. Montmorillonite is a layered silicate mineral that can be exfoliated into nanoscale layers and disseminated into a polymer matrix to form a nanocomposite.

In this example, the polypropylene matrix is blended with montmorillonite clay nanoparticles at a low loading level (e.g., 1-5% by weight). The nanoparticles are dispersed into the polymer matrix using various procedures such as melt compounding or solution mixing, guaranteeing equal distribution of the nanoparticles.

When montmorillonite nanoparticles are included into the polypropylene matrix the characteristics of the nanocomposite improve significantly when compared to pure polypropylene. The addition of nanoparticles increases the polypropylene matrix's tensile strength, modulus, and toughness. The nanoparticles serve as reinforcement, allowing for effective load transfer and increasing the nanocomposite's overall mechanical performance. Montmorillonite nanoparticle dispersion at the nanoscale provides complex diffusion routes for gas molecules, lowering gas permeability through the nanocomposite. This enhanced barrier feature is advantageous for applications requiring greater gas barrier performance such as packaging materials.

Polypropylene nanocomposites reinforced with montmorillonite nanoparticles are used in a variety of industries, including automotive components, packaging materials, electrical insulation, and building materials. It is especially important to note that the specific properties and performance of polymer nanocomposites are affected by a variety of parameters, including the kind of polymer, the nature of the nanofiller, the processing conditions, and the dispersion and interaction of the polymer matrix and nanoparticles.

2.5.3 Cellulose

Cellulose is a complex polysaccharide comprised of repeated glucose units linked together by -1,4-glycosidic linkages. It is the most prevalent organic chemical on the planet and is the principal structural component of plant cell walls. Cellulose forms long, linear strands that can interweave to form a strong, rigid network. The cellulose's amazing qualities is its great tensile strength. The hydrogen bonding between adjacent cellulose chains contributes to its strength and rigidity. These hydrogen bonds form a crystalline structure within cellulose known as cellulose I, which accounts for its mechanical stability. The crystalline sections are interspersed by amorphous parts, which give flexibility and allow for water absorption.

Other than that, cellulose is hydrophilic due to the presence of many hydroxyl (-OH) groups along its polymer chain. These hydroxyl groups make cellulose highly interacting with water molecules, allowing it to absorb and hold water. This feature is used in applications such as the manufacturing of paper where fibres made from cellulose absorb water to form a web-like structure that can be easily manipulated into varied shapes.

Overall, cellulose is a versatile, renewable, and sustainable polymer with several industrial applications. Its distinguishing characteristics are high tensile strength, hydrophilicity, and biodegradability.

2.5.4 Cellulose Nanocrystal

Cellulose nanocrystals (CNCs) are nanoscale particles generated from cellulose fibre hydrolysis or acid hydrolysis because of their highly crystalline structure and nanoscale dimensions. These nanocrystals have special features also CNCs are manufactured in a series of phases. To begin cellulose fibres are extracted from a renewable source. Impurities and non-cellulosic components are removed from the fibres using a chemical treatment. The cellulose then undergoes hydrolysis under acidic conditions which selectively removes the transparent portions while leaving behind the highly crystalline nanocrystals.

CNCs have excellent mechanical qualities such as high tensile strength, modulus, and stiffness. The strong intermolecular hydrogen bonding inside the crystalline regions and the high aspect ratio of the nanocrystals causes these features. CNCs can considerably improve the mechanical performance of nanocomposites when added into polymer matrix.

CNCs like cellulose are biodegradable and made from renewable materials. They are eco-friendly and provide a long-term alternative to synthetic nanoparticles manufactured from non-renewable sources. Their biodegradability also assures compatibility with environmentally friendly applications.

2.6 Method of Polymer Nanocomposite Preparation Solvent Casting and Etc.

Solvent casting is a process that involves dissolving a polymer in an appropriate solvent and then casting the polymer solution onto a substrate in order to create thin films or membranes. The solvent disappears, leaving behind a solid polymer sheet with the required characteristics. Numerous industries, including the biomedical, electronics, coatings, and optical sectors, heavily rely on the solvent casting technology. It makes it possible to create thin films or membranes with exact control over their composition, thickness, and other characteristics. Applications for these films include electronic devices, drug delivery systems, sensors, membranes for separation processes, and surface coatings.

2.6.1 Characterization Technique

i. XRD

The technique of X-ray Diffraction (XRD) is frequently used to analyse the crystallographic structure, phase composition, and crystallinity of materials, including polymer nanocomposites. The information provided by XRD on the arrangement and orientation of atoms within a material is critical for understanding its properties. XRD makes use of an X-ray beam that is aimed at the sample. The X-rays interact with the material's crystal lattice, causing constructive or destructive interference of the X-ray waves. A detector collects the diffracted X-rays and measures their intensity and angle of diffraction.

Other than that, the crystal structure of a material can be determined using XRD. The sample's diffraction pattern is a one-of-a-kind fingerprint that corresponds to the arrangement of atoms within the crystal lattice. The crystal structure and lattice parameters of the material can be determined by analysing the positions and intensities of the diffraction peaks.

XRD is a non-destructive technique for analysing solid samples such as thin films, fibres, and bulk materials. It is a useful tool for characterising the structure, phase composition, and crystallinity of polymer nanocomposites, allowing researchers to gain insight into their properties and behaviour. When combined with other characterization techniques such as SEM and thermal analysis, XRD helps to a thorough knowledge of the structure-property connection of the nanocomposite.

ii. Tensile Strength

Tensile testing is a popular method for evaluating the mechanical properties of materials, particularly polymer nanocomposites. This technique examines a material's response to tension, providing useful information about its strength, elasticity, and deformation behaviour. Sample Preparation rectangular-shaped specimens of the polymer nanocomposite material are prepared for tensile testing. The dimensions and shape of the specimens are determined by conventional testing protocols and the study's unique requirements. Tensile testing is done with a universal testing machine (UTM) or an Instron machine. The UTM exerts a controlled tensile force on the specimen while measuring both the applied force and the consequent deformation(Ejara et al., 2021).

2.7 Application of Polymer Nanocomposites as Fresh Fruit Packaging.

Polymer nanocomposites have demonstrated remarkable promise for use in fresh fruit packaging, including increased barrier characteristics, mechanical strength, and enhanced usefulness. These nanocomposites can aid in maintaining the quality and prolonging the shelf life of fresh fruits. These are a few components of the use of polymer nanocomposites in packaging fresh fruit. Brix is a measurement of a fruit's sugar content that shows how sweet and ripe it is. Polymer nanocomposites can be created using nanofillers or additives that interact with the gases released by the fruit, such as ethylene, a ripening indication. The nanocomposite can give real-time monitoring of fruit maturity by incorporating a Brix analyzer or a gas sensor within the packing material, enabling ideal conditions for fruit harvesting, transportation, and storage.

Nanofillers with built-in antimicrobial characteristics or antimicrobial compounds can be used to functionalize polymer nanocomposites. As a result, the fruit's shelf life is increased and the risk of rotting is decreased by limiting the growth of germs like bacteria or fungi on the fruit's surface. A persistent defence against microbial contamination can be achieved through the controlled release of antimicrobial compounds from the nanocomposite packing.

Fresh fruit's texture is a crucial aspect of its quality. To prevent physical harm to fruits during handling, shipping, and storage, polymer nanocomposites can be engineered to have desired mechanical qualities like flexibility, elasticity, and strength. The packaging made of nanocomposite materials aids in preserving the texture and look of the fruits by reducing bruising or impact-related injuries.

In general, the use of polymer nanocomposites in the packaging of fresh fruit has chances to enhance quality factors such barrier characteristics, brix monitoring, texture preservation, colour preservation, and antibacterial activity. By taking care of these issues, nanocomposite packaging can help increase the freshness of fruits, maintain their shelf life, and improve the entire consumer experience.

CHAPTER 3

MATERIALS AND METHOD

3.1 Materials

Polyvinyl Alcohol purchased from R&M Chemicals was in powder form. Cellulose nanocrystal from the cell force company, made in Canada. The CNC was also in powder form. The raw materials weighed around 0.5 g and were stored in a sample bottle at room temperature for the raw materials characterization process.

- Polyvinyl Alcohol (PVA) powder or pellets
- ε-polylysine
- Distilled water
- Heat-resistant container or beaker
- Stirrer or magnetic stir bar
- Hot plate or heating source
- Film casting surface (e.g., glass plate or petri dish)
- Film applicator or doctor blade (for achieving desired film thickness)

3.2 Methods

3.2.1 Preparation of PVA/CNC/PL bio-composite films

The CNC existed as a powder. For the raw materials characterization technique, the roughly 1.0 g of raw materials will be kept at room temperature in a sample container.

During a two-hour period, PVA was dissolved in water at a rate of 10% by weight while maintaining a 90°C temperature. A set amount of CNC dispersion and a predetermined amount of PVA solution were initially combined, and the combination was then sonicated at room temperature for 30 minutes to create the PVA/CNC films. The homogeneous liquid will pour into the petri dishes, where it will allow to cure at room temperature. The CNC dispersion was added to the PVA solution first, and then the combination will be sonicated at room temperature for 30 minutes to create the PVA/CNC/PL composite films. The mixture was then swirled for 1.5 hours and various amounts of PL were added after that. The mixture was then heated to 50 degrees Celsius while being stirred constantly for an hour. Finally, the liquid was distributed among various petri dishes and allowed to air dry at ambient temperature. The films will dry at a temperature of 40 degrees Celsius for five hours before receiving a final rinse in deionized water.

3.2.2 Film Casting

PVA, CNC, and Polylysine solution samples were cast on glass Petri dishes and will allow to dry at room temperature for 24 to 48 hours. The bio-composite thin films with a 1.0 wt% and 2.0 wt% -PL content will created. The bio-composite thin film is then kept for characterisation in a zip bag.

3.2.3 Drying

The bio-nanocomposite will be allowed to dry completely. This can be achieved by placing the glass plate or petri dish in an oven or drying chamber at a controlled temperature. The drying temperature and time will depend on the thickness of the film and the desired properties. It is important to ensure proper ventilation during the drying process to allow the solvent (water) to evaporate effectively.

3.2.4 Film Removal

Peel the film off the glass plate or petri dish gently after it has completely dried. After that, if necessary, trim the film to the desired size and form.

3.2.5 X-ray diffraction (XRD).

Using an XRD diffractometer (Bruker Kappa APEXII) with an operating power of 40 kV and a scan rate of 2 min-1, XRD patterns of e-polylysine and PVA film were produced.

3.2.6 Film thickness.

The bio-nanocomposite films' thickness was measured three times at random, and the digital thickness measuring gauge computed the average thickness to the nearest 0.001 mm.

3.2.7 Characterization

This characterization part will explain the method and device used to characterize the PVA/CNC film.

3.2.8 Visual Inspection

On this study, visual inspection includes transparency test and thickness test.

3.2.9 Transparency Test

A transparency test was used to determine an object's transparency. The optical clarity that allows an item to be seen when observed via plastic film, foil, glass, and so on is referred to as transparency. As a result, the bio composite thin film was put through a transparency test, and its transparency was assessed. The transparency test was carried out in a well-lit area. The background graphic is the UMK emblem. Various thin film proportions were utilised and examined.

3.3.0 Thickness Test

Calibrated digital micrometre Vernier calliper was utilised to measure the thickness of the film at three separate locations. The average of the three readings was then calculated.

3.3.1 Tensile Test

Using the Universal Testing Machine model M500-50CT, the breaking stress (BS) and elongation at break of the bioplastic composite sheets were measured and studied. By pushing the thin film sample along a major longitudinal axis at a consistent rate of extension until a predetermined stress or strain value was reached, the tensile test was performed. The films were divided into 40 mm x 13 mm rectangular portions, and analysis was carried out at a cross head speed of 10 mm/min with a gauge length of 40 mm. Prior to testing, the thickness of each sample was also considered(Ejara et al., 2021). Each specimen was tested using three samples, and the average was calculated. The average thickness of the films was 0.20 ± 0.02 mm.

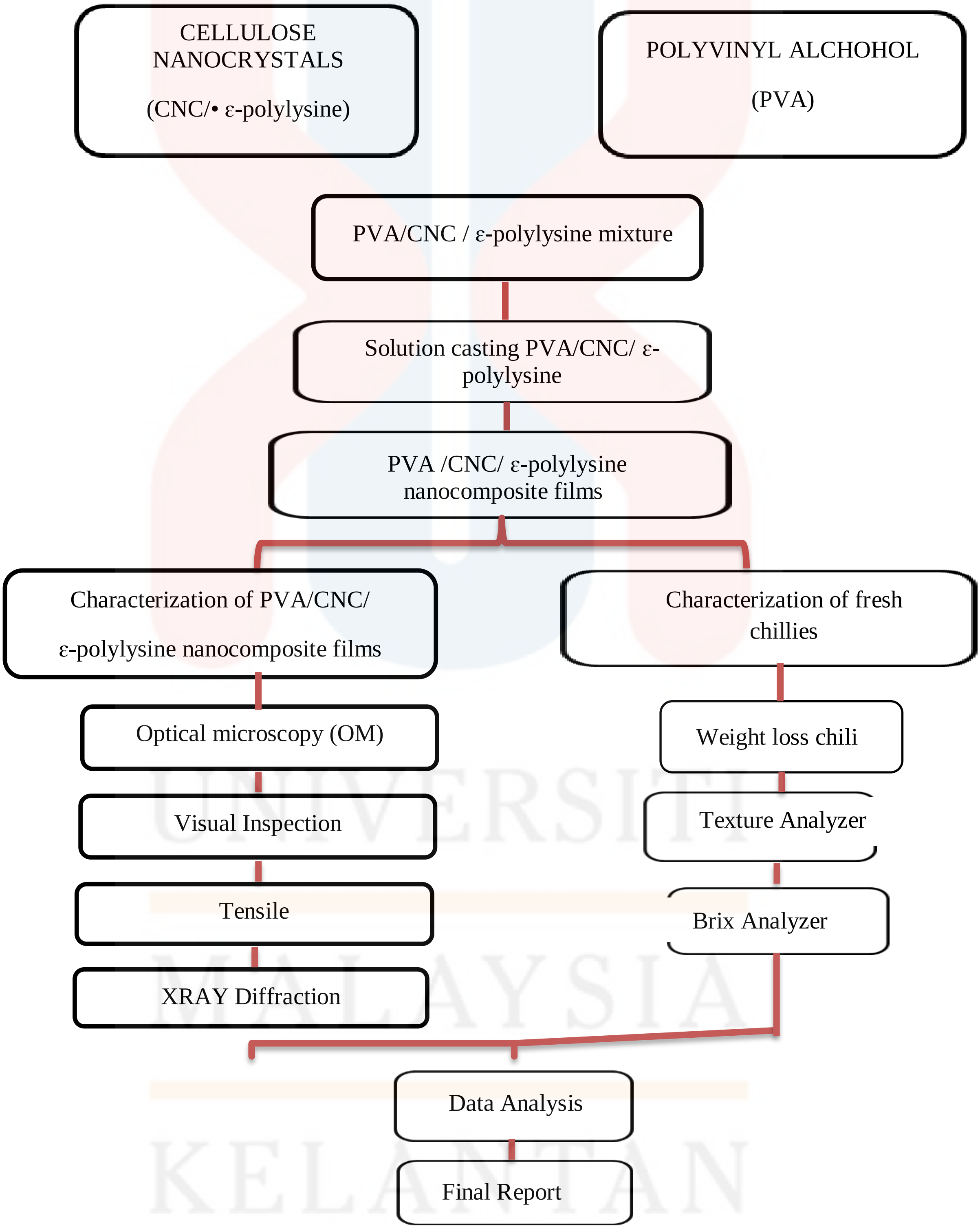
3.3.2 Optical Microscopy (Pom)

The POM was performed using an Olympus BH2 BHM microscope (Tokyo, Japan) equipped with crossed polarizers to observe the birefringence characteristics of the samples. The images were captured using a polarized optical microscope with a 10X objective. To guarantee that only the circularly polarized component of the reflected light was gathered, the imaging was done with a cross-polarization setting (El-Hadi, 2017).

3.3.3. CHILI TESTING

The fresh fruit will be test by physiological loss in weight. The cultivar Punjab Red Cherry of the cherry tomato (Lycopersicon esculentum Cerasiforme) was purchased from a nearby supermarket. Immediately after the purchase, the produce was precooled and cleaned using cold water combined with 100 ppm chlorine to reduce the respiration rate and microbial content. The fruits were consistent in length (37 ±1.6 mm), diameter (22 ±1.8 mm), weight (14.5±2.1 g) and ripeness. Before packing, they were visually screened to take out any damaged parts.

4. FLOW CHART

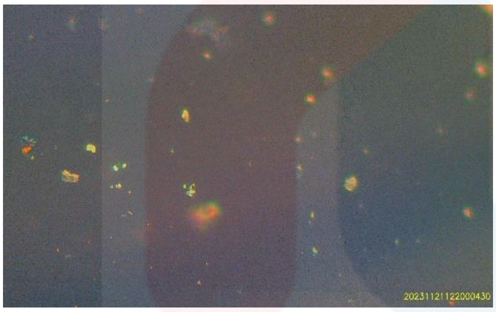

CELLULOSE NANOCRYSTALS
(CNC/• ε-polylysine)
POLYVINYL ALCHOHOL
(PVA)
PVA/CNC / ε-polylysine mixture
Solution casting PVA/CNC/ ε-polylysine
PVA /CNC/ ε-polylysine nanocomposite films
Characterization of PVA/CNC/
ε-polylysine nanocomposite films
Characterization of fresh chillies
Optical microscopy (OM)
Visual Inspection
Tensile
XRAY Diffraction
Weight loss chili
Texture Analyzer
Brix Analyzer
Data Analysis
Final Report

CHAPTER 4

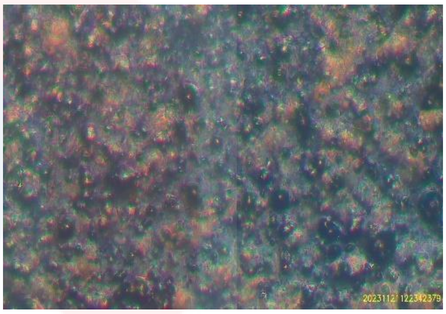
4.1 RESULTS AND DISCUSSION

4.1.1 Characterization of Raw Materials

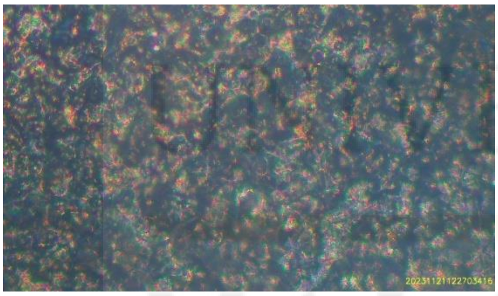
i. Polarized Optical Microscopy (OM)



(a) PVA 5%

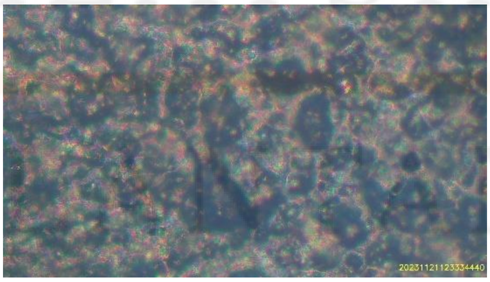


(b) PVA 5% + CNC 1%



(c) PVA 5% + CNC 1% + PL (0.3%)

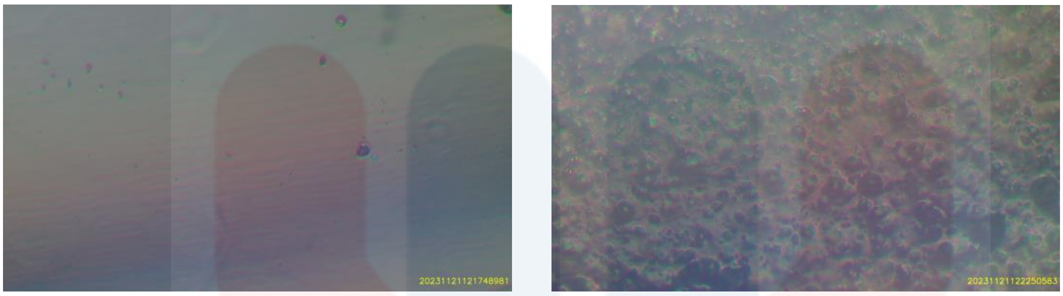
(d) PVA 5% + CNC 1% + PL
(0.6%)



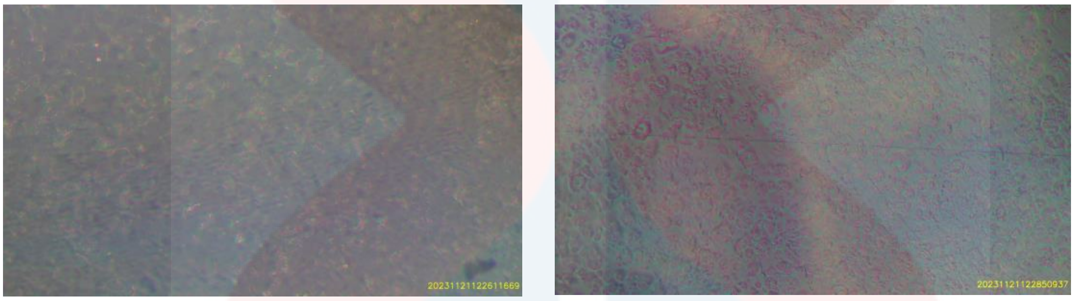
(e) PVA 5% + CNC 1% + PL (0.9%).

Figure 4.1: Optical microscopy images of bio-nanocomposite

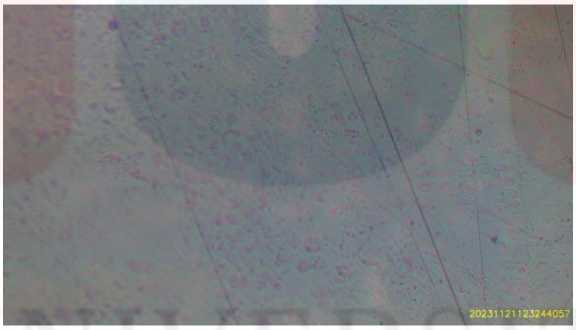
ii. non-polarized



(a) PVA 5% **(b) PVA 5% + CNC 1%**



(c) PVA 5% + CNC 1% + PL (0.3%) **(d) PVA 5% + CNC 1% + PL (0.6%)**



(e) PVA 5% + CNC 1% + PL (0.9%)

Figure 4.2: Optical microscopy images of bio-nanocomposite non polarized

From Figure 4.1, the bio-nanocomposites are composed of PVA (Polyvinyl Alcohol) at 5%, CNC (Cellulose Nanocrystals) at 1%, and varying concentrations of (ε-poly-L-lysine (ε-PL) at 0%, 0.3%, 0.6%, and 0.9%. The images (a), (b), (c), (d), and (e) likely represent the different compositions of these bio-nanocomposites. The use of optical microscopy suggests that the images are taken using visible light, providing insights into the structural and morphological features of the materials.

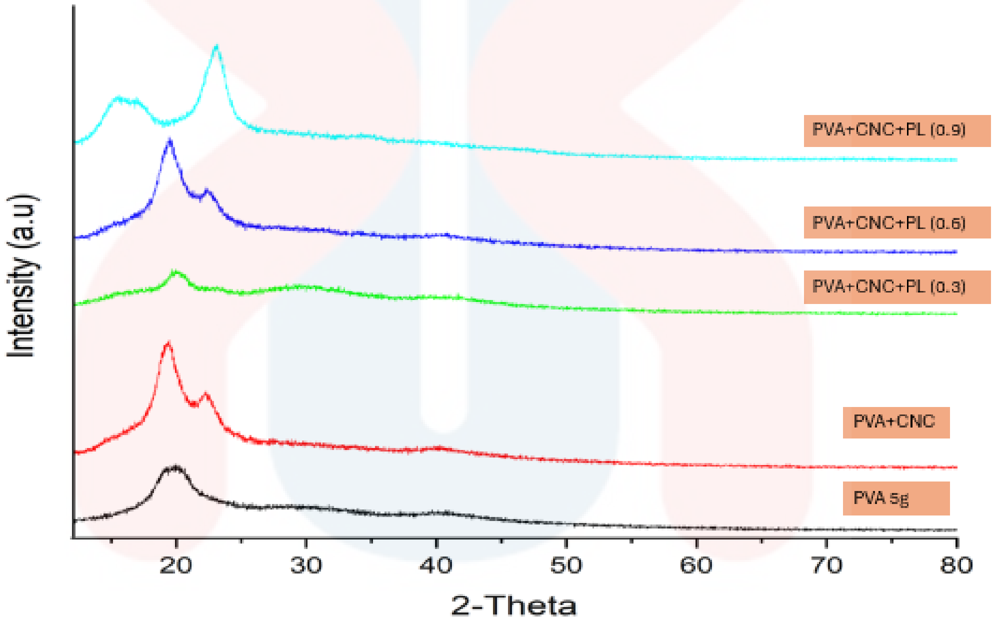
It appears to be an optical microscopy image of a bio-nanocomposite made of PVA (polyvinyl alcohol) and CNC (cellulose nanocrystals). The image is polarized, which means that the light passing through the sample has been filtered in a specific direction. This can help to reveal the orientation of the molecules in the sample. In the image, it can see a few different features example there are long, thin fibers that are aligned in a roughly horizontal direction. These fibers are likely the CNCs. The background is a mixture of light and dark areas. The light areas may be regions where the PVA and CNC are well-mixed, while the dark areas may be regions where the CNCs are more concentrated. There are also a few small, circular features in the image. These could be air bubbles or other voids in the material. Overall, the image suggests that the PVA and CNC are well-mixed in the bio-nanocomposite. The CNCs are aligned in a roughly horizontal direction, which may be due to the processing conditions used to make the material.

A bio-nanocomposite consisting of PL (ε-poly-L-lysine), CNC (cellulose nanocrystals), and PVA (polyvinyl alcohol) is seen in the polarized optical microscope image. The image is polarized, suggesting that a particular direction of light filtering has been applied to the sample. Figure 1. Optical microscopy images of bio-nanocomposite polarized (c) PVA 5% + CNC 1% + PL (0.3%) (ε-poly-L-lysine (ε-PL). The image is a polarized optical microscopy image of a bio-nanocomposite made of PVA (polyvinyl alcohol), CNC (cellulose nanocrystals), and PL (ε-poly-L-lysine). The image is polarized, which means that the light passing through the sample has been filtered in a specific direction. This can help to reveal the orientation of the molecules in the sample. There are some of the features that can see in the image such as there are long, thin fibers that are aligned in a roughly horizontal direction like fibers are likely the CNCs. Other than that, the background is a mixture of light and dark areas. The light areas may be regions where the PVA and CNC are well-mixed, while the dark areas may be regions where the CNCs are more concentrated. There are also a few small, circular features in the image. These could be air bubbles or other voids in the material. In addition to these features, there are also some additional features that are likely due to the presence of the PL, there are some bright, orange-coloured streaks in the image. These streaks are likely due to the PL, which is a fluorescent dye. The PL appears to be concentrated in the cell walls of the plant cells that are visible in the image. Overall, the image suggests that the PVA, CNC, and PL are well-mixed in the bio-nanocomposite. The CNCs are aligned in a roughly horizontal direction, which may be due to the processing conditions used to make the material. The PL is concentrated in the cell walls of the plant cells, which suggests that it may be able to be used to target drugs or other molecules to specific cells.

4.2 X-Ray Diffraction

4.2.1 Phase Identification of XRD

The milled composite powders' structural characteristics were determined through X-ray diffraction (XRD). Diffract. Eva software was employed to establish matched patterns for the composite powders. The data extracted from the peak patterns were utilized to compute both the crystallite size and internal strain.



SAMPLE	LATTICE PARAMETER			a/b Ratio	c/b Ratio
	a	b	c		
PVA	17.226	40.0522	19.6088	0.43009	0.48958
PVA/CNC	9.932	17.216	81.864	0.57691	4.75511
PVA/CNC/ ε-polylysine (0.3g)	13.115	18.992	17.926	0.69055	0.94387
PVA/CNC/ε-polylysine (0.6g)	15.8495	12.698	26.3078	1.24819	2.07181
PVA/CNC/ ε-polylysine (0.9g)	13.115	18.992	17.926	0.69005	0.94387

Figure 4.3: Phase Identification of XRD of each sample.

From Figure 4.3, it was showing phase identification of XRD of each sample. Lattice parameter refers to the fundamental unit cell dimensions of a crystalline material. a/b Ratio, c/b Ratio describe the relative lengths of the unit cell edges along different axes. While a, b, c represents the actual lengths of the unit cell edges in angstroms (Å). PVA, PVA/CNC, PVA/CNC/ε-polylysine (0.3g), are likely different material compositions being studied. PVA might stand for polyvinyl alcohol, CNC for cellulose nanocrystals, and ε-polylysine is a synthetic polymer. The numbers in parentheses could indicate the amount of ε-polylysine added. 17.226, 40.0522 these are numerical values for the corresponding lattice parameters for each material composition. The PVA curve has two broad peaks, indicating that the PVA is amorphous or has a very small crystallite size. The PVA/CNC curve has sharper peaks, indicating that the addition of CNC has induced some crystallinity in the PVA. The PVA/CNC/ε-polylysine curves have even sharper peaks, indicating that the addition of ε-polylysine has further increased the crystallinity of the material. The peak positions are consistent with the presence of cellulose I and cellulose II phases in the samples.Overall, the figure shows that the addition of CNC and ε-polylysine to PVA increases the crystallinity of the material.

4.3 Solution Mixture

4.3.1 Visual Inspection

Figure 4.4: Visual Inspection of each sample solution

From Figure 4.4 the sample solutions are PVA 5%, PVA 5% + CNC 1%, PVA 5% + CNC 1% + PL (0.3%), PVA 5% + CNC 1% + PL (0.6%) and PVA 5% + CNC 1% + PL (0.9%) arranged from left to right. When compared to the PVA solution, the PVA/CNC mixture becomes somewhat cloudy and transparent, but the PVA solution stays clear and transparent. All three of these solutions become thick and milky white when ε-poly-L-lysine was added. The solutions become entirely opaque and lose their transparency when is ε-poly-L-lysine added to the PVA/CNC mixture. This shift in appearance, which gives it a milky white look, is probably the effect of light being scattered by the ε-poly-L-lysine particles. The changes in transparency and colour you observed in the PVA solutions can be attributed to light scattering caused by the different components

PVA solution is being a polymer itself because PVA forms a clear and transparent solution due to its uniform structure and refractive index matching with water. Light passes through readily without significant scattering. Other than that, PVA/CNC mixture was adding CNC nanocrystals disrupts the homogeneity of the solution. These rod-shaped particles have a different refractive index than water and PVA, causing light to scatter around them as it passes through. This scattering effect leads to a slightly cloudy and translucent appearance compared to the pure PVA solution.

PVA/CNC/ε-poly-L-lysine introducing ε-poly-L-lysine particles further intensifies the light scattering. These positively charged, biocompatible polymers tend to aggregate in the solution, forming larger structures that scatter light more effectively. This results in a thicker, milky white appearance due to the combined effect of multiple scattering centers.

(a) PVA 5 (b) PVA 5% + CNC 1% (c) PVA 5% + CNC 1% + PL (0.3%),

(d) PVA 5% + CNC 1% + PL (0.6%) (e) PVA 5% + CNC 1% + PL (0.9%)

Figure 4.5: Visual Inspection Images of Nanocomposite

Figure 4.5 shows the nanocomposite films (a) PVA 5%, (b) PVA 5% + CNC 1%, (c) PVA 5% + CNC 1% + PL (0.3%), (d) PVA 5% + CNC 1% + PL (0.6%), and (e) PVA 5% + CNC 1% + PL (0.9%) are depicted in Figure 4.1.4. As we know, PVA 5% is a relatively dilute solution, which means that it has a low concentration of PVA. This makes it suitable for applications where a thin, clear film is desired. There are some properties of PVA 5% is a clear and transparent solution, which means that it does not absorb or scatter light. Other than that, PVA 5% is water-soluble which makes it easy to apply and clean up. Next is biodegradable, which means that it will break down over time into harmless products also PVA 5% is non-toxic, which makes it safe for use in food.

The composition from the solution consists of 5% polyvinyl alcohol (PVA) and 1% cellulose nanocrystals (CNC). PVA is a widely used synthetic polymer known for its properties like clarity, water solubility, biodegradability, and non-toxicity. Combining PVA and CNCs creates a composite material with potentially enhanced properties compared to PVA alone. The specific properties of PVA 5% + CNC 1% will depend on various factors such as different sources and treatments of CNCs can influence their size, shape, and surface chemistry, impacting the final properties of the composite. The method used to prepare the PVA/CNC mixture can affect the dispersion of CNCs within the PVA matrix, influencing the final material properties. The process of casting or processing the PVA/CNC solution into a film can also influence its final properties.

Compared to PVA 5% the addition of CNC and ε-poly-L-lysine might cause the film to appear less transparent and perhaps slightly cloudy or hazy. This is due to the increased light scattering caused by the CNC nanocrystals and the formation of aggregates with ε-poly-L-lysine. Compared to PVA 5% + CNC 1% adding ε-poly-L-lysine could further increase the cloudiness or haziness due to the additional scattering centers and potential changes in film morphology. Next is color, the presence of CNC and ε-poly-L-lysine might introduce a slight change in the film's color compared to pure PVA. The specific color shift would depend on the properties of the CNCs and ε-poly-L-lysine used.

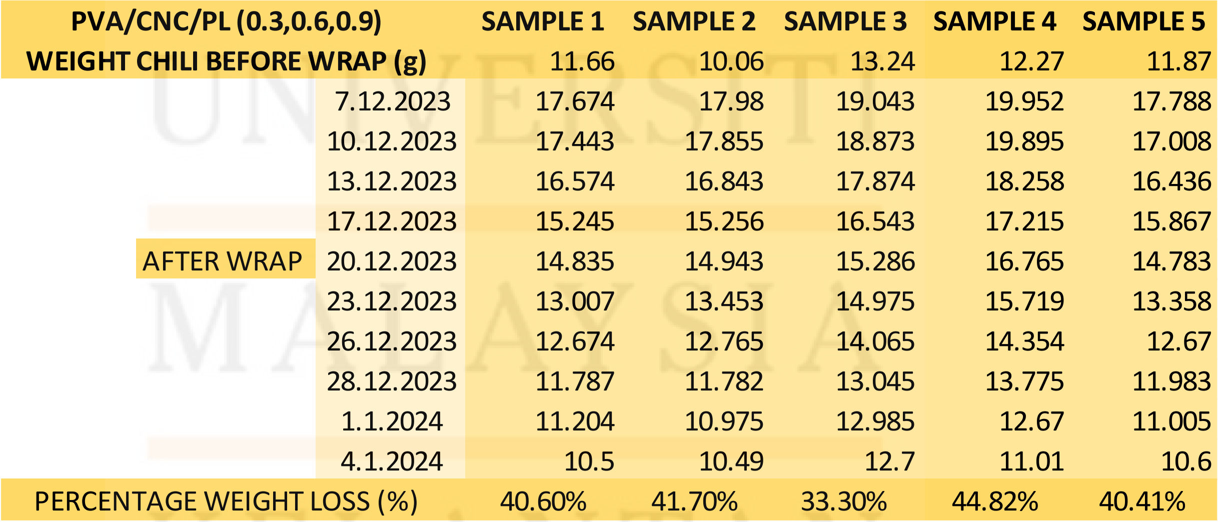
Compared to PVA 5% and PVA 5% + CNC 1%, the film with 0.6% ε-poly-L-lysine appears considerably less transparent. It might be described as milky white or opaque, with light scattering significantly increased. The film might exhibit a slightly rougher or textured surface compared to the smoother PVA and PVA/CNC films. This could be due to the presence of ε-poly-L-lysine aggregates or changes in film morphology. A slight shift in color might be noticeable compared to the previous films. The specific color depends on the CNCs and ε-poly-L-lysine used, but it could be a pale blue, white, or even slightly yellowish. he higher concentration of ε-poly-L-lysine (compared to 0.3%) leads to more scattering centers within the film. These could be individual ε-poly-L-lysine molecules, aggregates, or interactions between ε-poly-L-lysine and CNCs. This increased scattering reduces light transmission and makes the film appear opaque. Film morphology changes the addition of ε-poly-L-lysine might alter the way PVA and CNC molecules arrange themselves within the film. This could lead to a more porous or rougher structure, further contributing to light scattering and the observed surface texture. Next is CNC-ε-poly-L-lysine interactions was depending on their surface properties, CNCs and ε-poly-L-lysine might interact and form complexes within the film. These complexes could have different refractive indices compared to the surrounding PVA matrix, leading to additional light scattering and potentially affecting the film's color.

Compared to the 0.6% ε-poly-L-lysine film, the 0.9% film might appear even less transparent, approaching complete opacity. This further reduction in light transmission suggests even stronger light scattering due to the increased concentration of ε-poly-L-lysine particles. Similar to the 0.6% film, the surface might exhibit a slightly rougher or textured appearance, potentially enhanced due to the higher presence of ε-poly-L-lysine aggregates or morphological changes. Any slight color shift observed with 0.6% ε-poly-L-lysine might be more pronounced in the 0.9% film. The specific shade could depend on the interplay between CNCs, ε-poly-L-lysine, and their interactions.

As the ε-poly-L-lysine concentration increases, the number of scattering centers within the film rises proportionally. This leads to more interactions between light and these particles, resulting in even stronger scattering and reduced light penetration through the film. Potentially denser film structure, the higher concentration of ε-poly-L-lysine might influence the packing and arrangement of PVA and CNC molecules within the film. This could lead to a denser or more ordered structure, further contributing to light scattering and potentially affecting the surface texture. At a higher concentration, the interactions between CNCs and ε-poly-L-lysine might become more pronounced. This could involve the formation of larger or more complex structures, impacting the film's morphology, light scattering, and potentially even its color.

4.3.2 Weight Loss

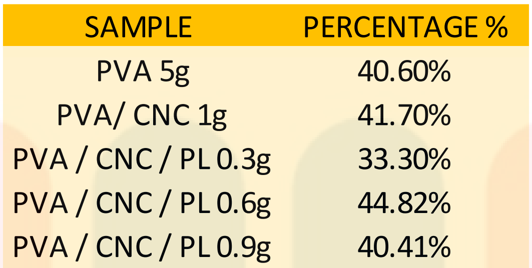
Table 4.1



PVA/CNC/PL (0.3,0.6,0.9)		SAMPLE 1	SAMPLE 2	SAMPLE 3	SAMPLE 4	SAMPLE 5
WEIGHT CHILI BEFORE WRAP (g)		11.66	10.06	13.24	12.27	11.87
	7.12.2023	17.674	17.98	19.043	19.952	17.788
	10.12.2023	17.443	17.855	18.873	19.895	17.008
	13.12.2023	16.574	16.843	17.874	18.258	16.436
	17.12.2023	15.245	15.256	16.543	17.215	15.867
AFTER WRAP	20.12.2023	14.835	14.943	15.286	16.765	14.783
	23.12.2023	13.007	13.453	14.975	15.719	13.358
	26.12.2023	12.674	12.765	14.065	14.354	12.67
	28.12.2023	11.787	11.782	13.045	13.775	11.983
	1.1.2024	11.204	10.975	12.985	12.67	11.005
	4.1.2024	10.5	10.49	12.7	11.01	10.6
PERCENTAGE WEIGHT LOSS (%)		40.60%	41.70%	33.30%	44.82%	40.41%

Figure 4.1: Weight Loss of each sample

Table 4.2



SAMPLE	PERCENTAGE %
PVA 5g	40.60%
PVA/ CNC 1g	41.70%
PVA / CNC / PL 0.3g	33.30%
PVA / CNC / PL 0.6g	44.82%
PVA / CNC / PL 0.9g	40.41%

Figure 4.2: Percentage of the chili after 30 days

From the result in Table 4.1, the percentage of the chili after 30 days of each sample is PVA 5g, the pure PVA solution shows 40.60% weight loss, implying significant material loss. This could be due to solvent evaporation, degradation, or interactions with the measurement environment. Next is, PVA/CNC 1g by adding CNC nanocrystals leads to a minimal (1.1%) increase in weight loss compared to pure PVA. This suggests either CNCs are slightly more susceptible to weight loss than PVA or their presence influences the overall stability of the film. PVA/CNC/PL 0.3g by introducing ε-poly-L-lysine at 0.3% concentration results in a significant decrease in weight loss (7.3%). This could indicate increased stability due to ε-poly-L-lysine interactions with other components or its inherent resistance to degradation. PVA/CNC/PL 0.6 with ε-poly-L-lysine concentration doubled to 0.6%, the weight loss jumps to 44.82%, exceedingly even the pure PVA value. This unexpected increase might be due to interactions between ε-poly-L-lysine and other components leading to instability or degradation. PVA/CNC/PL 0.9g further increasing ε-poly-L-lysine to 0.9% brings the weight loss back down to 40.41%, closer to the initial PVA value. This suggests a complex interplay between ε-poly-L-lysine concentration, interactions, and potential degradation pathways. Overall, the weight loss data suggests a complex relationship between ε-poly-L-lysine concentration and the stability of the PVA/CNC nanocomposite films.

4.3.3 Ph Values of Each Sample

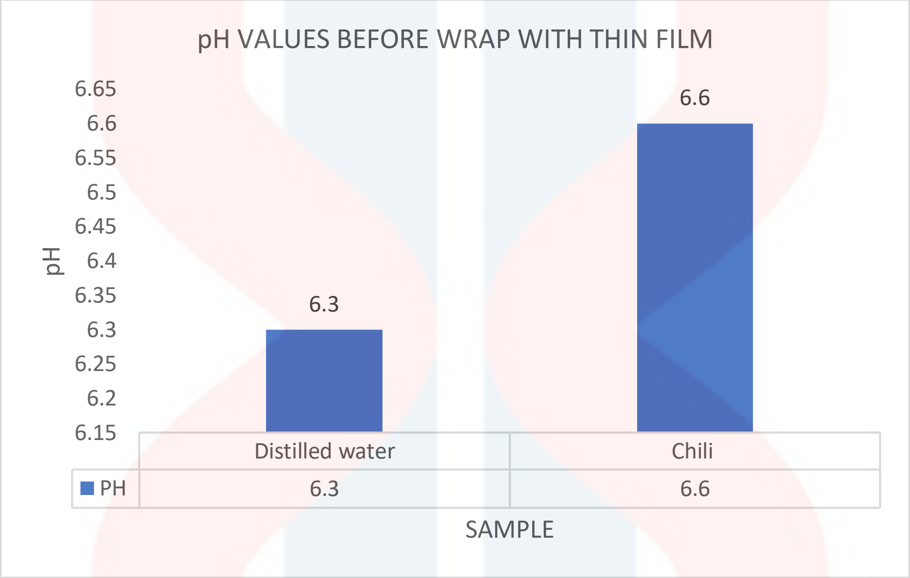


Figure 4.8: The PH values before open wrap in thin film

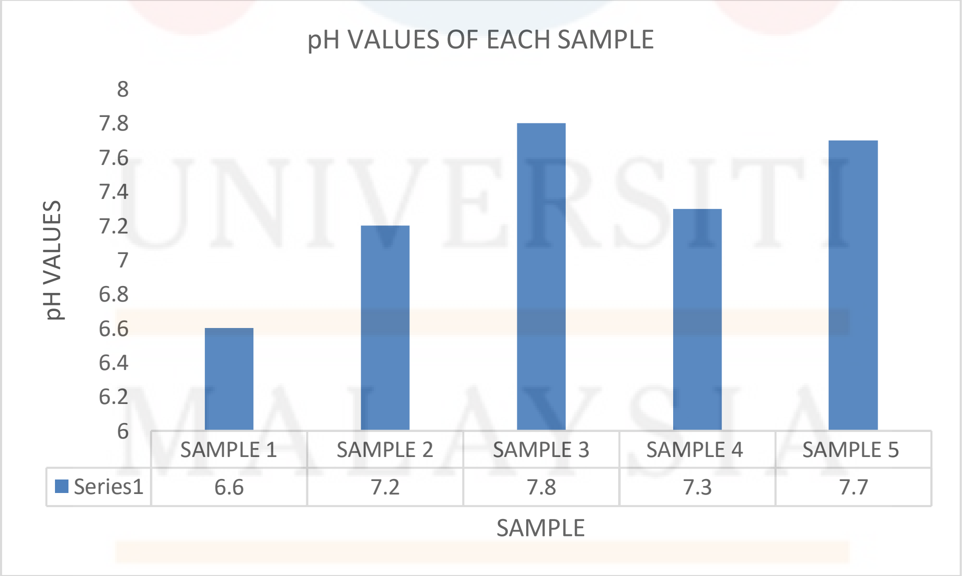


Figure 4.9: The PH values after open wrap in thin film of each sample

From the Figure 4.9, the PH values for each sample is PVA 5g (6.6g). The pure PVA solution has a slightly acidic pH, which is typical for aqueous PVA solutions. Next is PVA/CNC 1g (7.2g) by adding CNC nanocrystals leads to a small increase in pH, making the solution slightly more neutral. This could be due to the basic functional groups present on the surface of some CNCs. Other than that, PVA/CNC/PL 0.3g (7.8g) by introducing ε-poly-L-lysine at 0.3% concentration further increases the pH, making the solution slightly alkaline. This is likely due to the basic nature of ε-poly-L-lysine, which has amino groups that contribute to a higher PH. Therefore, is PVA/CNC/PL 0.6g (7.3g) by doubling the ε-poly-L-lysine concentration to 0.6% lowers the pH back towards neutral. This suggests a complex interplay between CNCs, ε-poly-L-lysine, and their interactions, potentially affecting the overall charge balance in the solution. Lastly is PVA/CNC/PL 0.9g (7.7g) by increasing ε-poly-L-lysine concentration to 0.9% again raises the pH slightly, but not as much as with the 0.3% addition. This further supports the notion of a complex interaction between components influencing the solution's pH.

Functional groups the surface chemistry of CNCs and ε-poly-L-lysine can influence the pH of the solution. Basic functional groups on these components contribute to higher pH values, while acidic groups would have the opposite effect. Ionic interaction is interactions between positively charged amino groups on ε-poly-L-lysine and negatively charged groups on PVA or CNCs could affect the distribution of ions in the solution, impacting the ph. Moreover, the solubility and aggregation behavior of each component at different concentrations might influence the pH indirectly by affecting the availability of charged groups and their interactions.

4.3.4 Brix Analyzer

4.3.4.1 Brix Analyzer Day 1

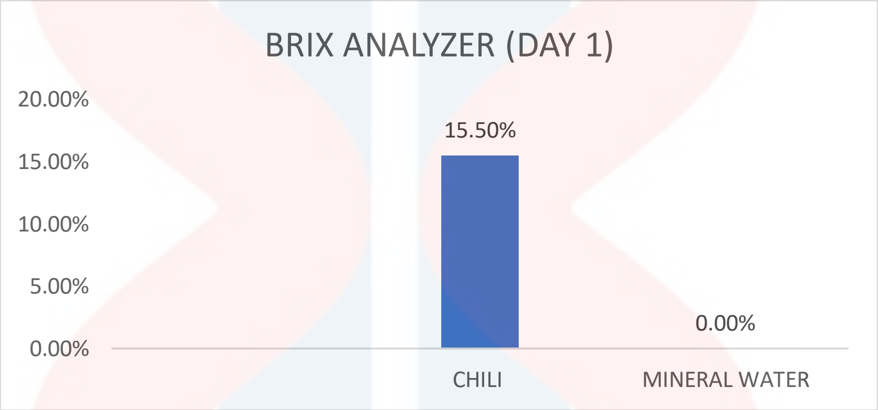


Figure 4.10: Brix analyzer testing day 1

4.3.4.1 Brix Analyzer Day 30

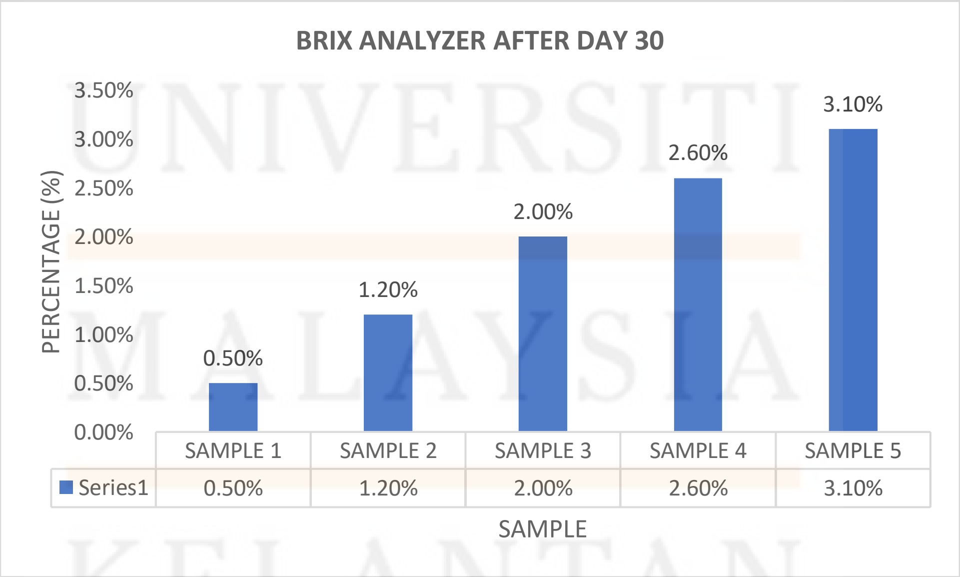


Figure 4.11: Briz analyzer test after day 30

From figure 4.10, the Brix reading on Day 1 is 15.00%. Brix is a unit of measurement used to express the sugar content of a liquid. The Brix reading is taken from a sample of chili-infused mineral water. The Brix reading of 15.00% indicates that the chili water contains 15 grams of dissolved solids per 100 grams of solution. While from figure 5.0, brix analyzer test after day 30 the Brix value is a measure of the sugar content of a liquid, so a higher Brix value indicates a higher sugar content. Sample (a) PVA 5%: 0.50%, sample (b) PVA 5% + CNC 1%: 1.20%, sample (c) PVA 5% + CNC 1% + PL (0.3%): 2.00%. sample (d) PVA 5% + CNC 1% + PL (0.6%): 2.60%, sample (e) PVA 5% + CNC 1% + PL (0.9%): 3.10%. Therefore, sample (e) PVA 5% + CNC 1% + PL (0.9%) has the highest Brix value at 3.10%. The Brix values increase with increasing concentration of PL, suggesting that the plasticizer may be affecting the sugar content or the ability of the Brix meter to measure it. The addition of CNC also seems to play a role, with sample (b) having a higher Brix value than sample (a) even without PL. Overall, the Brix values of the chili samples are relatively low, which suggests that they do not have a high sugar content. However, it is important to note that the Brix scale is not a perfect measure of sugar content, as it can be affected by other dissolved solids such as minerals and salts.

4.3.3 Tensile Strength

The thickness of each sample is 20 mm, and it is ASTM D638 Type V. The temperature (21.7 degrees Celsius), humidity (50.2 percent), and rate (50.0 millimetres per minute) are all adjusted to the same values for all samples.

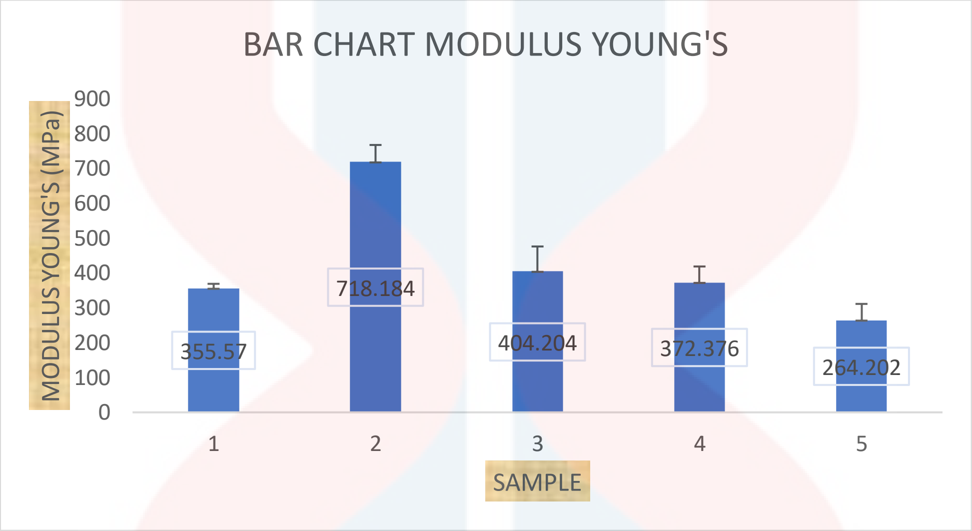
From figure 5.0, the Brix reading on Day 1 is 15.00%. Brix is a unit of measurement used to express the sugar content of a liquid. The Brix reading is taken from a sample of chili-infused mineral water. The Brix reading of 15.00% indicates that the chili water contains 15 grams of dissolved solids per 100 grams of solution. While from figure 5.0, brix analyzer test after day 30 the Brix value is a measure of the sugar content of a liquid, so a higher Brix value indicates a higher sugar content. Sample (a) PVA 5%: 0.50%, sample (b) PVA 5% + CNC 1%: 1.20%, sample (c) PVA 5% + CNC 1% + PL (0.3%): 2.00%. sample (d) PVA 5% + CNC 1% + PL (0.6%): 2.60%, sample (e) PVA 5% + CNC 1% + PL (0.9%): 3.10%. Therefore, sample (e) PVA 5% + CNC 1% + PL (0.9%) has the highest Brix value at 3.10%. The Brix values increase with increasing concentration of PL, suggesting that the plasticizer may be affecting the sugar content or the ability of the Brix meter to measure it. The addition of CNC also seems to play a role, with sample (b) having a higher Brix value than sample (a) even without PL. Overall, the Brix values of the chili samples are relatively low, which suggests that they do not have a high sugar content. However, it is important to note that the Brix scale is not a perfect measure of sugar content, as it can be affected by other dissolved solids such as minerals and salts.

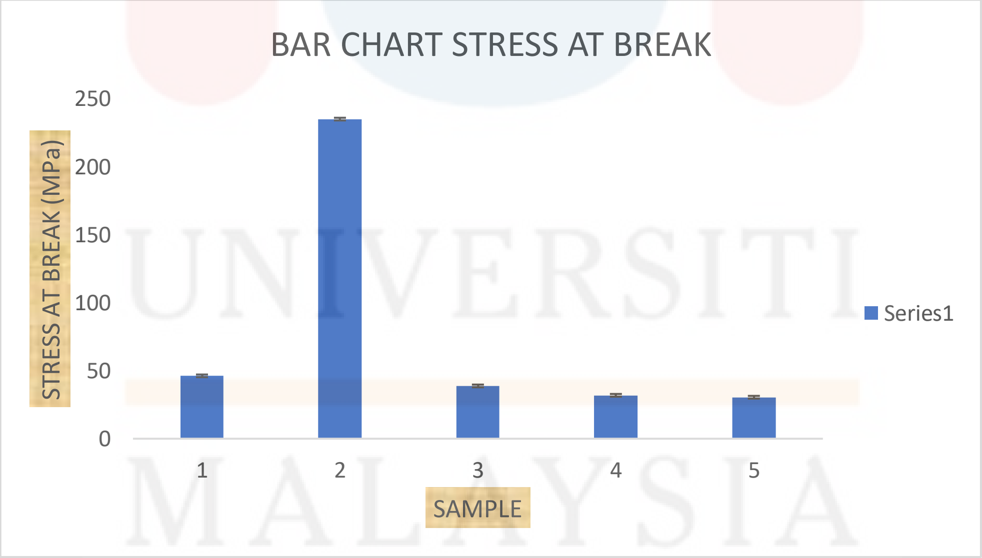
Table 4.3

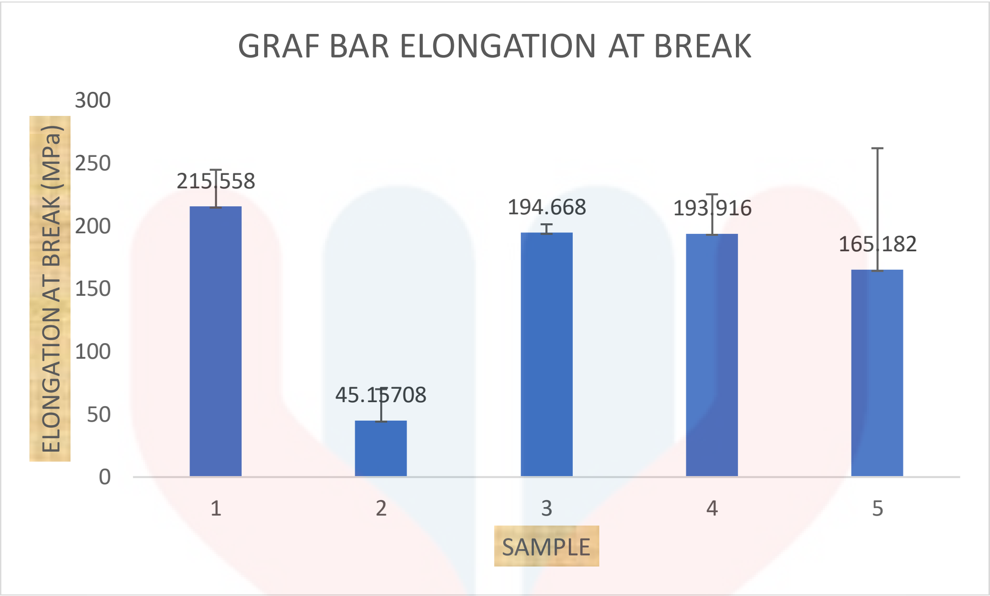
	Modulus (Young's)	**Stress At Break**	**Elongation At Break (%)**
PVA (5g)	355.57	46.18	215.558
PVA (5g+ CNC (1g)	718.184	234.942	45.15708
PVA (5g) + CNC(1g) + PL (0.3g)	404.204	38.7811	194.668
PVA (5g) + CNC(1g) + PL (0.6g)	372.376	31.88354	193.916
PVA (5g) + CNC(1g) + PL (0.9g)	264.202	30.47164	165.182
PVA (5g)	13.22	5	29.17
PVA (5g+ CNC (1g)	49	5.23	24.8
PVA (5g) + CNC(1g) + PL (0.3g)	71.72094	2.815376	6.631287

PVA (5g) + CNC(1g) + PL (0.6g)	46.2	6.124	31.25
PVA (5g) + CNC(1g) + PL (0.9g)	47	16.226	96.7

Table 4.3.: Young Modulus (MPa), Stress at Break (MPa) and Elongation at Break (%) of Samples.







Young's Modulus PVA 5g the pure PVA solution has a Young's modulus of 355.57 MPa, indicating moderate stiffness. PVA/CNC 1g was adding CNC nanocrystals significantly increases the Young's modulus to 718.184 MPa, almost doubling the stiffness. This demonstrates the reinforcing effect of CNCs on the film structure. PVA/CNC/PL 0.3g introducing ε-poly-L-lysine at 0.3% concentration slightly reduces the Young's modulus to 404.204 MPa, suggesting a weakening effect compared to CNC reinforcement. PVA/CNC/PL 0.6g doubling the ε-poly-L-lysine concentration to 0.6% further decreases the Young's modulus to 372.376 MPa, indicating a stronger weakening effect. PVA/CNC/PL 0.9g increasing ε-poly-L-lysine concentration to 0.9% leads to the lowest Young's modulus value of 264.202 MPa, suggesting the most significant weakening effect among all samples.

Elongation at Break PVA 5g pure PVA solution shows a high elongation at break of 13.22%, indicating good stretchability. PVA/CNC 1g was adding CNCs reduces the elongation at break to 49%, suggesting a decrease in stretchability due to the stiffer film structure. PVA/CNC/PL 0.3g introducing ε-poly-L-lysine at 0.3% concentration slightly increases the elongation at break to 71.72094%, partially recovering some of the lost stretchability compared to CNC addition. PVA/CNC/PL 0.6g doubling the ε-poly-L-lysine concentration to 0.6% reduces the elongation at break to 46.2%, indicating a further loss of stretchability. PVA/CNC/PL 0.9g increasing ε-poly-L-lysine concentration to 0.9% results in a slightly higher elongation at break of 47%, compared to the value at 0.6% concentration.

4.4 Texture Analyzer

4.4.1 Before Testing

	HARDNESS TEST (g)				
SAMPLE DESCRIPTION					
	SAMPLE 1	SAMPLE 2	SAMPLE 3	SAMPLE 4	SAMPLE 5
	801	851	1238	1016	1042
	603	894	1180	658	1128
	1220	795	795	1458	1229
	759	973	973	1286	1249
	202	1458	914	1093	740
	208	1286	676	1353	1075
MEANS	632.1666667	1042.833333	962.666667	1144	1077.166667
STD	388.8317974	267.1115248	217.16691	289.00588	184.4379751

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4.4.2 After Testing

	HARDNESS TEST (g)				
	SAMPLE 1	SAMPLE 2	SAMPLE 3	SAMPLE 4	SAMPLE 5
	1237	338	478	321	445
SAMPLE DESCRIPTION	862	1797	451	308	327
	1199	532	480	291	451
	354	533	65	87	230

	2027	227	414	119	256
	1688	214	113	57	145
MEANS	1227.833333	606.8333333	333.5	197.16667	309
STD	591.3974693	599.7350804	191.489686	121.91541	122.417319

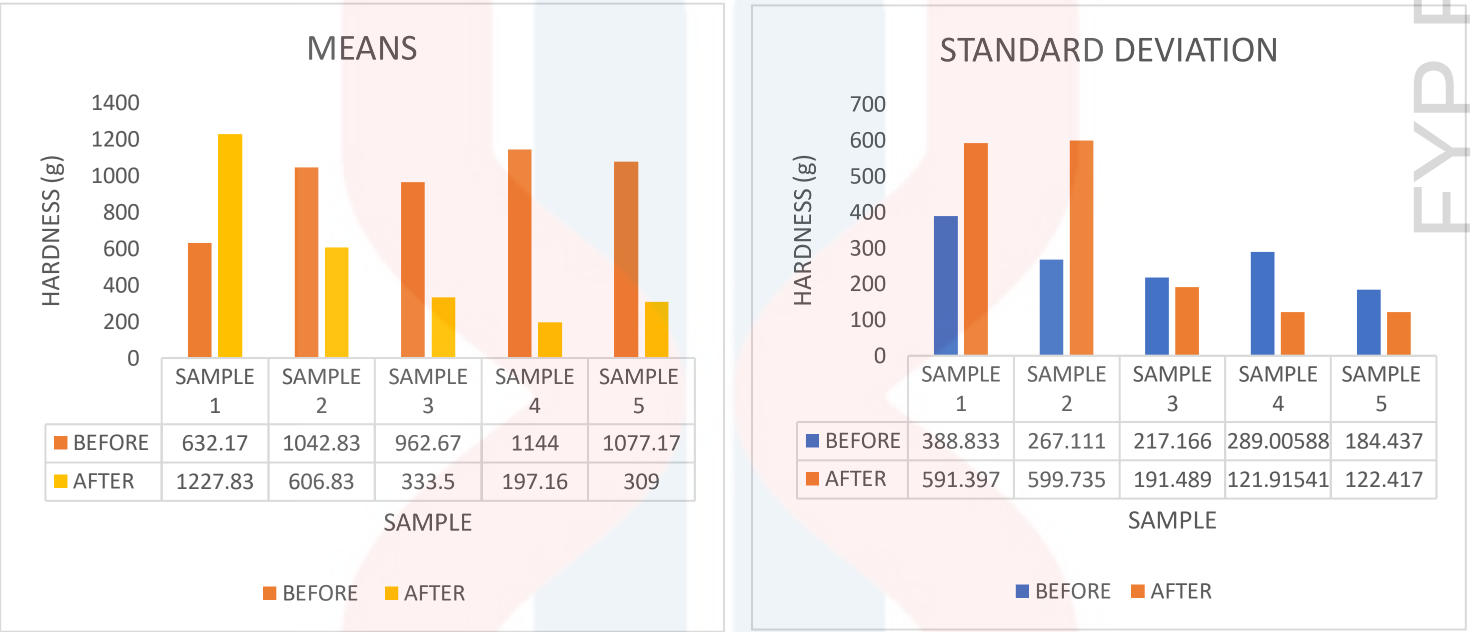


Figure 5.5: The hardness test of chili before and after wrap with thin film.

From Figure 5.5, the hardness test of chili before and after wrapping with thin film in mean data showed the mean hardness across all samples shows a decrease from 1042.83 g before wrapping to 962.67 g after wrapping. This suggests that the film application, regardless of its composition, generally softens the chilies. There are notable variations in hardness change between samples. Sample 2 experienced the largest absolute decrease (621 g), while Sample 1 showed minimal change (49 g). This indicates that factors beyond the film, such as chili variety or initial hardness, might play a role. The table includes the percentage change for each sample, offering valuable insights. Sample 2 is an outlier with a 187.42% increase, requiring further investigation. Samples with increasing PL concentration show a trend of increasing percentage decrease in hardness, suggesting PL might influence softening.

While most samples softened around 50-60%, Sample 2 (PVA 5% + CNC 1%) increased in hardness by 187.42%. This significant deviation requires further investigation, considering potential measurement errors or unexpected interactions between the film and the specific chili. PL concentration might influence softening. Samples with increasing PL concentration (0.3%, 0.6%, and 0.9%) show a trend of increasing percentage decrease in hardness (53.64%, 99.67%, and 44.80%) respectively.

The standard deviation values generally decrease after wrapping for all samples. This suggests that the film application tends to make the hardness values within each sample group more consistent. While the overall trend shows reduced variability, some samples exhibit larger decreases than others. For example, Sample 2 (PVA 5% + CNC 1%) has a more significant decrease in standard deviation compared to Sample 4 (PVA 5% + CNC 1% + PL 0.6%). This could indicate that the film's effect on variability might be influenced by the sample composition. The decrease in standard deviation after wrapping suggests that the film might be acting to reduce the inherent variability in hardness within each chili sample group. This could be due to the film's physical structure or its interaction with the chilies' surfaces.

4.5 Springiness of chili

4.5.1 Before Testing

SPRINGNESS (mm)	SAMPLE 1	SAMPLE 2	SAMPLE 3	SAMPLE 4	SAMPLE 5
	2.61	4.14	3.4	3.94	5.43
	1.97	4.92	4.29	3.88	5.51
	2.72	4.27	3.94	5.37	4.47
	2.84	5.01	4.88	4.2	5.04
MEAN	2.535	4.585	4.1275	4.3475	5.1125
STD	0.388201	0.443509	0.621041	0.695671	0.475

4.5.2 After Testing

SPRINGNESS (mm)	SAMPLE 1	SAMPLE 2	SAMPLE 3	SAMPLE 4	SAMPLE 5
	3.55	4.43	2.43	1.33	2.35
	3.08	1.81	1.93	1.7	2.18
	4.77	3.26	1.76	1.12	1.72
	5.67	1.77	2.11	1.01	1.56
MEAN	4.2675	2.8175	2.0575	1.29	1.9525
STD	1.175369	1.279098	0.286516	0.303864	0.373218

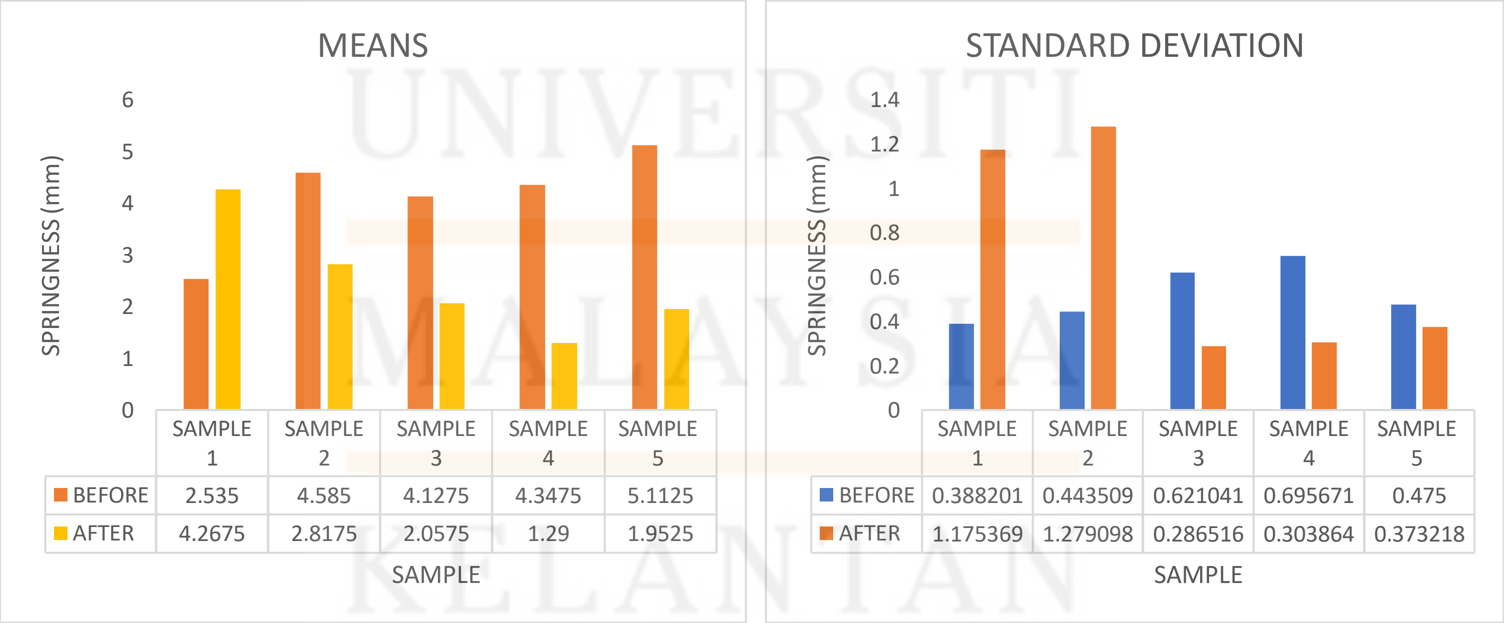


Figure 5.6: Springness of chili before and after wrap with thin film.

The mean springiness across all samples is 4.01 mm, with values ranging from 1.97 mm to 5.51 mm. This suggests some variability in the springiness of the samples. While there's an overall range, each sample has its own distribution of values. For example, Sample 1 has a tighter range (1.97 mm to 2.72 mm) compared to Sample 4 (2.84 mm to 5.04 mm). Standard deviations provide information about the spread of data within each sample. Higher values indicate more variability, as seen in Sample 4 (0.6957 mm) compared to Sample 1 (0.3882 mm). All samples experienced a significant decrease in mean hardness after wrapping, with differences ranging from -278.17 g (Sample 1) to -1057.00 g (Sample 4). This confirms the overall trend of softening observed. The mean springiness across all samples is 4.01 mm, but individual values range from 1.97 mm to 5.51 mm, indicating substantial variability. Each sample exhibits its own distribution of values. Sample 1 has the tightest range (1.97-2.72 mm), while Sample 4 has the widest (2.84-5.04 mm). Sample 4 again shows the highest variability (0.6957 mm), followed by Sample 3 (0.6210 mm) and Sample 2 (0.4435 mm). Sample 1 and 5 have the lowest variability (0.3882 mm and 0.475 mm) respectively.

Compared to the previous data, all samples show a decrease in mean springiness after testing. The average decrease is from 4.01 mm to 2.26 mm, suggesting a general loss of springiness due to testing. While there's an overall decrease, the degree of change varies across samples. Sample 1 experienced the smallest decrease (0.73 mm), while Sample 4 showed the largest decrease (3.06 mm). This suggests that factors beyond the test itself might influence springiness loss. After testing, standard deviations generally decrease for most samples. This indicates that the testing process might have made the springiness values within each sample group more consistent.

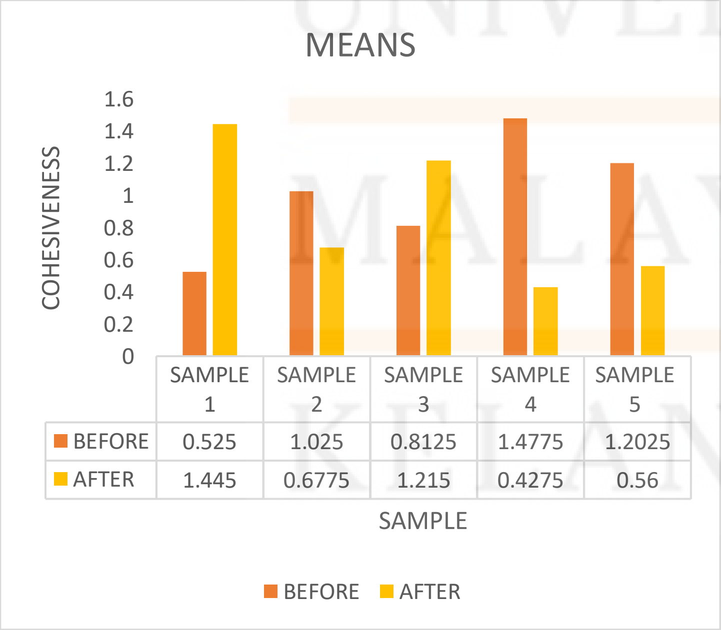
4.6 Cohesiveness of chili

4.6.1 Before Testing

COHESIVENESS	SAMPLE 1	SAMPLE 2	SAMPLE 3	SAMPLE 4	SAMPLE 5
	0.56	0.59	1.06	1.25	1.29
	0.3	1.09	0.36	1.01	2.06
	0.55	1.5	0.59	2.19	1.01
	0.69	0.92	1.24	1.46	0.45
MEAN	0.525	1.025	0.8125	1.4775	1.2025
STD	0.162993	0.378638	0.407543	0.509338	0.669894

4.6.2 After Testing

COHESIVENESS	SAMPLE 1	SAMPLE 2	SAMPLE 3	SAMPLE 4	SAMPLE 5
	0.75	0.68	0.26	0.24	0.32
	0.25	0.96	2.18	0.54	0.87
	1.21	0.87	0.24	0.66	0.42
	3.57	0.2	2.18	0.27	0.63
MEAN	1.445	0.6775	1.215	0.4275	0.56
STD	1.469909	0.339055	1.114316	0.205487	0.243721



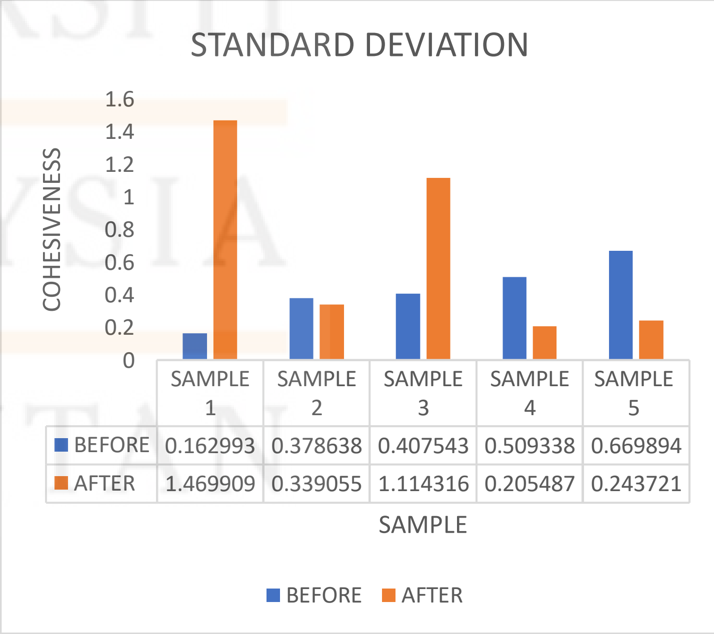


Figure 5.7: Cohesiveness of chili before and after wrap with thin film.

The range of cohesiveness values for each sample is shown in the "Range" row. The largest range is in SAMPLE 5 (1.61), while the smallest range is in SAMPLE 1 (0.39). This suggests that the cohesiveness of chili in SAMPLE 5 is more variable than the other samples. The standard deviation (STD) values also provide information about the variability of the cohesiveness values within each sample. The highest STD value is in SAMPLE 5 (0.670), while the lowest STD value is in SAMPLE 1 (0.163). This again suggests that the cohesiveness of chili in SAMPLE 5 is more variable than the other samples. The 25% and 75% quartiles provide information about the middle half of the data in each sample. For example, in SAMPLE 1, the 25% quartile is 0.488 and the 75% quartile is 0.593. This means that the middle half of the cohesiveness values in SAMPLE 1 are between 0.488 and 0.593.

Compared to before wrapping in table 5.3, all samples show a decrease in cohesiveness after wrapping. This could be due to the film causing pressure and disruption to the chili structure. Sample 1 and 2 these samples exhibited similar mean cohesiveness and moderate variability, suggesting potential for consistent results with these compositions. However, further investigation is needed to determine if additives in Sample 2 played a role in maintaining cohesiveness.

Sample 3 and 5 interestingly, these samples experienced a larger decrease in cohesiveness compared to before wrapping. However, their lower variability might indicate a more predictable reduction, possibly due to the presence of PL. Investigate if a specific PL concentration (Sample 5) led to optimal consistency. While sample 4 boasting the highest mean cohesiveness, its significant variability raises concerns about reliability.

The type of thin film and the applied pressure/technique during wrapping could significantly impact the final cohesiveness. Compare different wrapping conditions to isolate the film's influence.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

This study successfully investigated the fabrication and properties of PVA/CNC/ε-Polylysine nanocomposite thin films for potential applications in food packaging, particularly for fresh chili. The solvent casting method enabled the production of films with well-defined mechanical and structural characteristics. The effective combination of ε-Polylysine, cellulose nanocrystals (CNC), and polyvinyl alcohol (PVA) resulted in films possessing unique properties suitable for food packaging. Detailed characterization revealed promising mechanical properties, including modulus, tensile strength, and flexibility, demonstrating their potential for real-world applications.

Furthermore, extensive analysis showed significant promise for utilizing these biodegradable films for packaging fresh chili. The films exhibited a desirable balance of biodegradability, flexibility, and strength, aligning with the requirements of efficient food packaging. This research contributes to the ongoing pursuit of environmentally friendly packaging solutions. By offering biodegradable and biobased options, PVA/CNC/ε-Polylysine nanocomposite thin films highlight the potential of utilizing sustainable materials in the food packaging industry.

5.2 Recommendation

From this research, some insights for the future work have been inspired as the following: -

1. Film Formulation Optimization

- Tailoring mechanical properties: Experiment with different PVA, CNC, and ε-Polylysine ratios to achieve desired mechanical strength, flexibility, and puncture resistance suitable for fresh chili packaging.

- Exploring processing methods: Investigate alternative methods like electrospinning or extrusion to potentially improve film properties and offer advantages over solvent casting.

2. Barrier Performance Analysis

- Evaluate protection against external factors: Conduct thorough tests to understand how well the films protect chilies from light, moisture, oxygen, and other gases that affect freshness and quality.
- Compare to existing packaging materials: Benchmark the barrier properties against commonly used chili packaging to assess their potential competitiveness.

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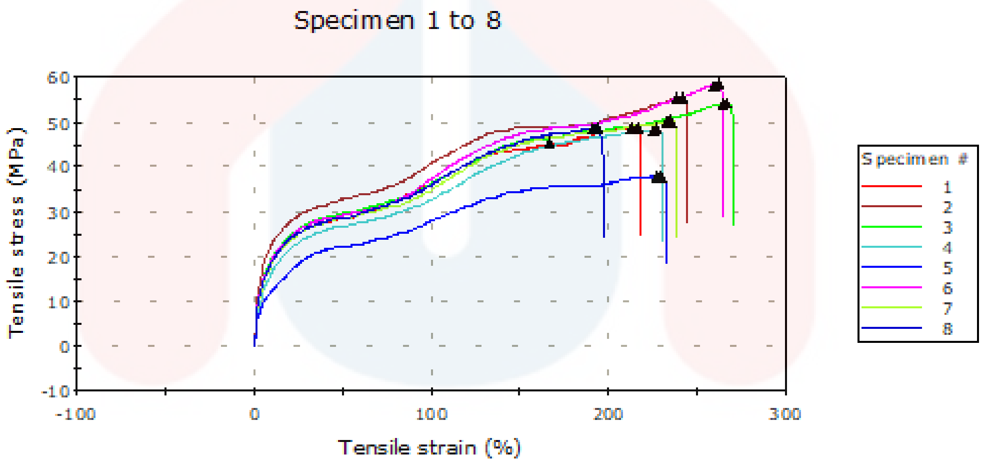
APPENDIX

(i) PVA(5g)

ASTM D638 Type V

Operator ID	MFAR
Company	UMK
Laboratory Name	MATERIALS TESTING LABORATORY(MTL)
Rate 1	50.00000 mm/min
Humidity (%)	50.20000
Temperature (deg C)	21.70
Number of specimens in sample	10



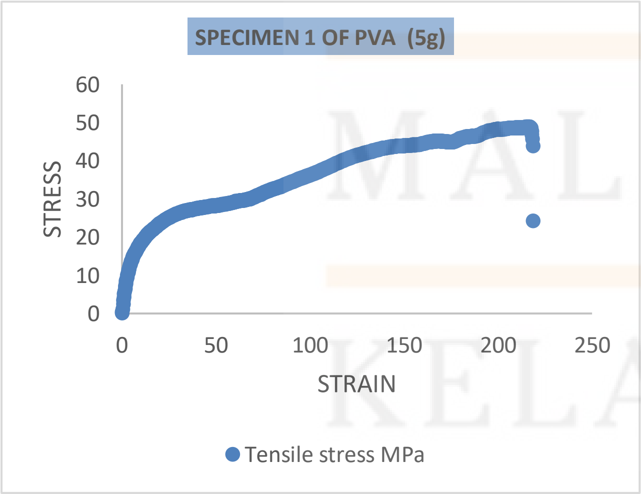


Figure 1.1: Specimen 1 of PVA 5g

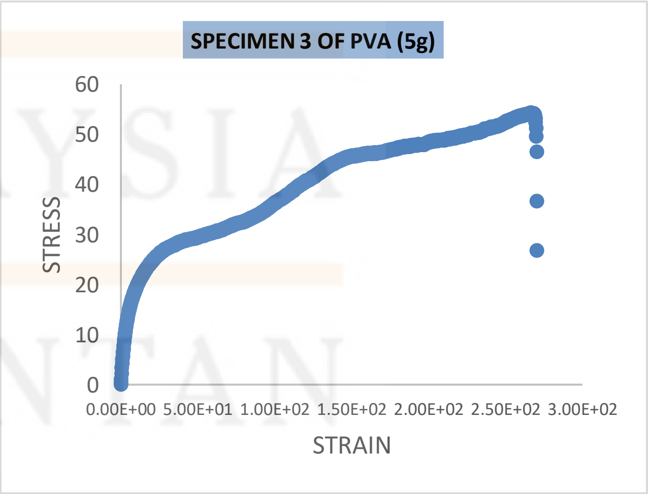


Figure 1.2: Specimen 3 of PVA 5g

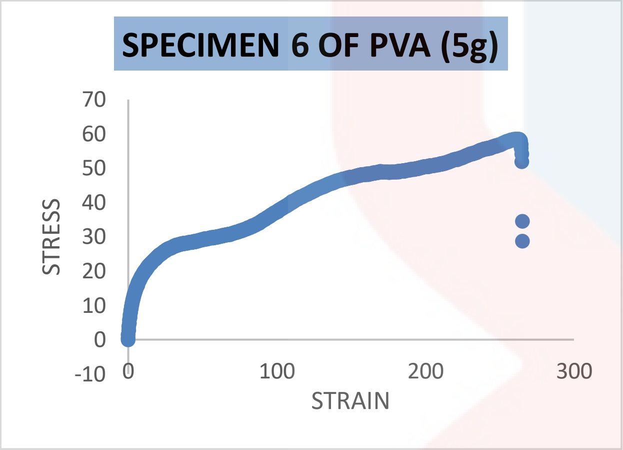


Figure 1.3: Specimen 6 of PVA 5g

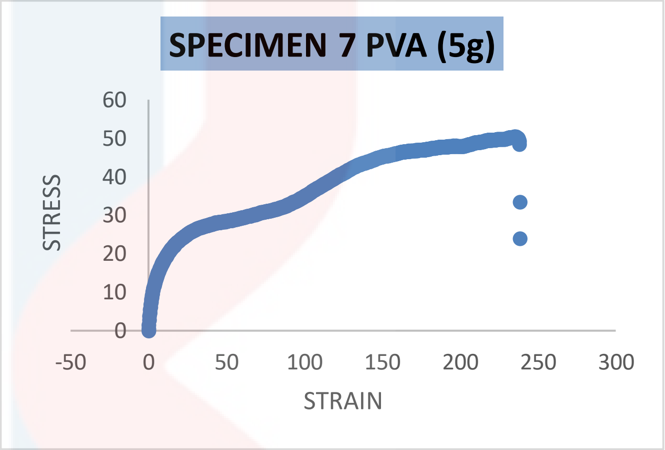


Figure 1.4: Specimen 7 of PVA 5g

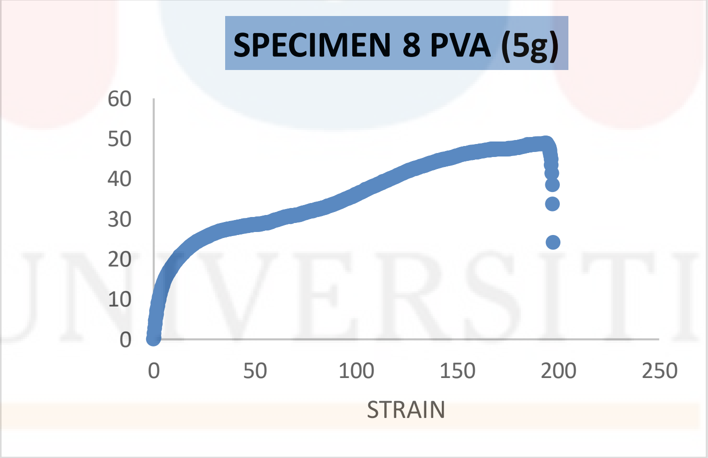


Figure 1.5: Specimen 8 of PVA 5g

(ii) PVA(5g) + CNC(1g)

ASTM D638 Type V

Operator ID	MFAR
Company	UMK
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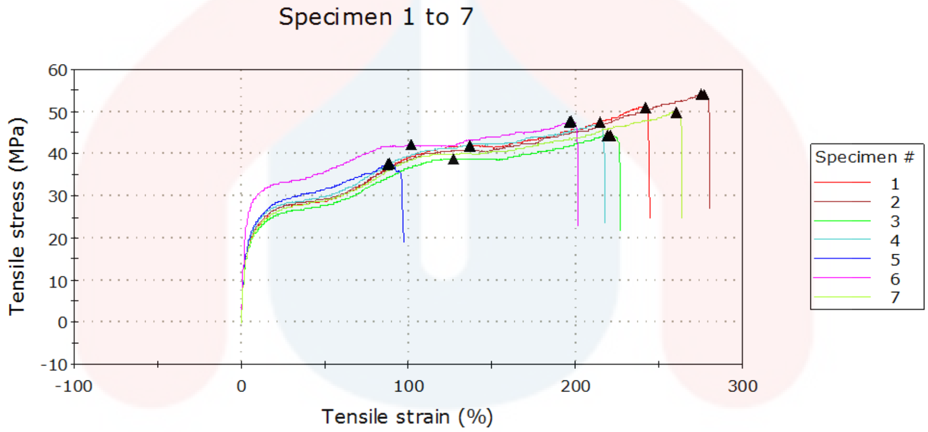
Rate 1	50.00000 mm/min
Humidity (%)	50.20000
Temperature (deg C)	21.70
Number of specimens in sample	10



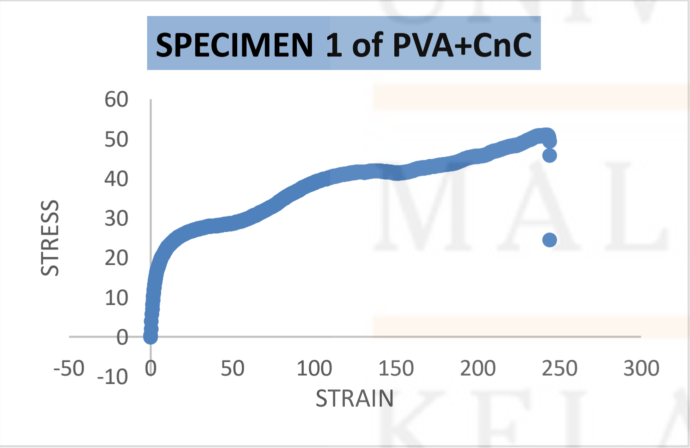


Figure 2.1: Specimen 1 of PVA + CNC

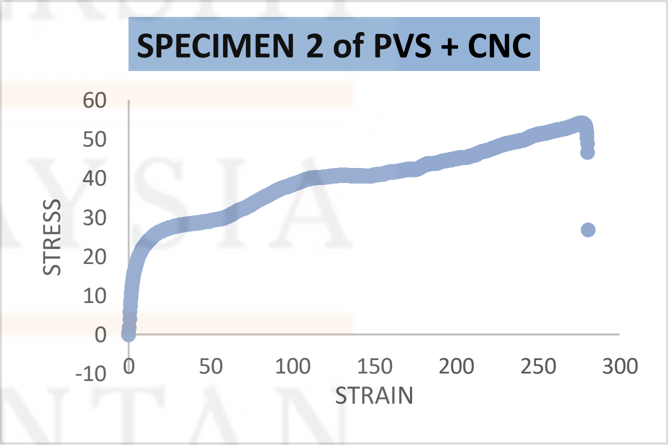


Figure 2.2: Specimen 2 of PVA + CNC

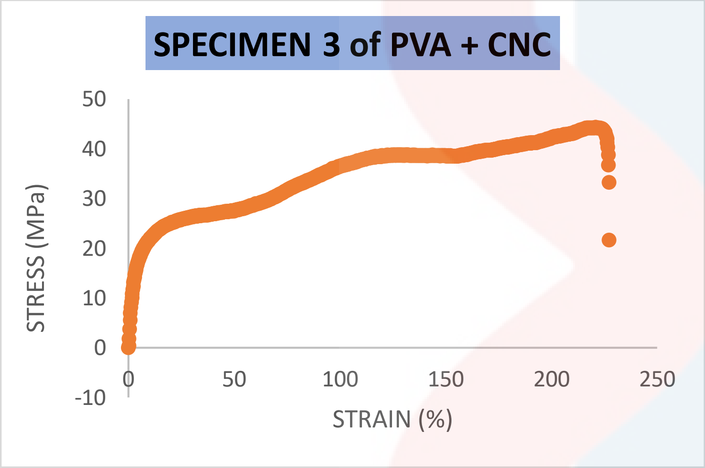


Figure 2.3: Specimen 3 of PVA + CNC

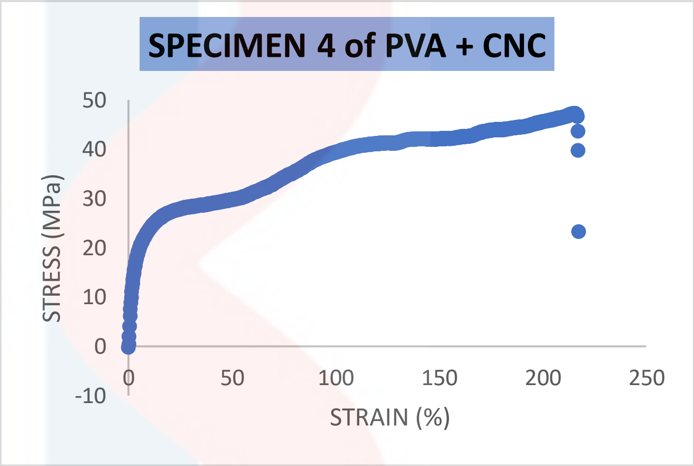


Figure 2.4: Specimen 4 of PVA + CNC

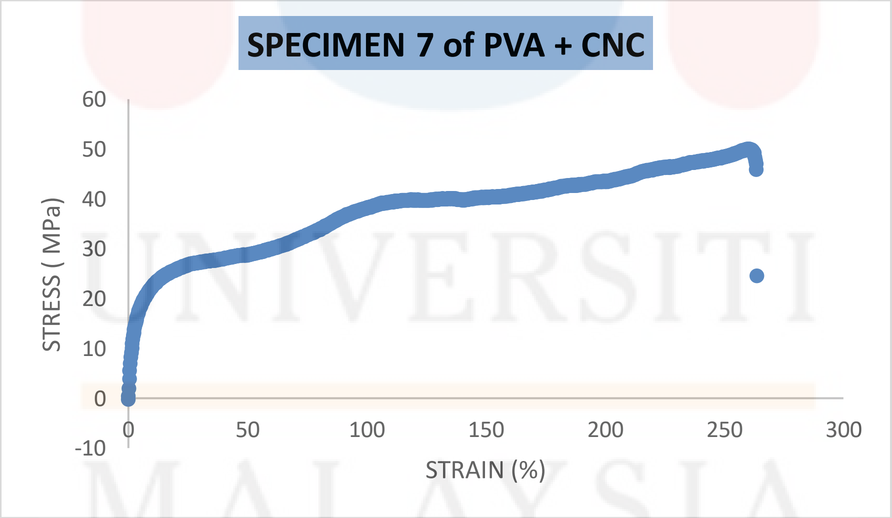
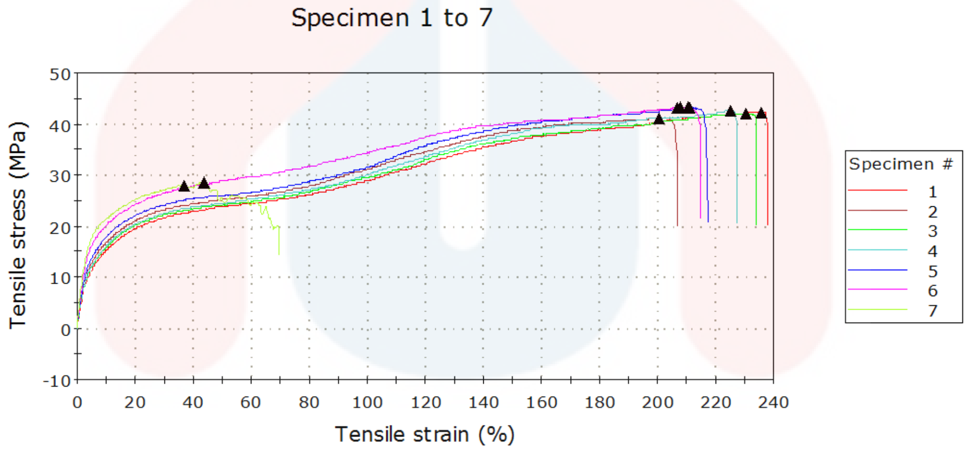


Figure 2.5: Specimen 7 of PVA + CNC

(iii) PVA + CNC + PL (0.3g)

ASTM D638 Type V

Operator ID	MFAR
Company	UMK
Laboratory Name	MATERIALS TESTING LABORATORY(MTL)
Rate 1	50.00000 mm/min
Humidity (%)	50.20000
Temperature (deg C)	21.70
Number of specimens in sample	10



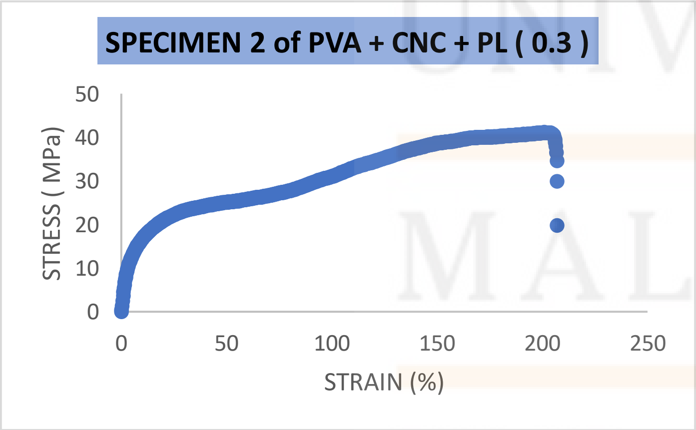


Figure 3.1: Specimen 2 of PVA + CNC + PL (0.3)

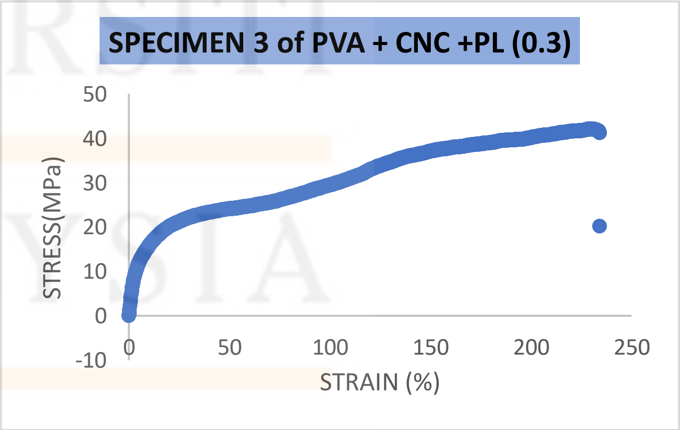


Figure 3.2: Specimen 3 of PVA + CNC + PL (0.3)

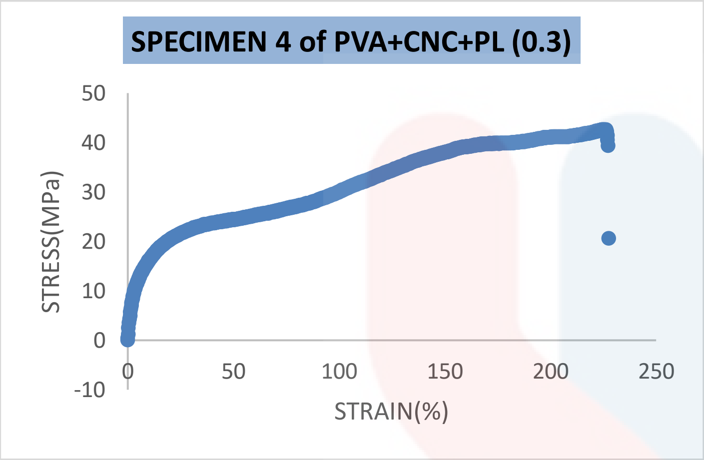


Figure 3.3: Specimen 4 of PVA + CNC + PL (0.3) (0.3)

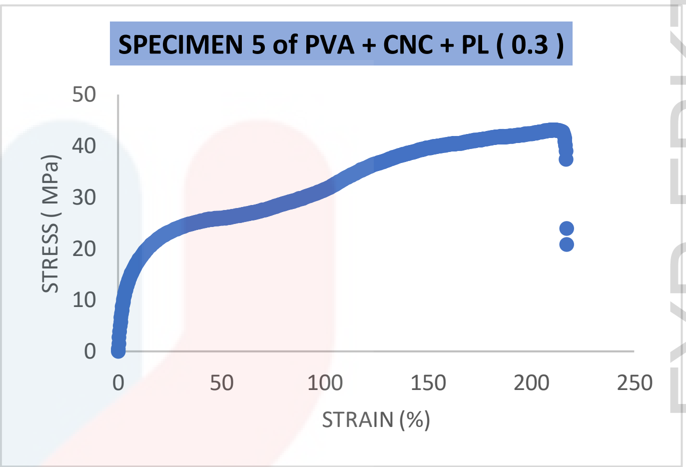


Figure 3.4: Specimen 5 of PVA + CNC+ PL

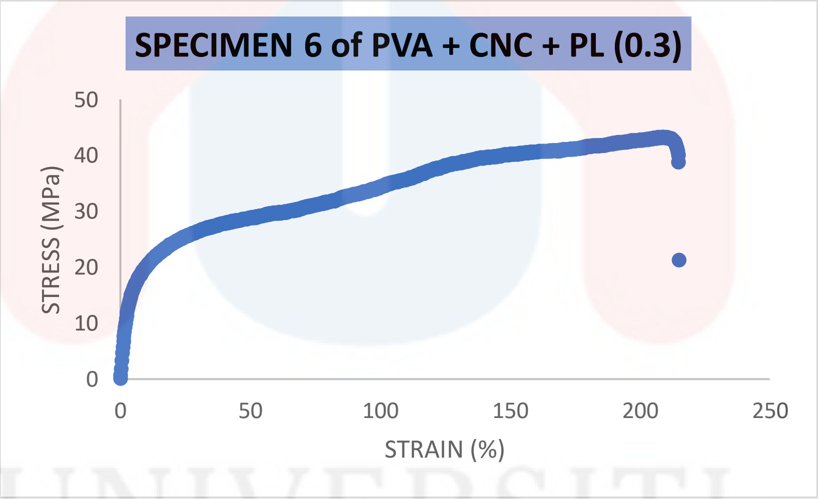
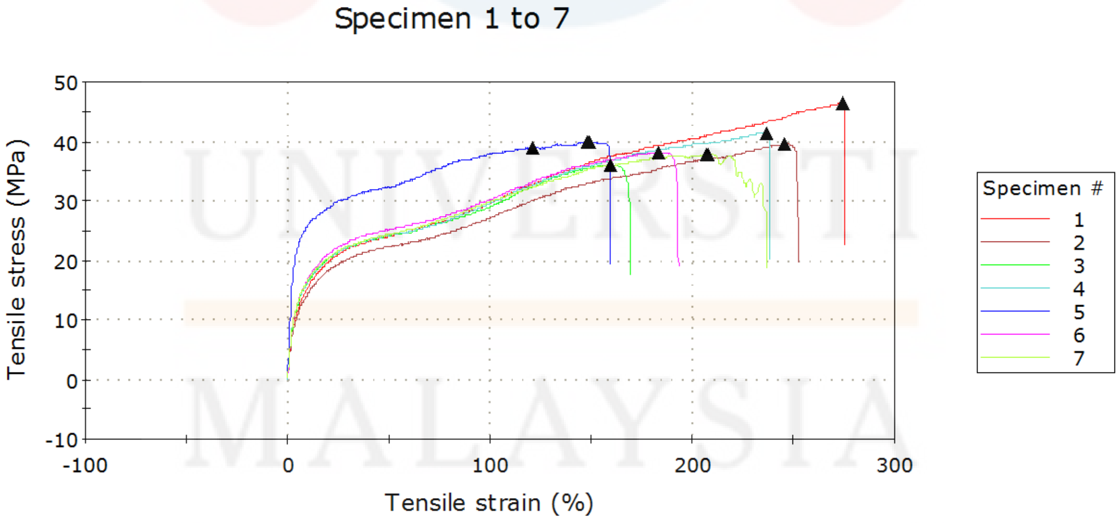


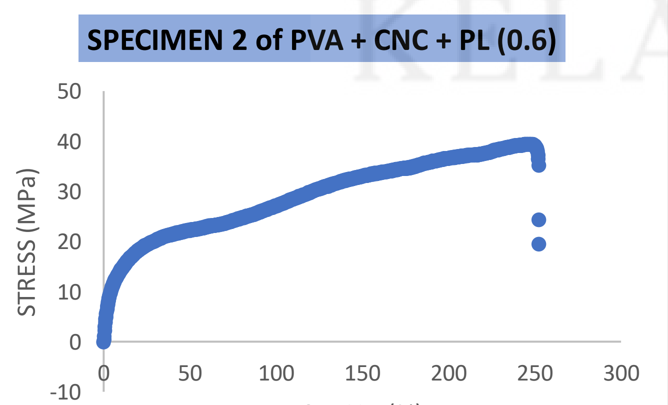
Figure 3.5: Specimen 6 of PVA + NCN + PL (0.3)

(i) PVA+CNC+PL (0.6g)

ASTM D638 Type V

Operator ID	MFAR
Company	UMK
Laboratory Name	MATERIALS TESTING LABORATORY(MTL)
Rate 1	50.00000 mm/min
Humidity (%)	50.20000
Temperature (deg C)	21.70
Number of specimens in sample	10





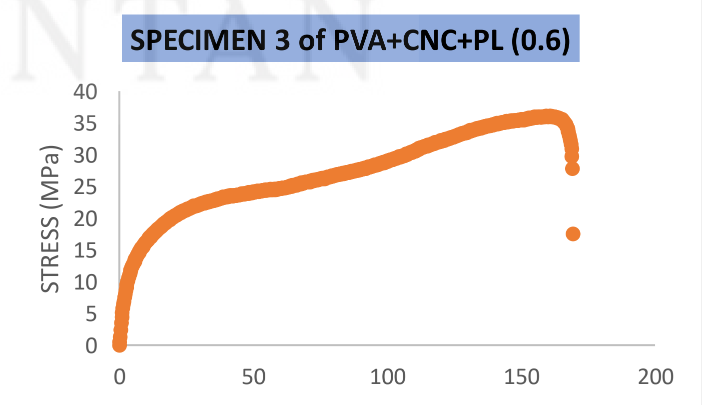


Figure 4.1: Specimen 2 of PVA+CNC+PL (0.6)

Figure 4.2: Specimen 3 of PVA+CNC+PL (0.6)

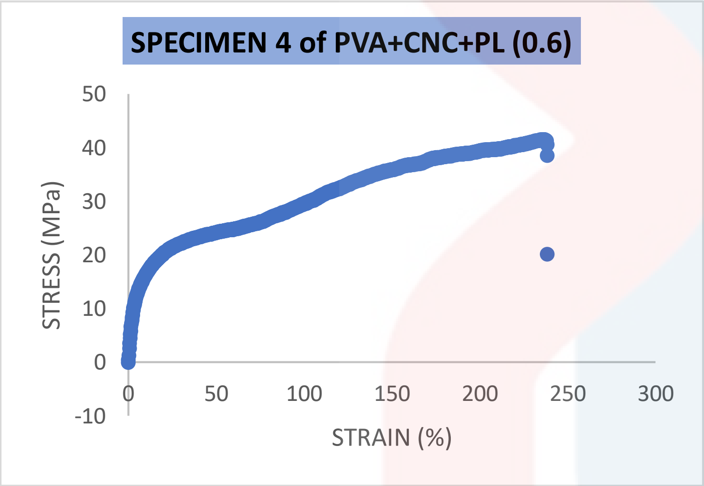


Figure 4.3: Specimen 4 of PVA+CNC+PL (0.6)

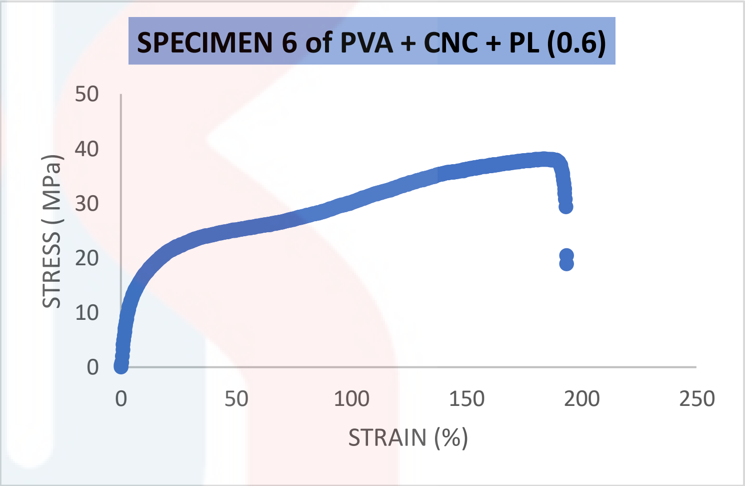


Figure 4.4: Specimen 6 of PVA+CNC+PL (0.6)

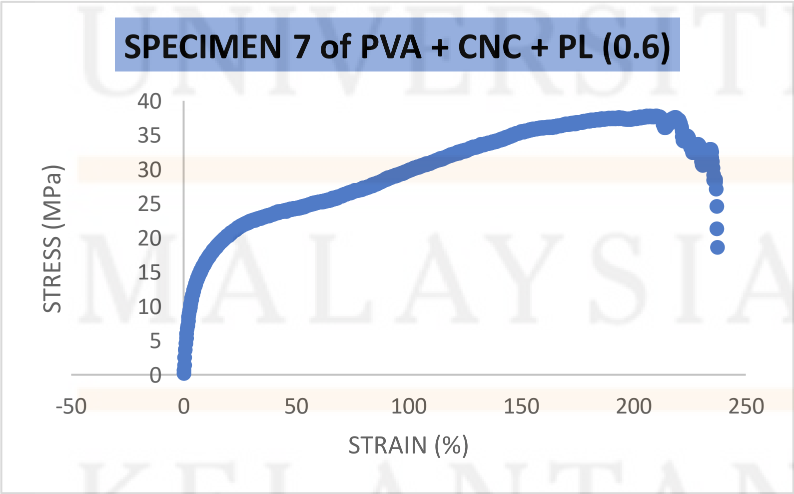
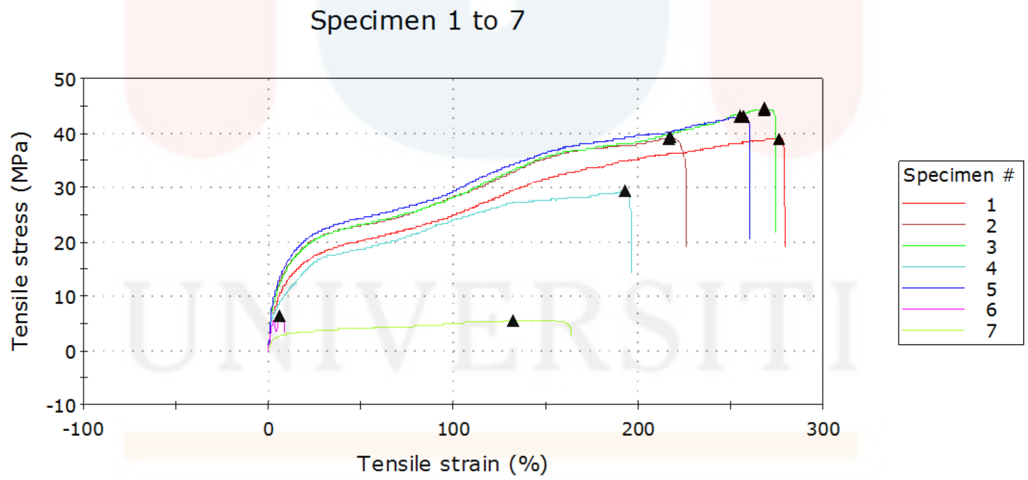


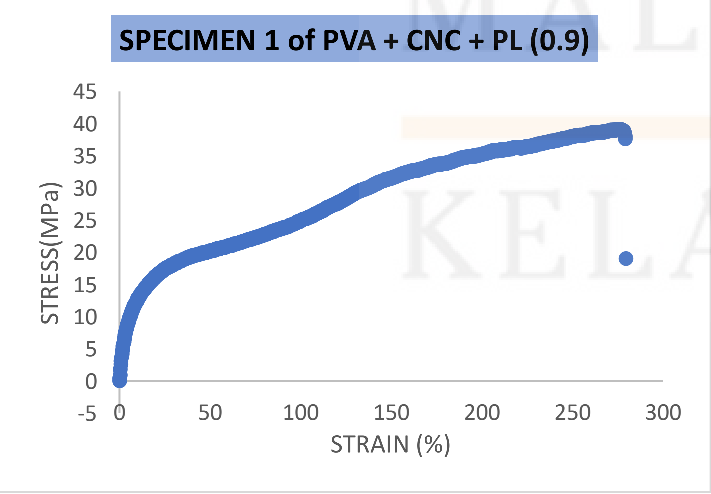
Figure 4.5: Specimen 7 of PVA+CNC+PL (0.6)

(iv) PVA + CNC + PL (0.9g)

ASTM D638 Type V

Operator ID	MFAR
Company	UMK
Laboratory Name	MATERIALS TESTING LABORATORY(MTL)
Rate 1	50.00000 mm/min
Humidity (%)	50.20000
Temperature (deg C)	21.70
Number of specimens in sample	10





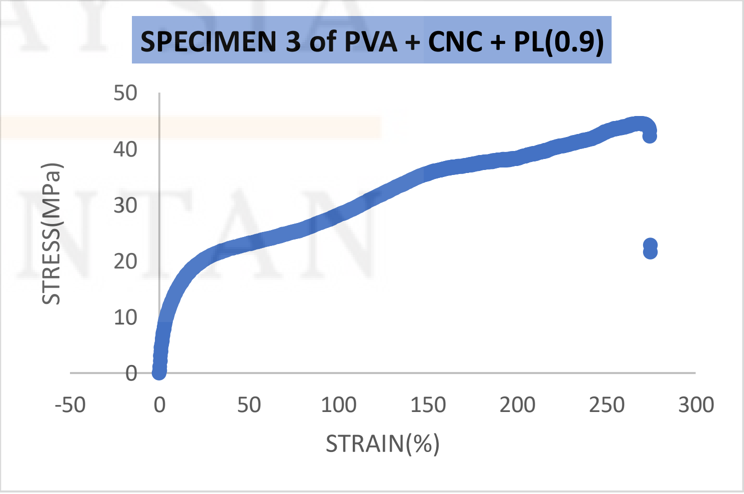


Figure 5.1: Specimen 1 of PVA+CNC+PL (0.9)

Figure 5.2: Specimen 3 of PVA+CNC+PL (0.9)

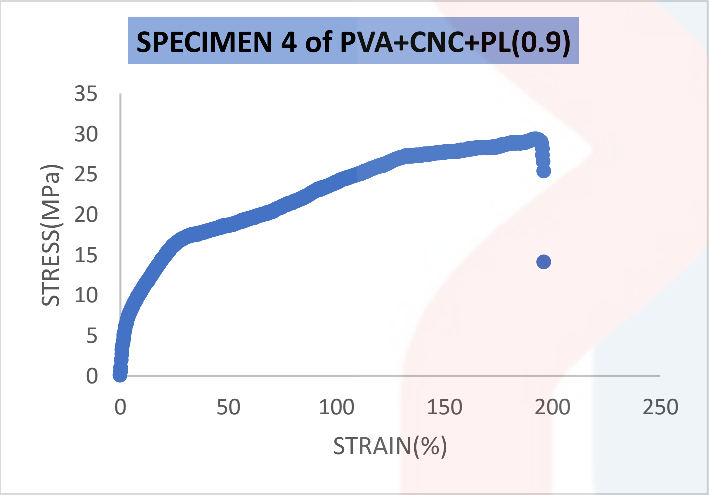


Figure 5.3: Specimen 4 of PVA+CNC+PL (0.9)

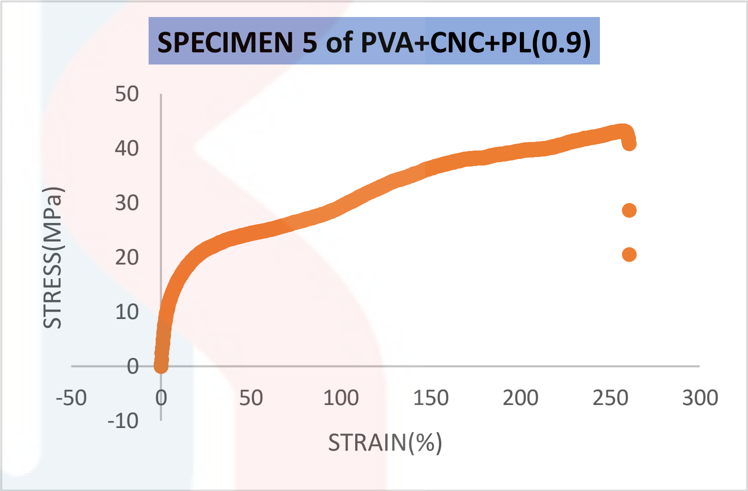


Figure 5.5: Specimen 5 of PVA+CNC+PL (0.9)

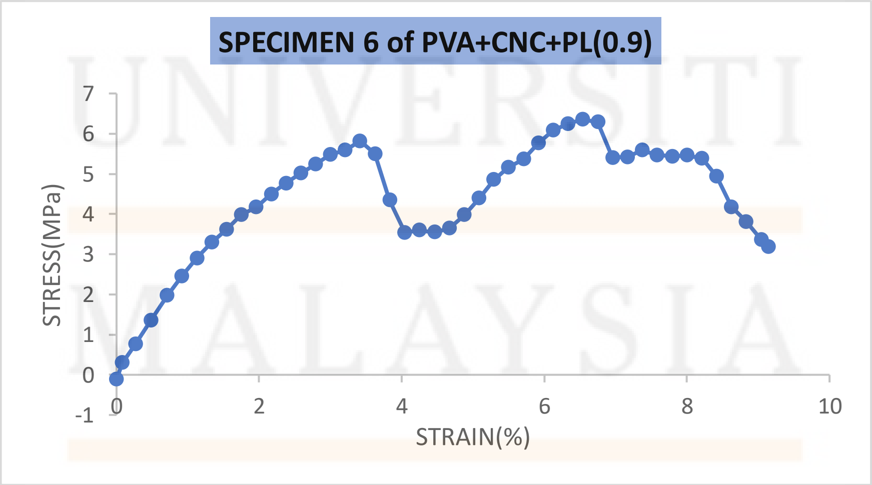


Figure 5.6: Specimen 6 of PVA+CNC+PL (0.9)