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Effect of Cellulose Nanocrystals/E-Polylysine Hybrid Filler on PVA/CNC/Polylysine Nanocomposite for Potential Fresh Food Packaging Application

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J20A0739

A thesis submitted in fulfilment of the requirements for the degree of Bachelor of Applied Science (Materials Technology) with Honours

FACULTY OF BIOENGINEERING AND TECHNOLOGY
UMK

2023

DECLARATION

I declare that this thesis entitled "Effect of Cellulose Nanocrystals/E-Polylysine Hybrid Filler on PVA/ CNC/ ε-Polylysine Nanocomposite for Potential Fresh Food Packaging Application" is the results of my own research except as cited in the references.

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ACKNOWLEDGEMENT

I would like to express my sincere gratitude to the individuals who have played a significant role in the completion of this thesis. Their unconditional support, guidance, and encouragement have been invaluable throughout this academic journey.

First and foremost, I am extremely thankful to my thesis supervisor, Prof. Madya. ChM. Ts. Dr Nor Hakimin Bin Abdullah for his unwavering support throughout this whole journey of my Final Year Project study and research, for his patience, immense knowledge, and constructive feedback. Also, his constant guidance and help that has been provided to me from the beginning of this Final Year Project up till it is completely concluded. I am truly thankful and grateful for the time and effort he has dedicated to mentoring me.

Next, I would like to express my appreciation to the lab assistants of University Malaysia Kelantan for helping and guiding me with lab conducts and characterization machines. I would like to thank Mrs Hanisah and Miss Syahirah for their guidance throughout the Final Year Project research. My sincere gratitude goes out to the rest of my final year project team for working through this challenge together.

Lastly, I would like to express my gratitude to my parents and my friends for their constant encouragement and support throughout this whole journey. I am truly thankful for everyone that has assisted me in finishing my Final Year Project, whether it was personally or indirectly.

Effect of Cellulose Nanocrystals/E-Polylysine Hybrid Filler on PVA/ CNC/ ε-Polylysine Nanocomposite for Potential Fresh Food Packaging Application

ABSTRACT

The purpose of this thesis is to prepare and characterize PVA/CNC/PL nanocomposite films. Nanocomposite film made from Poly (Vinyl Alcohol) (PVA), Cellulose Nanocrystals (CNC), and ε-Polylysine (PL) is beneficial as eco-friendly food packaging. The escalating environmental concerns over non-biodegradable plastics have encouraged the development of biodegradable alternatives. The main material was PVA, which has been chosen for its biocompatibility, film-forming capabilities, and biodegradability. CNC and ε-Polylysine have been added as the additives. CNC provides mechanical strength, barrier characteristics, and biodegradability while ε-Polylysine adds antibacterial qualities. Nanocomposite films with varying percentages of CNC and ε-PL were characterized by various methods such as Visual Inspection, thickness of the thin film, Optical Microscopy, Universal Tensile Machine, FTIR, and XRD. The nanocomposite film was used as food packaging for fresh chillies and the fresh chillies were characterized by using Visual Inspection, Texture Analyzer, pH, and weight loss. Overall, PVA/ CNC/ ε-PL 0.9g showed the best value in extending the shelf life of the fresh chilies over the 15 days of lab conduct. The goal of this study is to provide significant insights into the potential of PVA/CNC/ ε-Polylysine nanocomposite films as eco-friendly food packaging alternatives, addressing environmental issues associated with conventional plastics. The study proposes a long-term solution to reduce plastic waste and extend the shelf life of packaged food goods.

Keywords: Poly (Vinyl Alcohol) (PVA), Cellulose Nanocrystals (CNC), ε-Polylysine (PL), nanocomposite films, food packaging

Kesan Campuran Pengisi Hybrid Nanokristal Selulosa/E-Polilisina ke atas Nanokomposit PVA/CNC/Polilisina untuk Aplikasi Pembungkusan Makanan Segar yang Berpotensi.

ABSTRAK

Tujuan tesis ini adalah untuk menyediakan dan mengkarakteristikkan filem nanokomposit PVA/CNC/PL. Filem nanokomposit yang diperbuat daripada Poli (Vinil Alkohol) (PVA), Kristal Selulosa Nanokomposit (CNC), dan Polilisina (PL) adalah berguna sebagai pembungkusan makanan mesra alam. Kebimbangan alam sekitar yang meningkat berhubung dengan plastik bukan biodegradasi telah memberi dorongan kepada pembangunan alternatif yang boleh terdegradasi. Bahan utama adalah PVA, yang dipilih kerana keserasian biologinya, keupayaan pembentukan filem, dan biodegradabilitinya. CNC dan Polilisina ditambah sebagai aditif. CNC menyediakan kekuatan mekanikal, ciri-ciri penghad, dan kebolehdegradasian sementara Polilisina menambah sifat antibakteria. Filem nanokomposit dengan peratusan CNC dan PL yang berbeza dikarakteristikkan melalui pelbagai kaedah seperti Pemeriksaan Visual, ketebalan filem nanokomposit, Mikroskopi Optik, Mesin Tarik Universal, FTIR, dan XRD. Filem nanokomposit digunakan sebagai pembungkusan makanan untuk cili segar dan cili segar dikarakteristikkan dengan menggunakan Pemeriksaan Visual, Pengukur Tekstur, pH, dan kehilangan berat. Secara keseluruhan, PVA/CNC/ε-PL 0.9g menunjukkan nilai terbaik dalam memanjangkan jangka hayat cili segar selama 15 hari kajian makmal dijalankan.Kajian ini mencadangkan penyelesaian jangka panjang untuk mengurangkan sisa plastik dan memanjangkan jangka hayat barangan makanan yang dibungkus.

Kata Kunci: Poli (Vinil Alkohol) (PVA), Kristal Selulosa Nanokomposit (CNC), Polilisina (PL), filem nanokomposit, pembungkusan makanan

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LIST OF ABBREVIATIONS

PVA	Polyvinyl Alcohol
ε-PL	Epsilon-poly-L-lysine
CNC	Cellulose Nanocrystal
SA	sodium alginate
PL	Polylysine
MCC	Microcrystalline cellulose
LB	Ligno cellulose biomass
PHB	Polyhydroxybutyrate
TPS	thermoplastic styrenic
CS	Carbon steel
TA	thermal analysis
FT-IR	Fourier transform infrared
ATR	Attenuated total reflectance
g	gram
BS	breaking stress
EAB	elongation at break
cm	centimeter
MPa	mega pascal
kV	kilo volt
mL	milliliter
min^{-1}	per minute
cm^{-1}	per centimeter

Min	minute
mA	milliampere
ppm	parts per million
mm	milometer
mJ	millijoule

LIST OF SYMBOLS

°	degree
%	Percentage
λ	Lambda
Ɵ	Theta
σ	Sigma
°C	degree Celsius
±	plus minus

CHAPTER 1

INTRODUCTION

2.1 Background of Study

Polymer-based plastic films are commonly used for food packaging due to their inexpensive cost and long-lasting qualities. However, due to their non-degradability, most of polymeric materials would cause many environmental issues. Due to environmental issues, biodegradable polymers are being invested more as it will help with problems that have been rising ever since decades ago. (J. Li et al., 2022) Therefore, polyvinyl alcohol (PVA) has been selected to be one of the materials due to its remarkable biocompatibility film forming, gas barrier performance and biodegradability.

Polyvinyl alcohol (PVA) is the most in demand water-soluble polymer due to its manufacturing rate of several hundred ton/year worldwide. PVA is frequently a helpful product in the integrated production cycles of the petrochemical industry. PVA is not created via direct polymerization of the suitable monomer because vinyl alcohol spontaneously converts to the enol form of acetaldehyde due to thermodynamic reasons and with minimal kinetic control. Instead, the parent homopolymer poly (vinyl acetate) (PVAc) created PVA. (Chiellini et al., 2003) Polyvinyl alcohol (PVA) has several advantages due to its improved capacity to form the film, artificial water-soluble polymer, chemical resistance, biodegradability, and simplicity of manufacture. (Yuvaraj et al., 2021)

Cellulose nanocrystals (CNCs) are an excellent reinforcer in the biopolymer industry. The primary benefits of CNCs are low density, an extraordinary environmental impact, and simple recycling and processing. Changing their surface hydroxyl groups may easily adjust CNCs' characteristics and uses. The functionalization of covalent and noncovalent frameworks benefits dispersion by enhancing compatibility with the polymer matrix. In recent years, research has been increasing on PVA films reinforced by CNCs, such as CNC-modified biodegradable packaging materials. (Tan et al., 2021)

ε-Polylysine is a homopolymer of the essential amino acid l-lysine. The chemical formula for ε-polylysine is listed below; its systematic name is (S)-poly(imino(2-amino-1-oxo-1,6-hexanediyl). ε-Polylysine differs from other proteins in that the amide linkage is between the -amino and carboxyl groups rather than the alpha-amino and carboxyl groups seen in peptide bonds. Furthermore, ε-polylysine is a polymer with only one amino acid ingredient, unlike the numerous amino acids in proteins. The typical homopolymer molecule of ε-polylysine has the chemical formula C180H362N60O31 with a molecular weight of around 4700. (Hiraki et al., 2003) Due to ε-PL being water soluble, biodegradable, edible, and non-toxic to humans and the environment, it and its derivatives have attracted attention in recent years for various industrial uses, including food, medicine, the environment, and electronics. Food preservatives, emulsifying agents, nutritional agents, biodegradable fibers, highly water-absorbable hydrogels, drug carriers, anticancer agent enhancers, and biochip coatings are just a few of the uses for -PL and its derivatives. (Shih et al., 2006)

For the past decades, plastic pollution has been a major environmental concern with severe consequences on the health of our planet and its inhabitants. Plastics are long-lasting and are incapable of biodegrading, resulting in massive volumes of garbage ending up in landfills, seas, and other ecosystems. Both human life and natural ecosystem damages are caused by human activities. This is shown when plastics, such as plastic bottles and plastic bags, are used for packaging and it is discovered that they are dumped carelessly, with little regard for the consequences. When plastic garbage is not adequately discarded or disposed of, it litters the environment, damaging wildlife, wildlife habitat, and humans, causing choking and emitting a strong scent. As a result, petroleum-based plastic pollutes land, rivers, and seas. (Asiandu P. Angga et al., 2020)

Traditional petroleum-based plastic has been causing pollutions to the environment and to humans. Plastics are both cost-effective and long-lasting. These are why the plastic output is so high, and demand is growing daily. (Asiandu P. Angga et al., 2020). Therefore, PVA plastics are recommended as it has the capability to decay within a short time.

2.2 Problem Statement

Due to the problems caused by petroleum-based plastics, biodegradable plastics are the best solution to solve environmental issues. For example, biodegradable plastics that are made with Poly (Vinyl Alcohol). PVA is a type of biodegradable polymer that is water-soluble and biocompatibility. (Havstad, 2020) One of the most frequently used biodegradable plastics, polyvinyl alcohol (PVA)-starch blends- is used in packaging and as agricultural mulch. PVA is mostly used for food packaging because of its great film-forming ability, biodegradability, non-toxicity, thermoplastic capabilities, outstanding oxygen and aromatic barrier qualities, strong chemical resistance, and good thermal stability. (Sarwar et al., 2018) However, the comparison of PVA and traditional packaging materials, PVA has poor water vapor barrier qualities, low resistance to deformation, and poor water resistance in food packaging applications. Furthermore, PVA lacks important food packaging features such as antibacterial and UV barrier capabilities. (Nguyen & Lee, 2022a)

Therefore, in this research, CNC and ε-Polylysine is added as reinforcement to PVA. CNCs have a rod-like form at the nanoscale (3–20 nm in diameter and 100–1000 nm in length). CNCs have exceptional features like high mechanical strength and Young's modulus, good oxygen barrier qualities, a wide surface area, and great biodegradability. As a result, CNCs are regarded as ideal natural reinforcing fibres for increasing the mechanical characteristics of polymer matrices. Furthermore, CNCs and PVA can form hydrogen bonds. (Nguyen & Lee, 2022b) Additionally, ε-Polylysine is added into the mixture because PL is known for having a wide range of antibacterial action against Gram-positive and -negative bacteria, along with the characteristics of non-toxicity and having a great thermal stability. (Mai et al., 2023)

2.3 Expected Output

The expected output for this research proposal on Preparation and Characterization of PVA/ CNC/ ε-Polylysine Nanocomposites is to solve issues regarding plastic pollution and reduce plastic waste. Besides that, the expected out for this research proposal is to be able to improve the packaging of food and to increase shelf life of the food that is wrapped in the film.

2.4 Objectives

1. To prepare and characterize PVA Nanocomposite film with different content percentages of CNC and ε-Polylysine.
2. To investigate the potential of PVA/ CNC/ ε-Polylysine Nanocomposite film as food packaging for fresh chilies

2.5 Scope of Study

The goal of the research is to prepare and characterize Poly (Vinyl Alcohol)/cellulose nanocrystals/ ε-Polylysine Nanocomposite by observing the shelf life of food as food packaging.

2.6 Significances of Study

Due to the current environmental concerns and advantages of PVA/ CNC/ ε-Polylysine nanocomposite films, it has gained the interests and attention of researchers. PVA/ CNC/ ε-Polylysine nanocomposite film is more beneficial for living things and the environment. For example, PVA/ CNC/ ε-Polylysine nanocomposite film has greater properties than traditional plastic film, such as biodegradability, high tensile strength, biocompatibility, longer shelf life, and it is environmentally friendly. This nanocomposite plastic film will not bring harm to the environment like the traditional plastic film.

CHAPTER 2

LITERATURE REVIEW

3.1 Polymer

Polymeric materials are currently used as a substitute for metals, glass, and ceramics in packaging applications due to their excellent mechanical and barrier qualities, ease of processing, low cost, and widespread availability. Since the mid-twentieth century, synthetic polymers such as polystyrene (PS), polyethylene terephthalate (PET), polyamide (PA), polyvinylchloride (PVC), polypropylene (PP), and polyethylene (PE) have been widely used. (Abdullah et al., 2017.)

3.1.1 Poly (Vinyl Alcohol)

Polyvinyl alcohol (PVA) is an environmentally harmless polymer that may be efficiently combined with starch to substitute non-biodegradable synthetic polymers in packaging materials such as polyolefin and polystyrene. PVA is an excellent choice because of its exceptional features, such as water solubility, favourable optical and tensile properties, excellent chemical resistance, non-toxicity, and biodegradability. (Ismail & Zaaba, 2014)

3.1.2 Polystyrene (PS)

Polystyrene (PS) is an aromatic polymer created by polymerizing styrene monomers. Styrene (vinylbenzene) is derived from ethylene and benzene. Massive PS manufacturing is carried out by catalytic dehydrogenation of ethylbenzene, which usually does not exceed 20 ppm in the polymer sector, far lower than the calculated value of chronic toxicity. (Kik et al., 2020)

3.1.3 Polyethylene Terephthalate (PET)

Polyethylene terephthalate (also known as PET, PETE, or by the resin identification (recycling) code #1) is one of the market's most widely distributed thermoplastic polymers. PET is based on the polyester family, a broad class of polymers distinguished by ester functionalities inside macromolecular leading chains. Aside from PET, the polyesters family includes various oil-derived and naturally occurring polyesters (e.g., the PHA subgroup formed via bacterial-mediated fermentative processes). (Nisticò, 2020)

3.2 Polymer Nanocomposite

By utilizing a more suitable range of fillers, polymer nanocomposites are able to increase the functionality and usability of polymers while maintaining the inherent flexibility in manufacturing and processing that plastics, thermosets, and resins possess. Polymer nanocomposites have proved successful, particularly when overcoming usually antagonistic combinations of characteristics. (Winey. I. Karen & Vaia. A. Richard, 2007)

3.3 Reinforcement/Filler

Plastic composites are intimate combinations of resin and fillers or reinforcements. It defines a filler as a generally inert ingredient added to a plastic to adjust its strength, durability, working characteristics, or other features or to reduce expenses. The reinforced plastic has strength qualities much higher than the basic resin due to high-strength fillers incorporated in the composite. The reinforcing fillers are often fibres, textiles, or mats formed of fibre. Plastic laminates are the most frequent and durable form of reinforced plastic. An example of a polymer nanocomposite is a polymer with graphene as the reinforcement/filler. Carbon black, carbon nanotubes, and layered silicates have been employed in polymer nanocomposites for better mechanical, thermal, electrical, and gas barrier properties of polymers. A new class of polymer nanocomposites has been produced due to the discovery of graphene, which combines remarkable physical qualities with the capacity to disperse in a wide range of polymer matrices. (Kim et al., 2010)

3.3.1 ε-Polylysine

A natural cationic homopolymer comprising 25–30 lysine residues that have an amide linkage connecting the carboxyl and 3-amino groups is known as ε-polylysine (-PL). It is created by *Streptomyces albulus* and is thought to be stable in acidic and alkaline environments. Due to its wide range of antimicrobial activity, it is frequently used as an antimicrobial food additive, and the Food and Drug Administration (FDA) has designated it as a GRAS (generally recognized as safe) preservative. (Wu et al., 2019)

3.3.2 Cellulose

Cellulose is one of Earth's most prevalent natural polymers and an essential part of plant cell walls. It comprises -1,4-linked glucopyranose units that link together to produce a high-molecular-weight linear homopolymer in which each monomer unit is corkscrewed at 180° to its neighbours. Cellulose is a biodegradable, biocompatible, and renewable natural polymer used as an alternative to nondegradable polymers derived from fossil fuels. It comprises -1,4-linked glucopyranose units that link together to produce a high-molecular-weight linear homopolymer in which each monomer unit is corkscrewed at 180° to its neighbours. (George & Sabapathi, 2015)

3.3.3 Cellulose Nanocrystals

Colloidal cellulose suspensions were described in the 1950s because of controlled sulfuric acid-catalyzed breakdown. As a result, microcrystalline cellulose (MCC) was commercialized. Colloidal solutions of cellulose nanocrystals were shown to display nematic liquid crystalline alignment. (Habibi et al., 2010) With the advancement of nanotechnology, nanocellulose extracted from LB has piqued the interest of researchers all over the world due to its low cost, biocompatibility, renewable nature, exceptional reactivity, and ideal physical properties such as lightweight, high stiffness, tensile strength, strength to weight ratio, optical transparency, and low expansion on heating. (Rana et al., 2021)

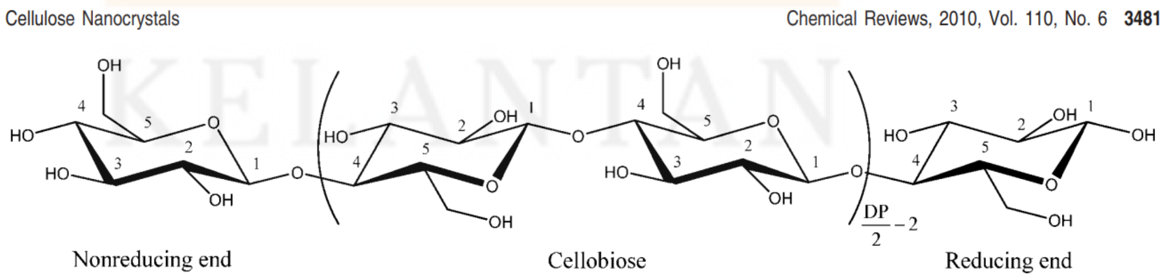

Figure 3.3.1: Chemical structure of cellulose.

(Habibi et al., 2010)

3.4 Solvent Casting

Solvent casting is the most convenient method to prepare a thin film. In the solvent casting method, the polymer is dissolved in a suitable solvent, and the solvent is evaporated to produce a high-quality film. When manufacturing variables such as solvent casting time and temperature are changed, the mechanical and optical characteristics of the film can be adjusted. The solvent casting method may create porous films with excellent optical clarity that degrade quickly under physiological settings and ultra-thin films with excellent optical clarity. Despite these benefits, solvent casting is only used in a few applications, such as cellulose triacetate films for photographic sheets and polyvinyl alcohol films for liquid crystal display polarizers. The cost of solvent casting can be reduced, allowing for manufacturing of high-quality PHB films using this method, providing a relatively safe and affordable solvent. (Anbukarasu et al., 2015)

Polymers and Typical Solvents for Casting Processes			
Cellulose Nitrate	Ether, Esters	Polyimides	Dimethylformamide
Cellulose Diacetate	Acetone, Methanol		
Cellulose Triacetate (TAC)	Methylene Chloride, Alcohols	Polyvinylalcohol	Water, Methanol
		Methyl Cellulose	Water
		Starch derivatives	Water
Polycarbonates	Methylene Chloride	Gelatine	Water, Methanol
Polyethersulfon	Methylene Chloride		
Polyetherimide	Methylene Chloride		
Polyvinylidene fluoride	Acetone		
PVC (Polyvinyl chloride)	Tetrahydrofuran		
	Methyl-ethylketon		

Table 1: Polymers and Typical Solvents for Casting Processes

(Siemann, 2005)

3.5 Characterization Techniques of Polymer Nanocomposite

The PVA/CNC/Polylysine Nanocomposite films will be characterized with multiple techniques to determine the potential of the film as a food packaging application.

3.5.1 X-ray diffraction (XRD)

X-ray diffraction was used to examine the crystallization of neat TPS and TPS/MCC biocomposites, along with the native components, using a Siemens D5000 diffractometer (Germany). The apparatus was run at a current of 30 mA, a voltage of 40 kV, and a wavelength of λ (Kα) = 1,54 Å for the cooper anode. At a scanning step rate of 0.05 and a step time of 4s, scans were conducted in the 2ϴ = 5° to 40° range. The crystallinity degree has been calculated as the ratio of the crystalline area under the diffraction peaks to the entire scope of the diffractogram. The amorphous area was calculated via an iterative smoothing curve. (Area et al., 2019)

3.5.2 Fourier-Transform Infrared Spectroscopy (FTIR-ATR)

The FT-IR spectrometer (type Alpha, Bruker Optik GmbH, Ettlingen, Germany) was used to record the infrared spectra of the various films in the ATR mode. Spectra were acquired in the 4000-400cm^{-1} wavenumber range from two separate sites on the top and bottom sides of the same samples by aggregating 64 images with a spectral resolution of 4 cm^{-1}. (Haghighi et al., 2021)

3.5.3 Tensile strength

An electronic universal testing instrument will be used to test the film's tensile strength and elongation at the breaking point. By using the equation: $\sigma_t = \frac{p}{b \times d}$ where σ_t is the tensile strength (MPa), p is the load at fracture (N), b is the sample width (mm), and d is the sample thickness (mm), the tensile strength was computed. (Chen et al., 2015)

3.6 Application of Polymer Nanocomposites as Fresh Chillies Packaging

The primary purposes of food packaging are to maintain the quality and to make sure that food products are safe to be consumed from the beginning point of production to the end of consumption and to increase the shelf life of the packaged food materials by warding off unfavourable changes that have been brought on by microbial spoilage, chemical contaminants, temperature change, oxygen, moisture, light, and external force. Packaging accomplishes the tasks above by establishing the right physicochemical conditions for products and acting as a barrier against gases, water vapor, light, and microbes. This extends packaged food products' shelf life while maintaining food quality and safety. The material used to package food must possess several other qualities besides the more fundamental ones like mechanical, optical, and thermal properties. These qualities include inhibiting moisture gain or loss, acting as a barrier against water vapor permeability, oxygen, carbon dioxide, and other volatile compounds like flavours, and preventing microbial growth and contamination. Food packaging serves as more than just a container for food; it also acts as a protective barrier with several sophisticated functions. The need for novel packaging materials to satisfy consumer demand for higher-quality food with safety, convenience, and sustainability is constantly growing. (Shankar & Rhim, 2016)

3.6.1 Preparation of Fresh Chilies

Peeling, cutting, washing, sanitizing agent treatments, drying, and other minimum processing processes are commonly used in mild technology. These items' physical integrity is altered during processing, which makes them less resistant to deteriorating than the original raw ingredients. Fresh produce damaged during processing processes is more susceptible to contamination due to the growth or survival of germs that cause deterioration or disease. (Abdul Khalil et al., 2018) The effect of PVA/ CNC/ ε-Polylysine film wrapping the fresh chilies for preservation will be tested. There will be three groups of samples: the control group, fresh chillies wrapped in petroleum-based film, and fresh chillies wrapped in PVA/ CNC/ ε-Polylysine film. The samples will be thermally sealed after being wrapped in the films. Analysis of the fresh chillies' sensory and quality changes will be done during the storage at 26 ± 1 ◦C, 37 ± 1 % RH.(Xiang et al., 2021)

3.7 Quality Assessment for Fresh Chillies

The wrapped fresh chillies will be analysed through a few testing for quality, freshness, taste, and texture.

3.7.1 Texture Analyzer

Texture analyzer is to analyze a food's surface. There are many different foods with drastically varying textural properties, and other people may have other definitions or expectations for certain types of food, which contributes to the difficulty in defining food texture. It is crucial to employ objective, standard methodologies to measure the textural aspects of food because many terminologies have been used to determine the characteristics of the surface. A food's mechanical and structural characteristics are directly related to texture, a qualitative trait. Understanding food's mechanical qualities is crucial for understanding its textural properties and procedures for measurement. Food rheology is a large field of study that includes both solid and liquid foods, and it focuses on the mechanical behaviour, or deformation and flow, of meals under applied stresses. (Lu, 2013)

CHAPTER 3

MATERIALS AND METHODS

4.1 Introduction

In this part of the research proposal, materials and methods is explained in detail to conduct the experiment. The method for preparation and characterization of Poly (Vinyl Alcohol)/ Cellulose Nanocrystals/ ε-Polylysine Nanocomposite for Food Packaging Applications will be detailly written.

4.2 Research Flowchart

The research flowchart shows the preparation of sample, and characterization steps as shown in **Figure 2**.

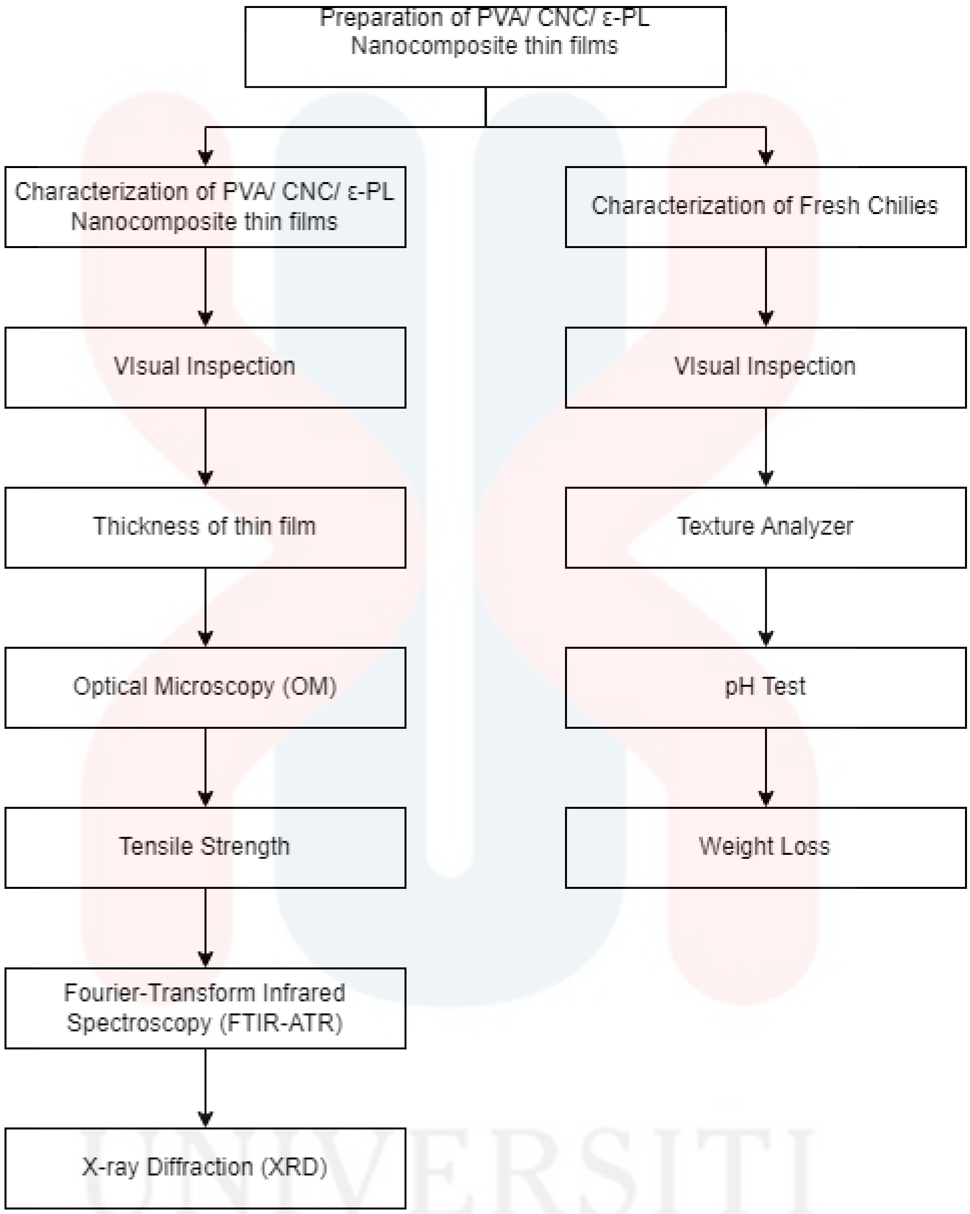

Figure 4.2.1: Research Flowchart

4.3 Raw Materials

The selected raw materials for PVA, PVA/ CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL0.6g, and PVA/ CNC/ ε-PL 0.9g nanocomposite films are powdered polyvinyl alcohol, powdered cellulose nanocrystals, and powdered ε-polylysine. These raw materials were weighed and mixed. Besides that, deionized water and distilled water was used in this experiment. These raw materials were weighed and mixed accordingly to their stochiometric ratio. The amount of each raw material was determined. The raw materials are solvent casted and left to dry on petri dishes at room temperature after preparation. The polyvinyl alcohol (PVA) was supplied by Shanghai Yuanye Bio-Technology Co., Ltd (China), which has a degree of polymerization of approximately 2500. The ε-polylysine was provided by the Silver-Elephant Bio-engineering Company, Ltd. (Zhejiang, China), which has a molecular weight of approximately 6Dka.

4.4 Sample Preparation

4.4.1 Preparation of PVA/ CNC/ ε-Polylysine Nanocomposite film

The powdered form of polyvinyl alcohol is provided by R&M Chemicals. The cellulose nanocrystals used for this research are manufactured in Canada by Celluforce and, they were in powdered form. The raw materials were weighed approximately 5g and, they were maintained in a sample container at room temperature to allow the raw materials to characterize. 5 g of PVA was be dissolved in water at the rate of 5 percent by weight for an hour at 90 degrees Celsius. The PVA/CNC nanocomposite films were made by mixing a predetermined quantity of CNC dispersion with a predetermined quantity of PVA solution, then, the mixture was homogenized for 45 minutes. Next, the homogenous mixture was poured into a petri dish, and it was dried at room temperature for 7 days. The PVA/ CNC/ ε-PL nanocomposite films were made by adding a specific amount of CNC dispersion to the PVA solution, and the mixture was homogenized for 45 minutes. After that, an amount of 0.3g, 0.6g and 0.9g ε-PL was added to the mixture and heated to 50 degrees Celsius while it was stirred continuously for an hour. Lastly, the liquid was divided into several Petri dishes, and it was left to dry at room temperature for 7 days. After the films are dried, they were given a final rinsing in deionized water before it was dried at 40 degrees Celsius for five hours.

4.4.2 Solution Casting Process

The PVA, CNC, and ε-Polylysine samples were cast in glass Petri dishes, and they were dried at room temperature for 7 days. The nanocomposite thin films with different amounts of ε-PL content, 0.3wt%, 0.6wt% and 0.9wt%, were produced. Then, the nanocomposite thin film was stored in a zipper bag for characterization.

4.5 Characterization of Thin Film

4.5.1 Transparency Test

To be able to determine the transparency of an object, a transparency test was performed. The term "transparency" refers to an object that can be perceived through things such as glass, plastic film, or sheet, and others. Therefore, a transparency test was performed on the nanocomposite thin film to find out the level of transparency of the thin film. The location of the transparency test was in the lightroom. The thickness of the thin films was tested and observed.

4.5.2 Thickness of the thin films

A thin film can be measured for thickness using a variety of techniques; the technique will be chosen depending on the material and purpose of the film. One of the various methods that are frequently used to measure the thickness of thin coatings is optical microscopy. This method uses the transmission or reflection of light as the determining factor to calculate the thickness of a thin layer. A microscope with a micrometer scale was used to measure the distance between the substrate of the film and its top surface. Three different places were used to measure the film's thickness by using a calibrated digital gauge. The average of three readings was calculated.

4.5.3 Universal Tensile Machines

By using a Universal Tensile Machine model M500-50CT, the nanocomposite thin film sheets' breaking stress (BS) and elongation at break (EAB) was measured and analyzed. The thin film sample was carried out by moving along a central longitudinal axis at a consistent extension rate during the tensile test until a target stress or strain value is reached. This process was repeated until the value is attained. The films were sliced into rectangles of 40mm by 13mm, and the examination was carried out using a gauge length of 40mm at a crosshead speed of 1mm per minute. The average of three separate samples of each specimen was calculated.

4.5.4 Light Optical Microscope

Light Optical Microscope was used to observe the samples' birefringence features. LOM was used to observe the small structures by magnifying the image of the sample with visible light. Absorption, reflection and, scattering of the sample were observed. The thin film was captured with 10x magnification and 10x polarized magnification.

4.5.5 Fourier-Transform Infrared Spectroscopy (FTIR-ATR)

FTIR Spectroscopy spectrometer (type Alpha, Bruker Optik GmbH, Ettlingen, Germany) generates spectra of patterns that will provide information about the object's structure and modifies the data generated by the detector to a range that can be understood. The FTIR spectra of the films were recorded by using an attenuated total reflectance-Fourier transform infrared (FTIR-ATR) spectrophotometer. These spectra were ranged from 4000 cm-1 to 500 cm-1. It is frequently used in petrochemical production, organic synthesis, material technology, the medical industry, and the food industry. In addition, it is feasible to study the mechanisms underlying chemical reactions and the detection of radioactive chemicals by combining FTIR spectrometers with chromatography. This approach will examine the structural alteration in the extracted cellulose powder.

4.6 X-ray Diffraction (XRD)

The phase structure of PVA, PVA/ CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, and PVA/ CNC/ ε-PL 0.9g nanocomposite film is determined via X-Ray Diffraction (XRD). The XRD method was based on the constructive interference of monochromatic X-rays with crystalline samples. After generating, X-rays are collimated, filtered to produce monochromatic radiation, and directed towards the material for examination. The sample interacts with incoming rays, resulting in constructive interference and a diffracted beam. This occurs when the sample interacts with the rays. Converting diffraction peaks to d-spacing helps identify compounds, as each has a unique d-spacing.

4.7 Chilies Packaging Test

The fresh chilies for this research will be tested by physiological loss in weight. The fresh chilies for this research were cleaned using cold water with a combination of 100ppm chlorine to reduce the respiration rate and microbial content. Before packing, the fresh chilies will be visually screened to avoid defective samples.

4.7.1 Visual Inspection of Fresh Chilies

The picture of the fresh chilies wrapped in PVA, PVA/ CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, and PVA/ CNC/ ε-PL 0.9g nanocomposite film was taken to observe the color, the condition, and the changes of the fresh chilies. The picture of the fresh chilies taken on Day 15 was compared to the picture of the fresh chilies taken on Day 1.

4.7.2 Texture Analysis

The texture of the fresh chilies wrapped in PVA, PVA/ CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, and PVA/ CNC/ ε-PL 0.9g nanocomposite film were tested using CT3 Texture Analyzer, Brookfield. The Hardness (g), the Adhesiveness (mJ), the Fracturability (g), the Cohesiveness, the Springiness (mm), and the Gumminess (g) of the fresh chilies were tested.

4.7.3 pH Test

The pH level of the fresh chilies wrapped in PVA, PVA/CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, and PVA/ CNC/ ε-PL 0.9g nanocomposite film were tested using TQC Sheen pH meter pHTestr 30.

4.7.4 Weight Loss

The weight loss of the fresh chilies wrapped in PVA, PVA/CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, and PVA/ CNC/ ε-PL 0.9g nanocomposite film was measured by a weighing balance on Day 0, Day 5, Day 10 and Day 15.

CHAPTER 4

RESULT AND DISCUSSION

5.1 Visual Inspection

The transparency of the nanocomposite film was observed macroscopically.

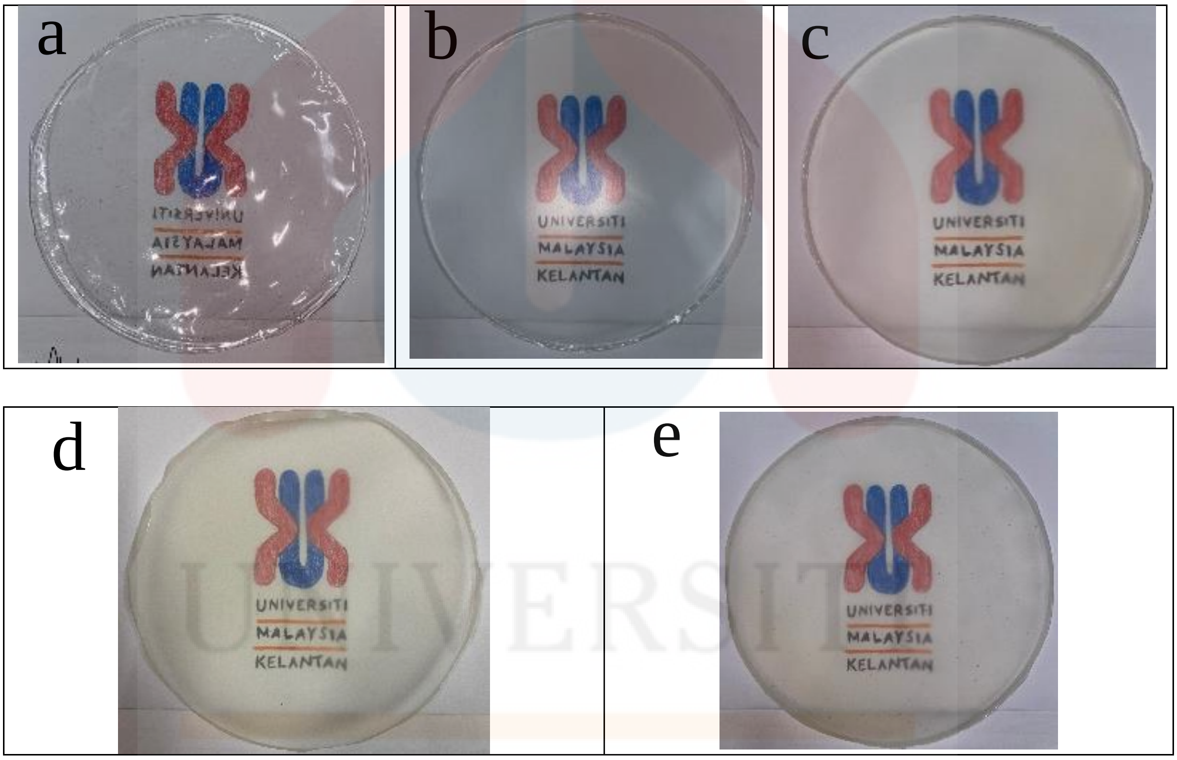

Figure 5.1.1: Visual Inspection images of nanocomposite films (a) PVA; (b) PVA/ CNC; (c) PVA/ CNC/ ε-PL 0.3; (d) PVA/ CNC/ ε-PL 0.6; (e) PVA/ CNC/ ε-PL 0.9

As shown in Figure 4.1.1, image (a) is PVA nanocomposite film, (b) is PVA/ CNC nanocomposite film, (c) PVA/ CNC/ ε-PL is 0.3wt.%, (d) is PVA/ CNC/ ε-PL 0.6 wt.%, (e) is PVA/ CNC/ ε-PL 0.9 wt.%. (a) Polyvinyl alcohol (PVA) film shows a

100% transparency while (b) PVA with 5 wt.% of cellulose nanocomposite (CNC) is slightly translucent. As for (c) PVA/ CNC/ ε-PL 0.3 wt.%, (d) PVA/ CNC/ ε-PL 0.6 wt.%, (e) PVA/ CNC/ ε-PL 0.9 wt.% is translucent. The colour of nanocomposite film that contains ε-polylysine came out to be slightly yellow.

5.2 Thickness of the thin films

Table 5.2.1: The thickness of the thin films

	PVA	PVA/ CNC	PVA/ CNC/ ε-PL 0.3g	PVA/ CNC/ ε-PL 0.6g	PVA/ CNC/ ε-PL 0.9g
Thickness (mm)	0.09	0.15	0.13	0.16	0.16

Based on Table 4.2.1, the thickness of the thin films are 0.09mm for PVA nanocomposite film, 0.15mm for PVA/ CNC film, 0.13mm for PVA/ CNC/ ε-PL 0.3g, 0.16mm FOR PVA/ CNC/ ε-PL 0.6g and 0.16mm for PVA/ CNC/ ε-PL 0.9g.

5.3 Optical Microscopy

The nanocomposite films were characterized by Optical Microscopy, MT8000, Meiji Techno.

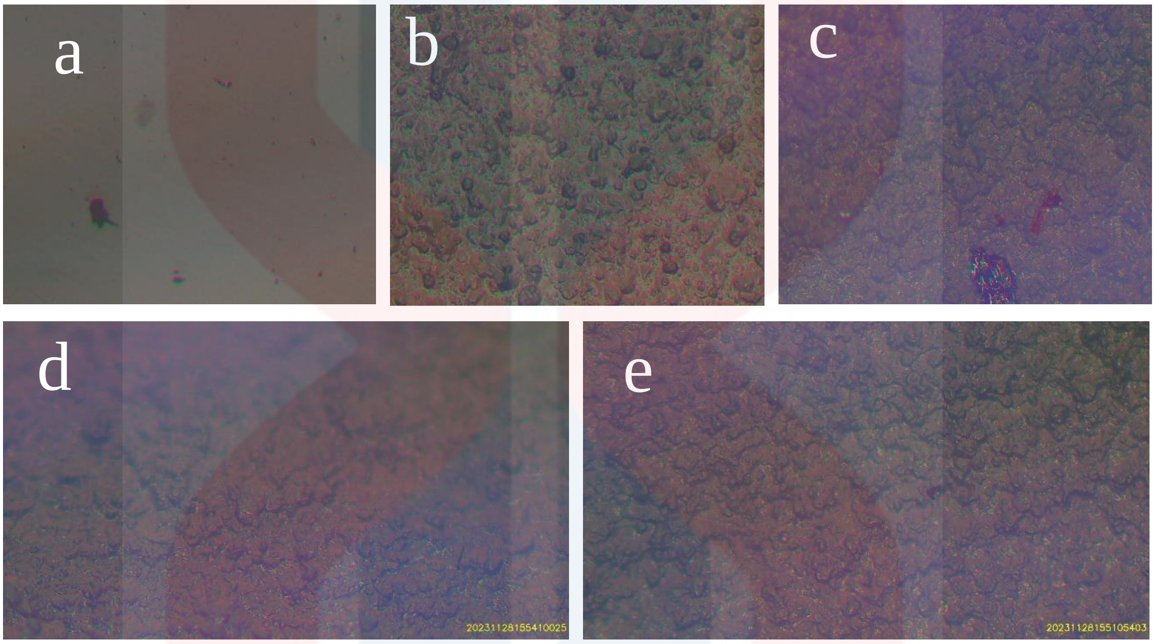

Figure 5.3.1: OM images of nanocomposite film (a) PVA; (b) PVA/ CNC; (c) PVA/ CNC/ ε-PL 0.3g; (d) PVA/ CNC/ ε-PL 0.6g; (e) PVA/ CNC/ ε-PL 0.9g

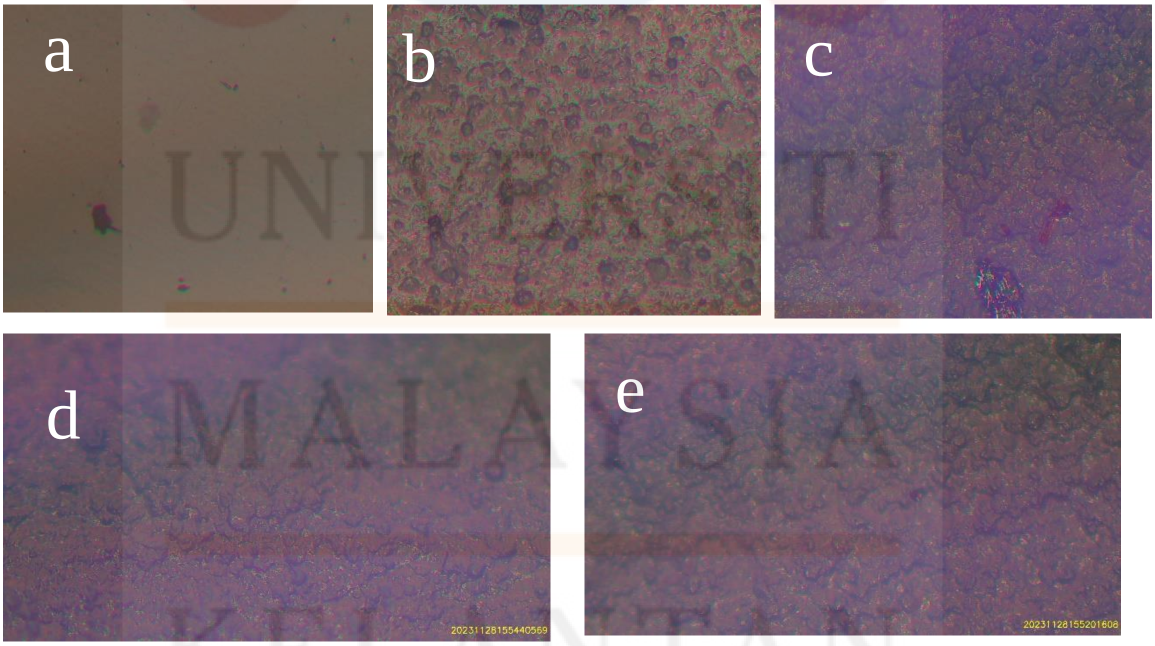

Figure 5.3.2: OM polarized images of nanocomposite film (a) PVA; (b) PVA/ CNC; (c) PVA/ CNC/ ε-PL 0.3g; (d) PVA/ CNC/ ε-PL 0.6g; (e) PVA/ CNC/ ε-PL 0.9g

Figure 4.2.1 shows the images of PVA, PVA/ CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, and PVA/ CNC/ ε-PL0.9g nanocomposite films under an optical microscopy at 10x magnification. While Figure 4.2.2 shows the polarized images of PVA, PVA/ CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL0.6g, and PVA/ CNC/ ε-PL 0.9g nanocomposite films under an optical microscopy at 10x magnification. There were no differences found between the non-polarized and the polarized images of the thin films as it failed to achieve the intention of using OM on the thin films.

The sole PVA film showed a smooth surface under optical microscopy while the PVA nanocomposite film that incorporated 5wt% of CNC showed clumps on the surface. Besides that, nanocomposite film (c), (d), and (e) all showed the same clumpy texture on the surface under optical microscopy. This is caused by an increased content of CNC that has resulted in porosity in polymer matrix of the CNC/ PVA/ ε-PL nanocomposite. Due to the limited interfacial adhesion between both components, voids and clumping between the nanocomposite's matrix occurred. (Ulaganathan et al., 2022)

5.4 Tensile Strength

Each sample is ASTM D638 Type V, with thickness of 20mm. All samples have the same settings for temperature (21.7 degree C), humidity (50.2%), and rate (50.0mm/min).

Table 5.4.1: Young Modulus (MPa), Stress at Break (MPa) and Elongation at Break (%) of the 5 Samples.

Sample	Young Modulus (MPa)	Stress at Break (MPa)	Elongation at Break (%)
PVA	303.86 ± 58.75	41.62 ± 6.31	191.70 ± 13.64
PVA/ CNC	3175.29 ± 391.15	30.67 ± 3.19	25.68 ± 3.14
PVA/ CNC/ ε-PL 0.3g	1812.36 ± 69.69	28.91 ± 1.27	36.12 ± 2.68
PVA/ CNC/ ε-PL 0.6g	1599.35 ± 61.26	27.74 ± 0.49	44.86 ± 4.86
PVA/ CNC/ ε-PL 0.9g	1488.75 ± 103.50	25.03 ± 1.17	42.15 ± 6.36

Toughness is defined as the area under the stress-strain curve, whereas tensile strength and strain at break represent the highest stress and strain the films could bear before breaking. Each sample's data points are an average of measurements from 5-8 specimens. Table 4.3.1 shows the Young Modulus (MPa), Stress at Break (MPa) and the Elongation of Break (%) for all five samples of PVA, PVA/ CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g and PVA/ CNC/ ε-PL 0.9g.

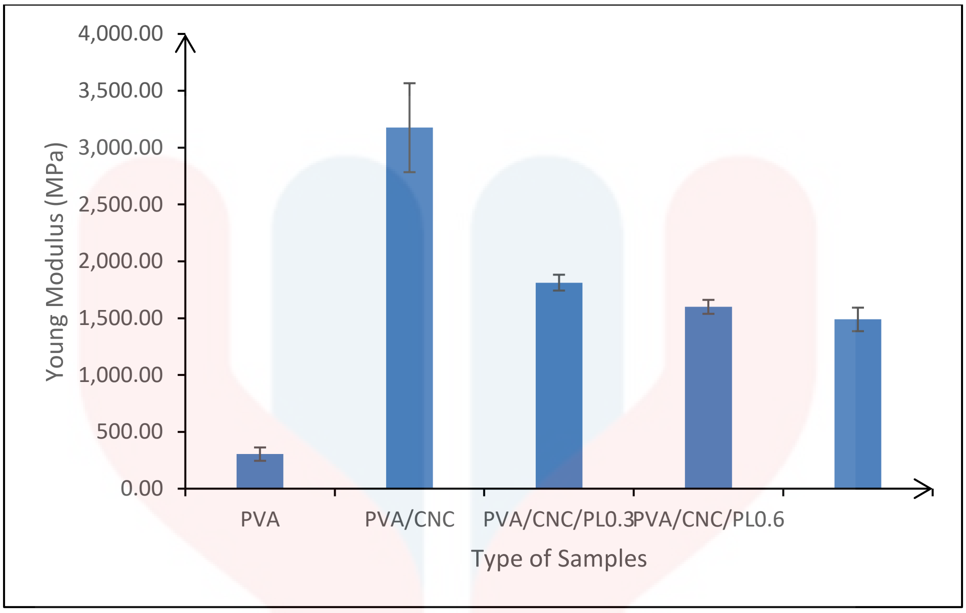

Figure 5.4.1: Young Modulus (MPa) of sample PVA, PVA/ CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, PVA/ CNC/ ε-PL 0.9g

Based on Figure 4.3.1, PVA nanocomposite film shows the lowest tensile strength compared to the other films with 303.86 (± 58.75) MPa. The nanocomposite film with 5 wt%. of CNC added into the PVA solution, it had increased the tensile strength of the film by almost 100% from 303.86 (± 58.75) MPa to 3175.29 (± 391.15) MPa With Polylysine added into the mixture, the graph for tensile strength of the nanocomposite film shows a chart that gradually dropped. It had dropped to 1812.36 (± 69.69) MPa for PVA/ CNC/ ε-PL 0.3g sample, 1599.35 (± 61.26) MPa for the sample PVA/ CNC/ ε-PL 0.6g and followed by 1488.75 (± 103.50) MPa for the last sample of PVA/ CNC/ ε-PL 0.9g.

Tensile strength may have increased due to the creation of hydrogen bonds between CNC and PVA, which increased their contact. This eventually resulted in a limitation of motion and increased stiffness, lowering the elongation at break. (Pavalaydon et al., 2022)

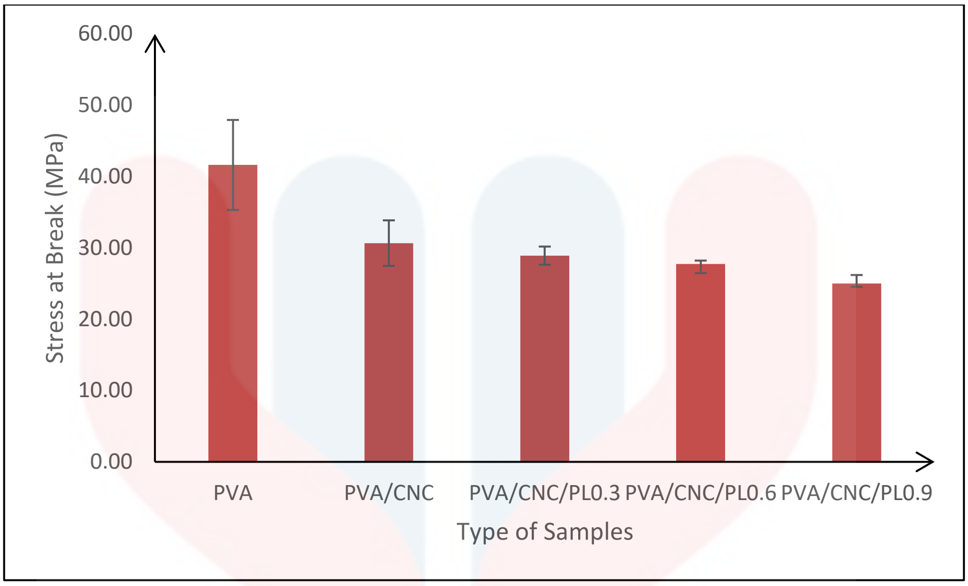

Figure 5.4.2: Stress at Break (MPa) of sample PVA, PVA/ CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, PVA/ CNC/ ε-PL0.9g

As for the stress at break (MPa), Figure 4.3.2 shows that PVA nanocomposite film has the highest stress at break which is 41.62 (± 6.31) MPa. The graph shows a slight drop as the stress at break for PVA/CNC nanocomposite film is 30.67 (± 3.19) MPa. The graph shows a continuous drop as the samples incorporated ε-Polylysine. Starting with PVA/ CNC/ ε-PL 0.3g with stress at break of 28.91 (± 1.27) MPa, PVA/ CNC/ ε-PL 0.6g with 27.74 (± 0.49) MPa and 25.03 (± 1.17) MPa for the last sample with the highest amount of Polylysine but lowest stress at break which is PVA/ CNC/ ε-PL 0.9g.

PVA are known to have high flexibility and it was proved in the graph showing that it has the highest stress at break. This is because of the strong hydrogen bonding of the PVA chains' intermolecular contact. But with the addition of CNC and PL, it increases the crystallinity index therefore, it lowers the mobility of the PVA chains. (Abral et al., 2020)

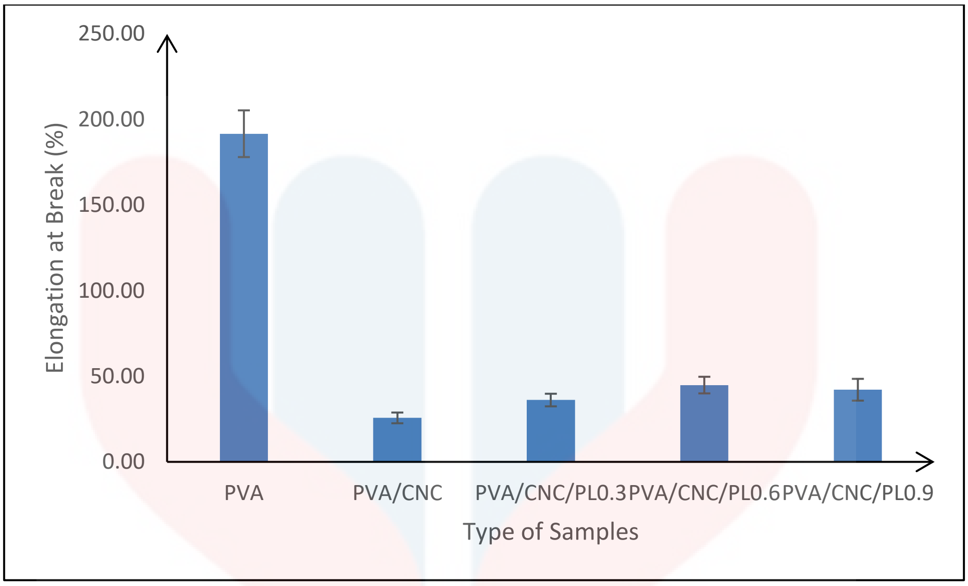

Figure 5.4.3: Elongation of Break (%) of sample PVA, PVA/ CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, PVA/ CNC/ ε-PL 0.9g

Based on Figure 4.3.3 shown above, PVA nanocomposite film has the highest elongation of break (%) at 191.70 (± 13.64) %. With CNC incorporated in PVA, the elongation of break for PVA/ CNC is the lowest out of all samples with the amount of 25.68 (± 3.14) %. The elongation of break for PVA/ CNC/ ε-PL 0.3g is slightly higher compared to PVA/ CNC nanocomposite film at 36.12 (± 2.68) %. PVA/ CNC/ ε-PL 0.6g shows an increase on the graph with 44.86 (± 4.86) % for elongation at break. Lastly, for PVA/ CNC/ ε-PL 0.9g shows a slight decline after PVA/ CNC/ PL 0.6g on the graph with 42.15 (± 6.36) % for the elongation of break.

With CNC and PVA present in the sample, it increases the hydrogen bond of them. This causes the sample to be more brittle as it has limited motion and an increased stiffness which decreases the elongation at break. (Pavalaydon et al., 2022)

5.5 Fourier-transform Infrared Spectroscopy (FTIR)

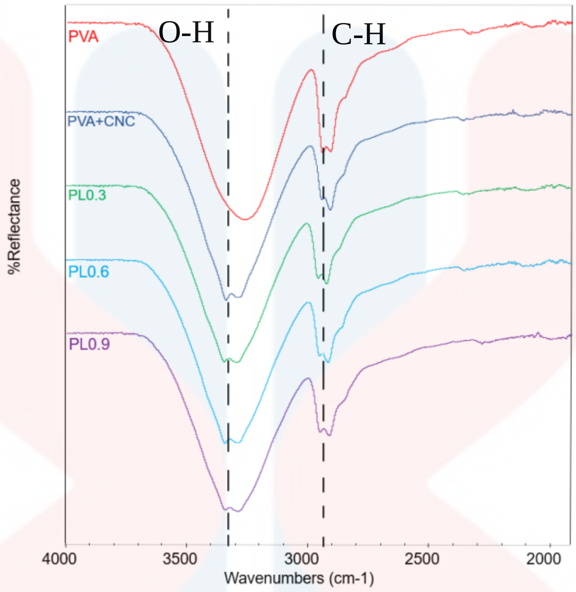

Figure 5.5.1: FTIR results of PVA, PVA/ CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, PVA/ CNC/ ε-PL 0.9g sample.

The interactions of PVA, CNC and ε-PL were studied by using FTIR-ATR. Figure 4.4.1 shows a graph of FTIR results for PVA, PVA/ CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, and PVA/ CNC/ ε-PL 0.9g sample. The nanocomposite film was successfully prepared.

The peak of PVA at 3258.74cm-1 indicates the stretching and deformation vibration of the O-H groups for pure PVA. On the second peak of PVA at approximately 2900cm-1 shows the stretching, deformation and rocking vibration of C-H bonds. (Soliman et al., 2021)

The graph of PVA/ CNC at 3332.57cm-1, PVA/ CNC/ ε-PL 0.3g at 3280.82cm-1, PVA/ CNC/ ε-PL 0.6g at 3322.95cm-1, and PVA/ CNC/ ε-PL 0.9g at 3276.46cm-1 shows characteristics bands for stretching and in-plane deformation of O-H groups. On the second peak at around 2900cm-1, it shows the stretching, deformation and rocking vibration of C-H groups like pure PVA sample. (Popescu, 2017)

5.6 X-ray Diffraction (XRD)

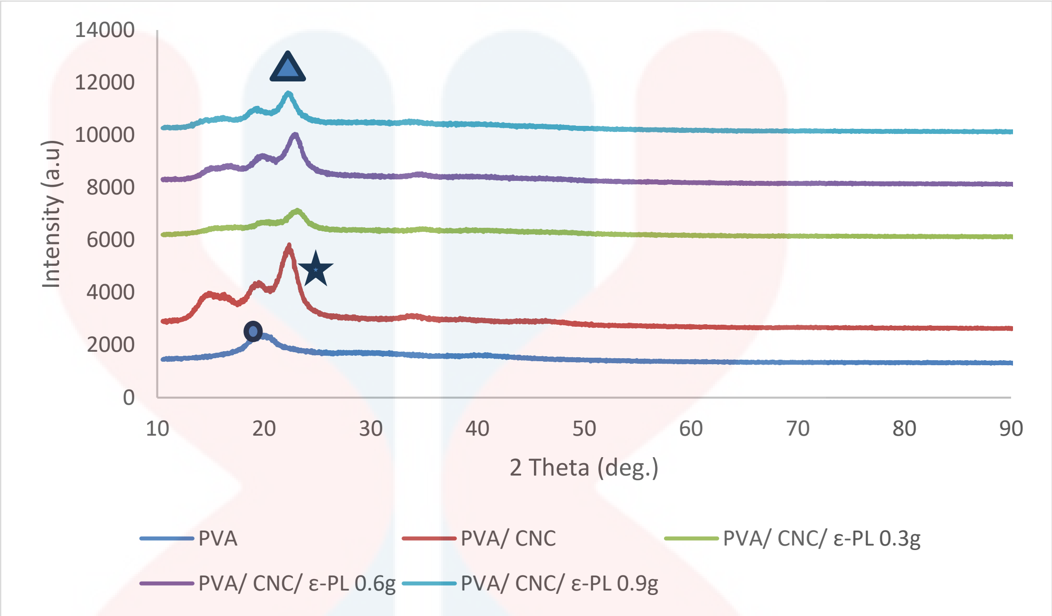

Figure 5.6.1: XRD results of PVA, PVA/ CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, and PVA/ CNC/ ε-PL 0.9g nanocomposite film.

As Figure 4.6.1 shown, a circle symbol on the graph shows the peak found in PVA nanocomposite film are boarder indicating that it is amorphous. The peaks represent possibility of residual crystallinity or imperfections in the sample. The amorphous peak of PVA might be caused by polymer chains form strong intramolecular and intermolecular connections. (Deghiedy & El-Sayed, 2020). The star on the graph represents the crystalline peak of CNC. The triangle symbol on the graph shows a semicrystalline peak for PVA/ CNC in the nanocomposite film. Naturally, Cellulose is a natural occurring crystalline polymer. Cellulose nanocrystals (CNC) are made from cellulose and can show discrete diffraction peaks corresponding to the crystalline cellulose structure. As a result, adding CNC to PVA matched its semi-crystalline character. (Ejara et al., 2021). By adding ε-PL into the PVA/CNC nanocomposite film, it reduces the intensity of the peaks as observed in Figure 4.6.1. (Yurong & Dapeng, 2020).

5.7 Visual Inspection of Fresh Chilies

The fresh chilies were observed macroscopically on Day 1 and Day 15.

	Day 1	Day 15
PVA		
PVA/CNC		
PVA/ CNC/ ε-PL 0.3g		

PVA/ CNC/ ε-PL 0.6g		
PVA/ CNC/ ε-PL 0.9g		

Figure 5.7.1: Visual Inspection of Fresh Chilies on Day 1 and Day 15.

The red chilies were observed on Day 1 and Day 15 as shown in Figure 4.5.1. The colour of the red chilies on the first day was bright red and the colour of the red chilies turned darker on the fifteenth day. The head of the red chilies has grown mouldy over the 15 days and some of the chilies has black spots on them.

5.8 Texture Analysis

Table 5.8.1: Hardness (g) of Fresh Chilies on Day 1, and Control sample, PVA, PVA/ CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, and PVA/ CNC/ ε-PL 0.9g nanocomposite film on Day 15.

Hardness (g)	Day 1	Control	PVA	PVA/ CNC	PVA/ CNC/ ε-PL 0.3g	PVA/ CNC/ ε-PL 0.6g	PVA/ CNC/ ε-PL 0.9g
Mean	1306.67	945.33	734	1238	1123.33	1266.33	1279
Standard Deviation	308.2	200.85	80.62	217.37	90.94	280.39	238.79

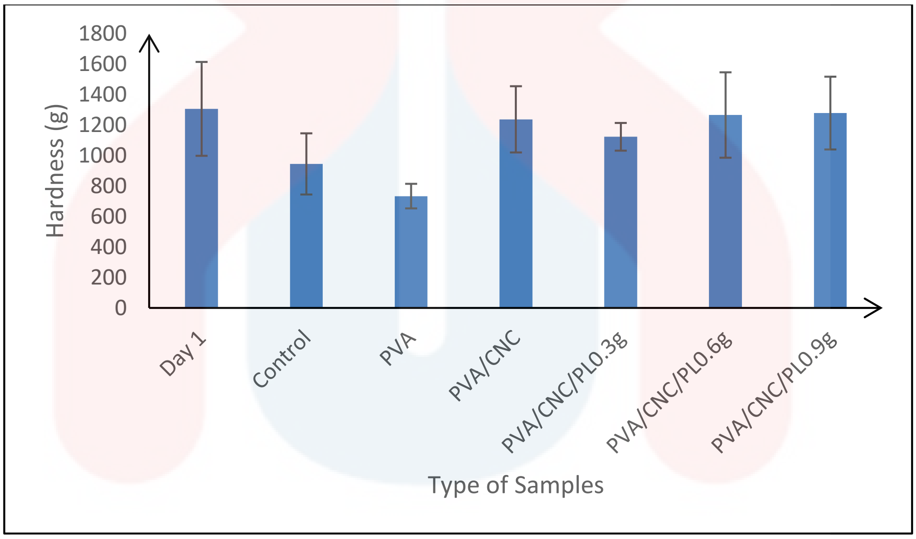

Figure 5.8.1: Hardness (g) of Fresh Chilies on Day 1, and Control sample, PVA, PVA/ CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, and PVA/ CNC/ ε-PL 0.9g nanocomposite film on Day 15.

Based on Table 4.6.1 and Figure 4.6.1, the data of the hardness of the fresh chilies on Day 1 was 1306.67g (± 308.20g). On the last day of wrapping, which is Day 15, the hardness of the fresh chilies for Control sample was 945.33g (± 200.85g). The fresh chilies wrapped in PVA nanocomposite film were tested 734g (± 80.62g) and the fresh chilies wrapped in PVA/CNC nanocomposite film were 1238g (± 217.38g). Next, the fresh chilies wrapped in samples that are added with ε-Polylysine, fresh chilies in

PVA/ CNC/ ε-PL 0.3g nanocomposite film with the hardness of 1123.33g (± 90.94g), PVA/ CNC/ ε-PL 0.6g nanocomposite film with 1266.33g (± 280.39g), and lastly, the hardness of the fresh chilies wrapped in PVA/ CNC/ ε-PL 0.9g nanocomposite film was tested 1279g (± 238.79g).

The highest amount of hardness of the fresh chilies are the fresh chilies wrapped in PVA/ CNC/ ε-PL 0.9g nanocomposite film with 1279g (± 238.79g) while the lowest amount of hardness of the fresh chilies are 734g (± 80.62g) which is the fresh chilies wrapped in PVA nanocomposite film as shown in Figure 4.6.1.

Table 5.8.2: Adhesiveness (mJ) of Fresh Chilies on Day 1, and Control sample, PVA, PVA/ CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, and PVA/ CNC/ ε-PL 0.9g nanocomposite film on Day 15.

Adhesiveness (mJ)	Day 1	Control	PVA	PVA/ CNC	PVA/ CNC/ ε-PL 0.3g	PVA/ CNC/ ε-PL 0.6g	PVA/ CNC/ ε-PL 0.9g
Mean	1.27	0.13	0.23	0.63	0.2	0.27	0.47
Standard Deviation	0.29	0.1	0.15	0.15	0.1	0.21	0.25

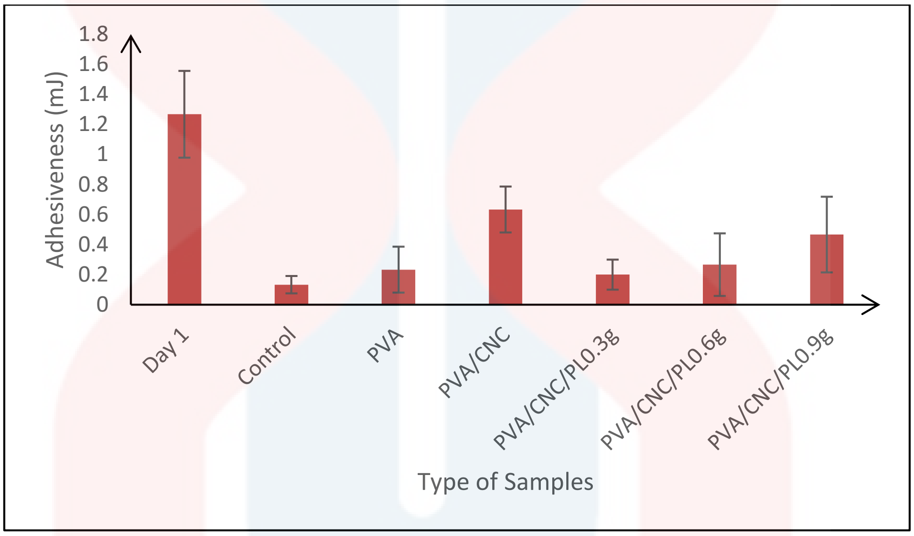

Figure 5.8.2: Adhesiveness (mJ) of Fresh Chilies on Day 1, and Control sample, PVA, PVA/ CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, and PVA/ CNC/ ε-PL 0.9g nanocomposite film on Day 15.

As Table 4.6.3 and Figure 4.6.3 shown, the adhesiveness of the fresh chilies on the first day was recorded as 1.27mJ (± 0.29mJ). On the fifteenth day, the fresh chilies for Control sample were tested 0.13mJ (±0.06mJ). 0.23mJ (± 0.15mJ) for the adhesiveness of the fresh chilies wrapped in PVA nanocomposite film and for PVA/ CNC nanocomposite film, it was tested 0.63mJ (± 0.15mJ). As for the following samples, the adhesiveness for the fresh chilies wrapped in PVA/ CNC/ ε-PL 0.3g nanocomposite film was 0.2mJ (± 0.1mJ), PVA/ CNC/ ε-PL 0.6g nanocomposite film with 0.23mJ (± 0.20mJ), and lastly for the adhesiveness of the fresh chilies wrapped in PVA/ CNC/ ε-PL 0.9g nanocomposite film was 0.47mJ (± 0.25mJ).

The most amount of adhesiveness of the fresh chilies was 0.63mJ (± 0.15mJ) which were wrapped in PVA/ CNC nanocomposite film while the least amount of adhesiveness were the fresh chilies wrapped in PVA/ CNC/ ε-PL 0.3g nanocomposite film with 0.2mJ (± 0.1mJ).

Table 5.8.3: Fracturability (g) of Fresh Chilies on Day 1, and Control sample, PVA, PVA/ CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, and PVA/ CNC/ ε-PL 0.9g nanocomposite film on Day 15.

Fracturability (g)	Day 1	Control	PVA	PVA/ CNC	PVA/ CNC/ PL 0.3g	PVA/ CNC/ PL 0.6g	PVA/ CNC/ PL 0.9g
Mean	1211.33	945.33	736.67	1204.67	1123.33	1045.33	1386
Standard Deviation	179.86	200.85	176.82	251.37	90.94	242.2	191.82

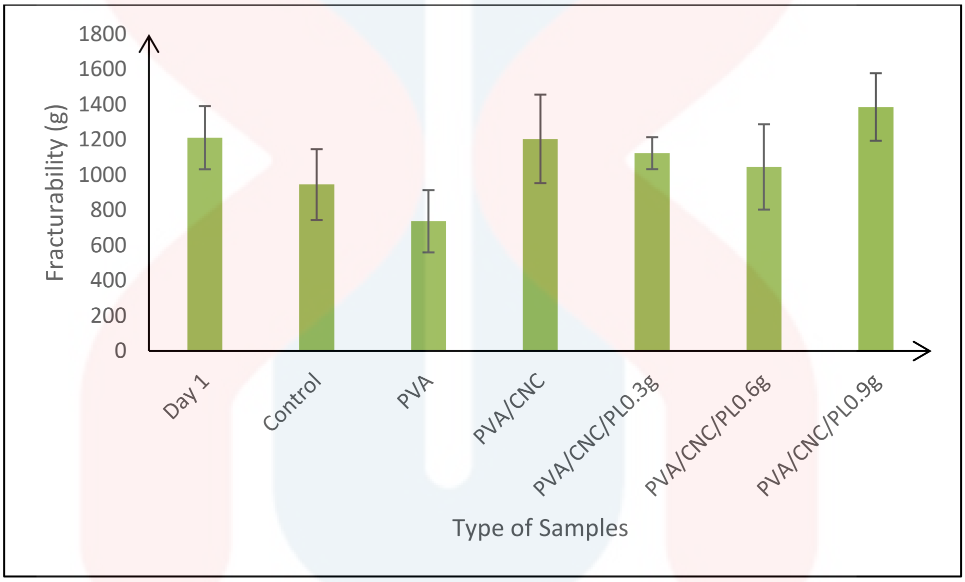

Figure 5.8.3: Fracturability (g) of Fresh Chilies on Day 1, and Control sample, PVA, PVA/ CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, and PVA/ CNC/ ε-PL 0.9g nanocomposite film on Day 15.

As shown in Table 4.6.3 and Figure 4.6.3, the first day of wrapping, the fresh chilies were tested for texture analysis and the fracturability of the fresh chilies was recorded at 1211.33h (± 179.86g). On the last day of the wrapping, which is 15 days later, the fresh chilies that were for Control sample was tested 945.33g (±200.85g). The fracturability of the fresh chilies wrapped with PVA nanocomposite film was 736.67g (±176.82g) and the fresh chilies wrapped in PVA/ CNC nanocomposite film was 1204.67g (± 251.37g). Next, for the fracturability of the fresh chilies wrapped in PVA/

CNC/ ε-PL 0.3g nanocomposite film was 1123.33g (± 90.94g), 1045.33g (± 242.20g) for PVA/ CNC/ ε-PL 0.6g nanocomposite film and lastly, the fracturability of the fresh chilies wrapped in PVA/ CNC/ ε-PL 0.9g nanocomposite film was 1386.00g (191.82g).

The most fracturability of the fresh chilies are the ones that were wrapped with PVA/ CNC/ ε-PL 0.9g nanocomposite film at 1386.00g (191.82g) while the least fracturability of the fresh chilies are the ones that were wrapped in PVA nanocomposite film at 736.67g (±176.82g).

Table 5.8.4: Cohesiveness of Fresh Chilies on Day 1, and Control sample, PVA, PVA/CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, and PVA/ CNC/ ε-PL 0.9g nanocomposite film on Day 15.

Cohesiveness	Day 1	Control	PVA	PVA/ CNC	PVA/ CNC/ PL 0.3g	PVA/ CNC/ PL 0.6g	PVA/ CNC/ PL 0.9g
Mean	1.1	0.67	1.03	1.1	0.9	0.94	1.5
Standard Deviation	0.11	0	0.1	0.17	0.16	0.24	0.28

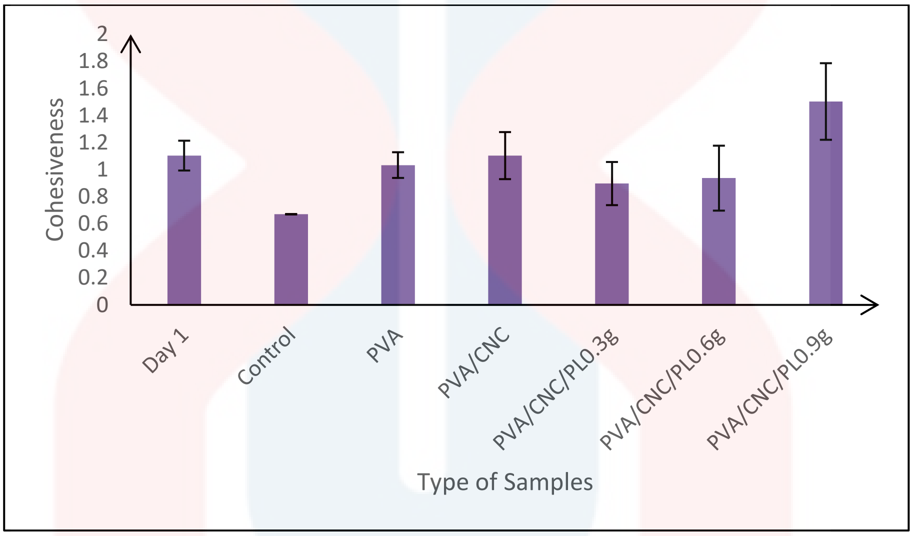

Figure 5.8.4: Cohesiveness of Fresh Chilies on Day 1, and Control sample, PVA, PVA/ CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, and PVA/ CNC/ ε-PL 0.9g nanocomposite film on Day 15.

Based on Table 4.6.4 and Figure 4.6.4, the cohesiveness of the fresh chilies on Day 1 was 1.10 (± 0.11). On Day 15, the cohesiveness of the fresh chilies for Control sample was 0.67 (± 0). The cohesiveness of the fresh chilies that were wrapped in PVA nanocomposite film was 1.03 (± 0.95) and 1.10 (± 0.17) for the fresh chilies wrapped in PVA/ CNC nanocomposite film. Next, the fresh chilies that were wrapped in PVA/ CNC/ ε-PL 0.3g nanocomposite film has the cohesiveness of 0.90 (± 0.16), 0.94 (± 0.24) for PVA/ CNC/ ε-PL 0.6g nanocomposite film and lastly, 1.50 (± 0.28) for PVA/ CNC/ ε-PL 0.9g nanocomposite film.

The highest cohesiveness of the fresh chilies are the ones that were wrapped in PVA/ CNC/ ε-PL 0.9g nanocomposite film at 1.50 (± 0.28) while PVA/ CNC/ ε-PL 0.3g nanocomposite film has the lowest cohesiveness of 0.90 (± 0.16).

Table 5.8.5: Springiness (mm) of Fresh Chilies on Day 1, and Control sample, PVA, PVA/ CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, and PVA/ CNC/ ε-PL 0.9g nanocomposite film on Day 15.

Springiness (mm)	**Day 1**	**Control**	**PVA**	**PVA/ CNC**	**PVA/ CNC/ PL 0.3g**	**PVA/ CNC/ PL 0.6g**	**PVA/ CNC/ PL 0.9g**
Mean	4.41	2.76	4.37	4.1	4.05	4.04	4.1
Standard Deviation	0.55	0.34	0.85	0.32	0.48	0.44	0.46

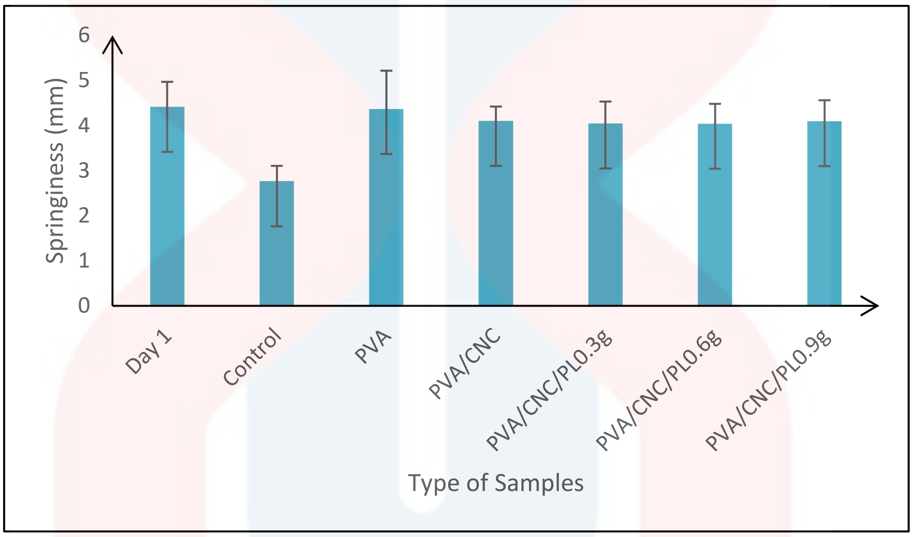

Figure 5.8.5: Springiness (mm) of Fresh Chilies on Day 1, and Control sample, PVA, PVA/ CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, and PVA/ CNC/ ε-PL 0.9g nanocomposite film on Day 15.

As shown in Table 4.6.5 and Figure 4.6.5, the springiness of the fresh chilies on the first day was tested 4.41mm (± 0.55mm). On Day 15, the fresh chilies for Control sample were 2.76mm (± 0.34mm) followed by 4.37mm (± 0.85mm) for the fresh chilies wrapped in PVA nanocomposite film, and 4.10mm (± 0.32mm) for the fresh chilies wrapped in PVA/ CNC nanocomposite film. For PVA/ CNC/ ε-PL 0.3g nanocomposite film, the springiness of the fresh chilies was 4.05mm (± 0.48mm), 4.04mm (± 0.44mm) for PVA/ CNC/ ε-PL 0.6g nanocomposite film and lastly, the springiness of the fresh chilies for PVA/ CNC/ ε-PL 0.9g was 4.10mm (± 0.46mm).

The fresh chilies that remained with the highest springiness are the fresh chilies wrapped in PVA nanocomposite film at 4.37mm (± 0.85mm) while the lowest springiness are the fresh chilies wrapped in PVA/ CNC/ ε-PL 0.6g nanocomposite film at 4.04mm (± 0.44mm).

Table 5.8.6: Gumminess (g) of Fresh Chilies on Day 1, and Control sample, PVA, PVA/ CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, and PVA/ CNC/ ε-PL 0.9g nanocomposite film on Day 15.

Gumminess (g)	Day 1	Control	PVA	PVA/ CNC	PVA/ CNC/ PL 0.3g	PVA/ CNC/ PL 0.6g	PVA/ CNC/ PL 0.9g
Mean	1683.67	910.33	1129.33	1352	903	922	1482.67
Standard Deviation	115	51.83	222.72	261.17	113.03	424.87	339.48

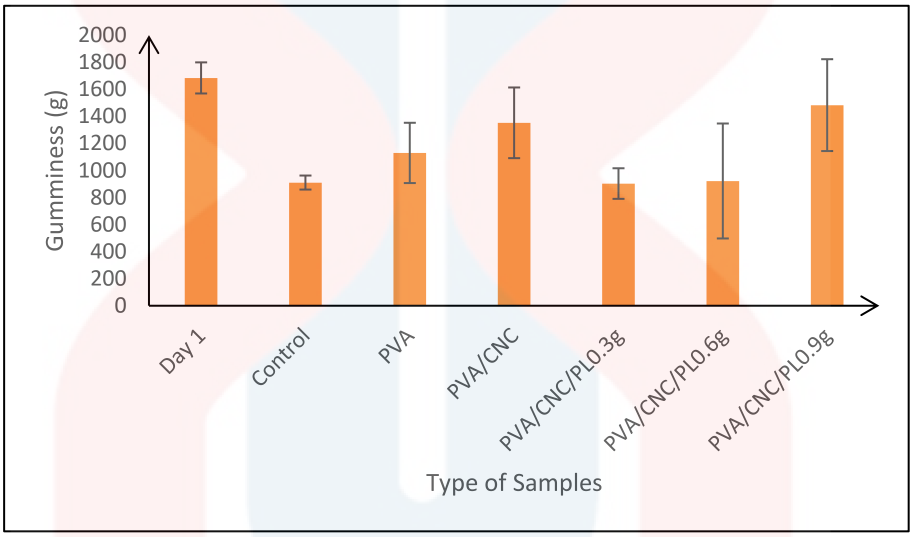

Figure 5.8.6: Gumminess (g) of Fresh Chilies on Day 1, and Control sample, PVA, PVA/ CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, and PVA/ CNC/ ε-PL 0.9g nanocomposite film on Day 15.

For the last graph for Texture Analysis as shown in Table 4.6.6 and Figure 4.6.6, the gumminess of the fresh chilies was tested. On Day 1, the gumminess of the fresh chilies was 1683.67g (± 115.00g). On Day 15, the gumminess of the fresh chilies for Control sample was 901.33g (± 51.83g). The fresh chilies that were wrapped in PVA nanocomposite film and PVA/CNC nanocomposite film were 1129.33g (± 222.72g) and 1352.00g (± 261.17g). Next, for the fresh chilies that were wrapped in PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, and PVA/ CNC/ ε-PL 0.9g nanocomposite films were 903.00g (± 113.03g), 922.00g (± 424.88g) and 1482.67g (± 339.48g).

The fresh chilies that remained the most gumminess is the ones that were wrapped in PVA/ CNC/ ε-PL 0.9g while the least gumminess is the ones that were wrapped in PVA/ CNC/ ε-PL 0.3g.

The texture of the fresh chilies changed after 15 days of wrapping in the nanocomposite film is due to the improper ways of storage of the samples. The fresh chilies lose the water in them overtime therefore it affects the overall texture of the chilies. With the environment and humidity, it speeds up the ripening process. The nanocomposite film helped in slowing down the ripening process due to the properties of the films. With the properties of antibacterial from CNC and ε-Polylysine, it has prolonged the shelf life of the fresh chilies by preventing bacteria to grow quickly. (Noronha et al., 2021) Red chillies lose their hardness during storage due to chemical modifications in the cell walls caused by protopectin, which is not soluble in water and softens the texture. (Teoriman et al., 2022)

5.9 pH Test

Table 5.9.1: pH Level of sample on Day 1 and Control sample, PVA, PVA/ CNC, PVA/ CNC/ ε-PL 0.3g, PVA/CNC/ ε-PL 0.6g, and PVA/ CNC/ ε-PL 0.9g nanocomposite film on Day 15.

pH	Day 1	CONTROL	PVA	PVA/CNC	PVA/CNC/PL 0.3	PVA/CNC/PL 0.6	PVA/CNC/PL 0.9
	6.6	7	7.6	6.9	6.1	5.7	6.3

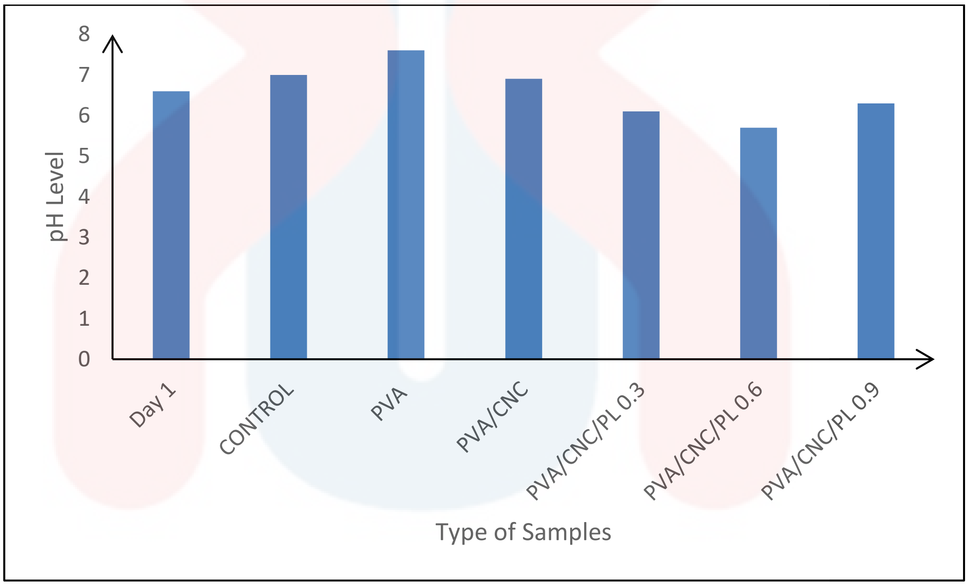

Figure 5.9.1: pH Level of sample on Day 1 and Control sample, PVA, PVA/CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, and PVA/ CNC/ ε-PL 0.9g nanocomposite film on Day 15.

Based on Table 4.7.1 and Figure 4.7.1, the graph shows the pH level of sample on Day 1 and Day 15. On the first day, the fresh chillies have the pH level of 6.6. On Day 15, the pH level of fresh chillies for Control sample is 7.0, PVA sample with pH level of 7.6, PVA/ CNC sample with 6.9, PVA/ CNC/ ε-PL 0.3g sample with 6.1 level of pH, 5.7 pH for the sample PVA/ CNC/ ε-PL 0.6g and lastly, 6.3 pH level for sample PVA/ CNC/ ε-PL 0.9g sample.

For sample from Day 1, sample PVA/ CNC from Day 15 along with PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g and PVA/ CNC/ ε-PL 0.9g are all scaled as acidic. Meanwhile, for Control sample and PVA sample are neutral. This is because PVA usually is neutral, although contaminants in the PVA may alter its pH. If the PVA is pH-neutral, it may not significantly influence the chilies' pH. (Mok et al., 2020) However, for the changes in pH for the fresh chilies after 15 days are also affected by the environment of the sample such as the temperature and the humidity when it was kept. (W. Li et al., 2020)

5.10 Weight Loss

Table 5.10.1: Weight Loss of Fresh Chilies

Sample / Day	Day 0	Day 5	Day 10	Day 15	Weight Loss (%)
CONTROL	6	5.5	5	4.9	18.33
PVA	9	8.86	8.74	8	11.11
PVA/CNC	8	7.54	7.48	7	12.5
PVA/CNC/ ε-PL0.3	9	8.66	8.57	8.43	6.33
PVA/CNC/ ε-PL0.6	7	7	6.96	6.87	1.86
PVA/CNC/ ε-PL0.9	6	6.07	5.97	5.81	3.17

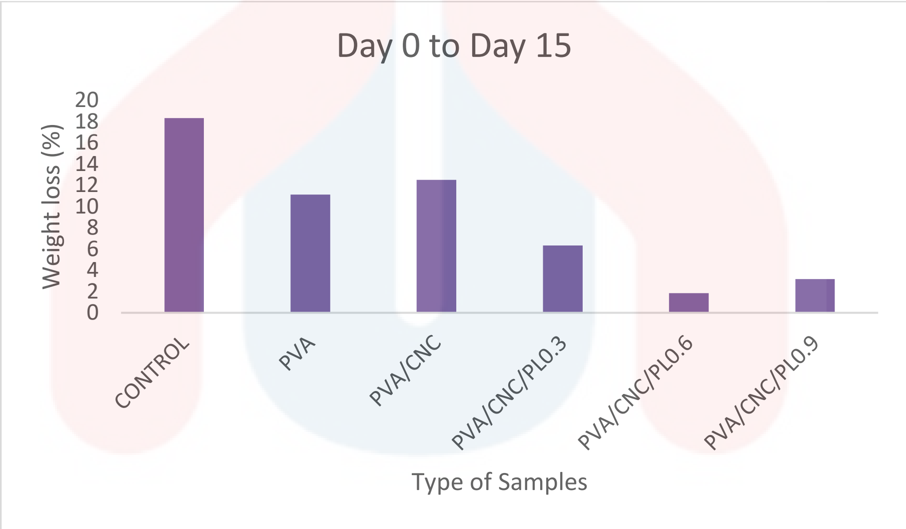

Figure 5.10.1: Weight loss of Fresh Chilies wrapped in PVA, PVA/ CNC, PVA/ CNC/ ε-PL 0.3g, PVA/ CNC/ ε-PL 0.6g, and PVA/ CNC/ ε-PL 0.9g nanocomposite films from Day 0 to Day 15

As shown in Table 4.8.1 and Figure 4.8.1, the weight of the fresh chilies on Day 1 for Control sample weighed 6g and it only weighed 4.9g on Day 15. It has 18.33% of weight loss. For the fresh chilies wrapped in PVA nanocomposite film, it weighed 9g on Day 0 and by Day 15, the chilies weighed 8g. It has 11.11% of weight loss. The fresh chilies wrapped in PVA/ CNC nanocomposite film weighed 8g on Day 0 and 7g on Day 15, having 12.5% of weight loss. On Day 0, the fresh chilies wrapped in

PVA/ CNC/ ε-PL0.3g weighed 9g and 8.43g on Day 15. It has 6.33% of weight loss. Next, the weight of the fresh chilies wrapped in PVA/ CNC/ ε-PL 0.6g were 7g on Day 0 and 6.87g on Day 15. There was 1.86% of weight loss. Lastly, the fresh chilies wrapped in PVA/ CNC/ ε-PL0.9g weighed 6g on Day 0 and 5.81g on Day 15. It has 3.17% of weight loss. The highest percentage of weight loss is 18.33% which is the Control, and the lowest percentage of weight loss is 1.86% which is the fresh chilies wrapped in PVA/ CNC/ ε-PL0.6g.

The reduction in crop weight after harvest is due to water loss through transpiration. Chillies lose weight due to respiration and transpiration, resulting in substrate and water loss and wilting on the surface. Mechanical trauma, such as cuts and bruising, leads to increased respiration and transpiration of complex chemicals in cells (carbohydrates), breaking volatile molecules like CO2 and H2O and causing chillies to lose weight. (Siahaan & Purwanto, 2022) With the characteristic of antimicrobial, the nanocomposite film managed to prolong the shelf life of the fresh chilies.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

6.1 Conclusion

In conclusion, this research has successfully achieved the objectives, which were to prepare and to characterize Poly (Vinyl Alcohol) (PVA), Cellulose Nanocrystals (CNC), and ε-Polylysine (ε-PL) nanocomposite films with different content percentages of CNC and ε-Polylysine and to investigate the potential of PVA/ CNC/ ε-PL nanocomposite film as food packaging for fresh chilies. The use of PVA as the primary material, chosen for its biocompatibility, film-forming capacity, and biodegradability, was effectively supplemented using CNC and ε-Polylysine as reinforcing additives. The nanocomposite films were thoroughly examined utilising Visual Inspection, Optical Microscopy, Universal Tensile Machine, FTIR, and XRD methods. Furthermore, using these nanocomposite films as food packaging for fresh chillies achieved the study's primary purpose. The findings shed light on the possibility of PVA/ CNC/ ε-PL nanocomposite films as ecologically harmless alternatives for food packaging, which aligns with the goals of minimising plastic waste and increasing the shelf life of packaged foods.

6.2 Recommendations

Based on the outcomes and the findings of this research, there are several recommendations that can be put forward such as conducting a longer study on the packaged fresh chilies to evaluate the prolonged shelf life and preservation performance of the PVA/ CNC/ ε-PL nanocomposite films. Besides that, it is also important to compare the overall environmental effects of PVA/ CNC/ ε-PL nanocomposite films to traditional plastic films. Antimicrobial test is recommended as it is important to know if the antimicrobial properties of ε-PL play a crucial role in the nanocomposite film.

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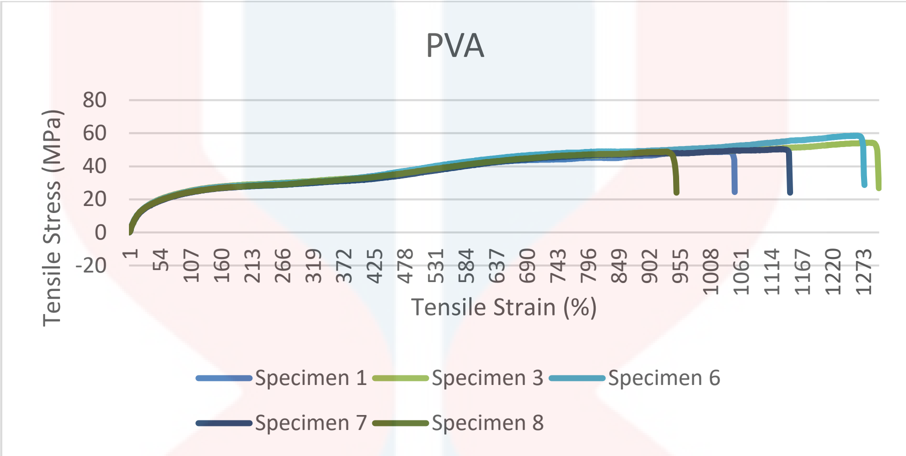
APPENDIX A

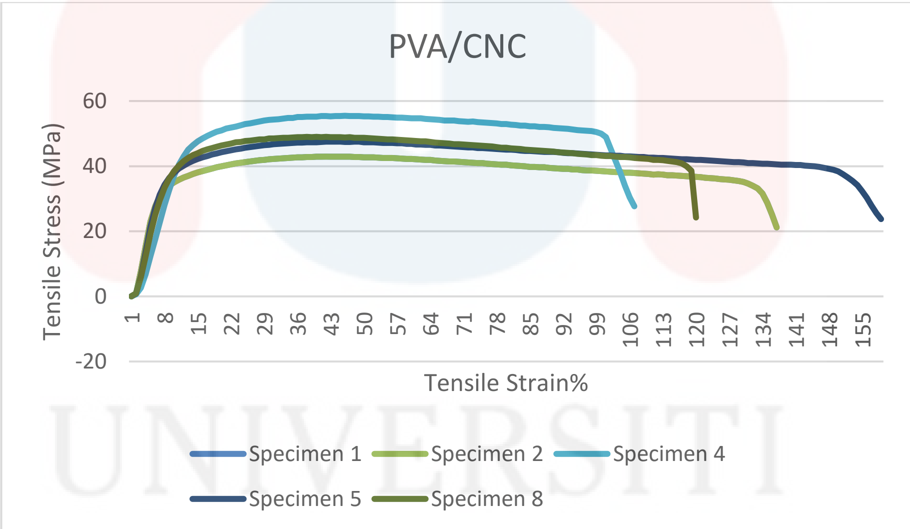
Sample solution of PVA, PVA/ CNC, PVA/ CNC/ PL0.3g, PVA/ CNC/ PL0.6g, and PVA/ CNC/ PL0.9g (left to right)

PVA/ CNC nanocomposite film

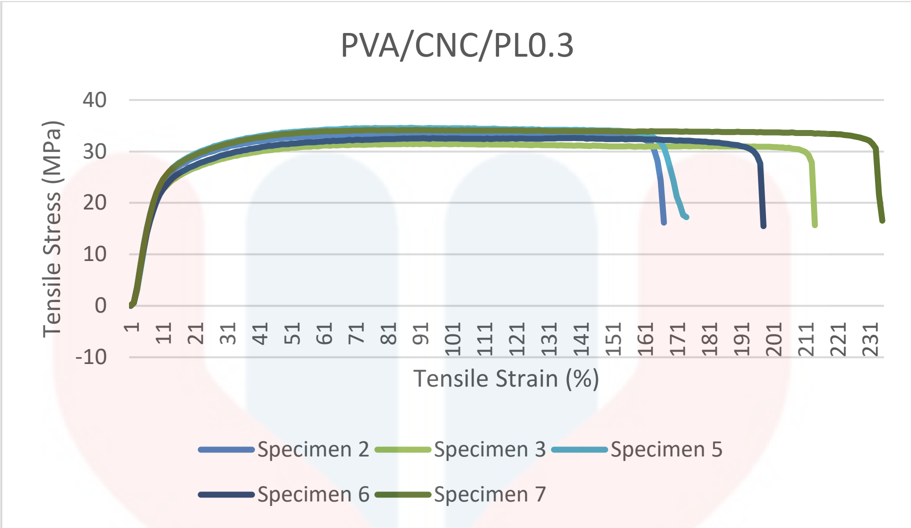
APPENDIX B



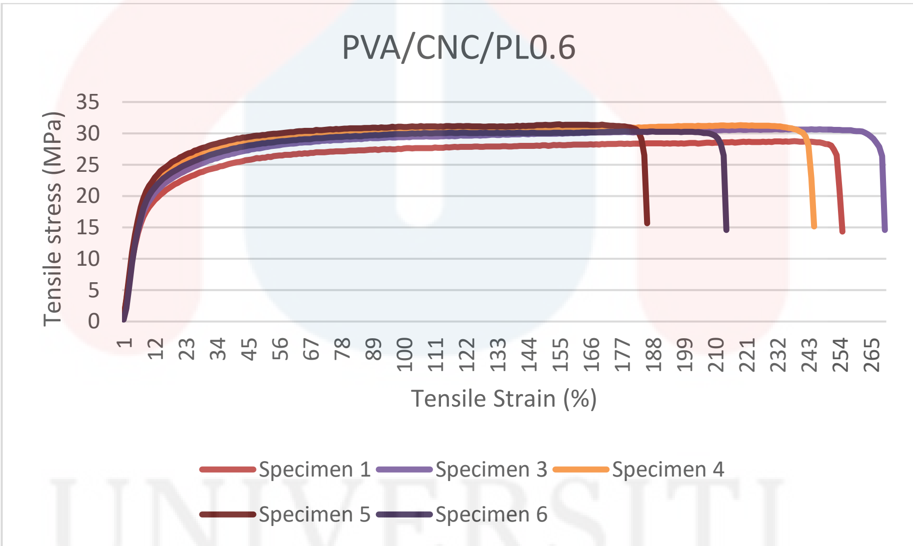
Tensile Stress (MPa) graph of PVA sample



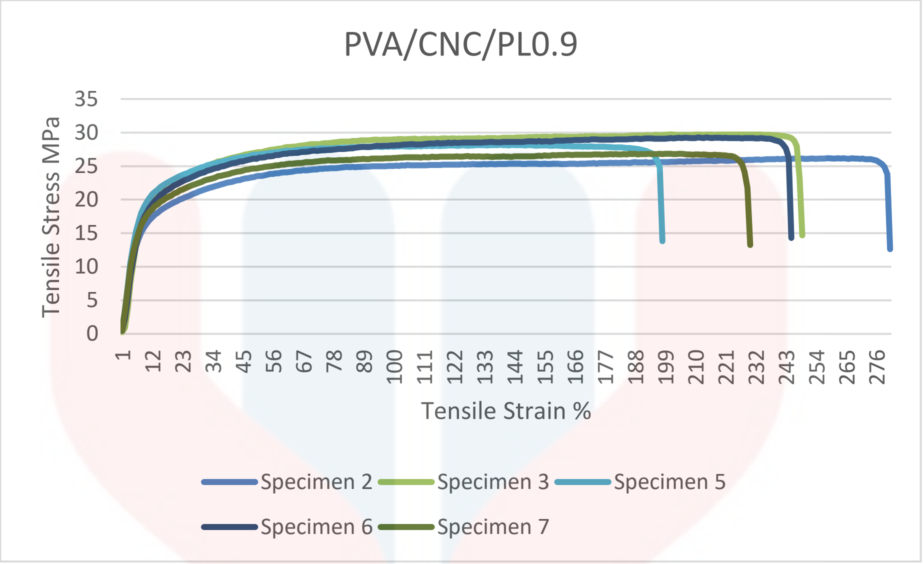
Tensile Stress (MPa) graph of PVA/ CNC sample



Tensile Stress (MPa) graph of PVA/ CNC/ PL 0.3g sample



Tensile Stress (MPa) graph of PVA/ CNC/ PL 0.6g sample



Tensile Stress (MPa) graph of PVA/ CNC/ PL 0.9g sample