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Preparation and characterization of PVA /Cellulose Nanocrystals/ e-polylysine Nanocomposite as Potential Food Packaging Application

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ABSTRACT

The thesis revolves around the synthesis and analysis of Polyvinyl Alcohol (PVA) Cellulose Nanocrystals (CNC) ε-Polylysine thin films, targeting their potential application in packaging food. The process of preparing these thin films involves solvent casting method, ensuring the proper incorporation of cellulose nanocrystals and polylysine into the PVA matrix. The characterization aspect of the research involves a comprehensive investigation into the physical and chemical properties of the resulting thin films. Mechanical properties, such as tensile strength and flexibility, x-ray diffraction analysis are examined to assess the material's durability. While for fresh fruit testing such as weight loss, brix analyzer, texture analyzer and pH are examined to know the freshness of chili before and after wrapped with the thin film.

By shedding light on these characteristics, the research aims to contribute valuable insights to the field of sustainable packaging. The goal is to provide an effective and eco-friendly alternative for food packaging applications, addressing both the mechanical and antimicrobial requirements essential for maintaining the quality and safety of packaged food products.

Keywords: cellulose nanocrystals, polyvinyl alcohol, biocomposite, tensile strength, x-ray diffraction.

ABSTRAK

Tesis ini berkisar tentang sintesis dan analisis filem nipis Polyvinyl Alcohol (PVA) Cellulose Nanocrystals (CNC) ε-Polylysine, menyasarkan potensi penggunaannya dalam pembungkusan makanan. Proses penyediaan filem nipis ini melibatkan kaedah tuangan pelarut, memastikan penggabungan nanokristal selulosa dan polilisin yang betul ke dalam matriks PVA. Aspek pencirian penyelidikan melibatkan penyiasatan menyeluruh terhadap sifat fizikal dan kimia filem nipis yang terhasil. Sifat mekanikal, seperti kekuatan tegangan dan kelenturan, analisis pembelauan sinar-x diperiksa untuk menilai ketahanan bahan. Manakala untuk ujian buah segar seperti penurunan berat badan, brix analyzer, texture analyzer dan pH diperiksa untuk mengetahui kesegaran cili sebelum dan selepas dibalut dengan lapisan nipis.

Dengan menjelaskan ciri-ciri ini, penyelidikan bertujuan untuk menyumbangkan pandangan berharga kepada bidang pembungkusan mampan. Matlamatnya adalah untuk menyediakan alternatif yang berkesan dan mesra alam untuk aplikasi pembungkusan makanan, menangani kedua-dua keperluan mekanikal dan antimikrob yang penting untuk mengekalkan kualiti dan keselamatan produk makanan yang dibungkus.

Kata kunci: nanokristal selulosa, polivinil alkohol, biokomposit, kekuatan tegangan, pembelauan sinar-x.

TABLE OF CONTENTS

CHAPTER 1

INTRODUCTION

1.1 Background

Polymer plastic is widely used for food packaging, but plastic mediums commonly require hundreds of years to disintegrate in the natural environment, at present, the world has shown many environmental issues mainly due to landfill and plastic pollution. After that,Food waste and the production of food byproducts are major issues that have negative impacts on the environment, the economy, and society.. Food waste reduction may be a significant way to cut production costs and create more effective food systems. Additionally, waste reduction can help establish more ecologically friendly food systems and increase food security and nutrition (Scholarly community Enclopedia,2021). Furthermore, the effect is landfill burden. It is because food waste constitutes a substantial portion of municipal solid waste, occupying limited landfill space. As food decomposes in landfills, it generates methane gas, contributing to climate change and air pollution. (Jean Buzby, (2022)).

Cellulose nanocrystals (CNCs) are tiny, rod-like particles that are derived from cellulose, which is the most abundant organic compound found in nature. CNCs have unique properties such as high mechanical strength, high aspect ratio, low toxicity, and biodegradability, which make them an attractive material for various applications, including food packaging. (Maria Paula Crioda, 2018)

Polyvinyl alcohol (PVA) is a water-soluble synthetic polymer that has excellent film-forming properties, making it suitable for use as a packaging material. PVA is widely used in the food industry because of its biocompatibility, non-toxicity, and ability to prevent moisture and gas transmission. (Jitendra Pratap Singh, 2017).

ε-Polylysine is a natural antimicrobial peptide that is produced by fermentation of Streptomyces albulus. It has a broad spectrum of antimicrobial activity against bacteria, yeasts, and molds, and is used as a food preservative. ε-Polylysine is considered safe for consumption and has been approved for use in many countries. The key advantage of ε-Polylysine is natural and healthy, broad-gauge antibacterial spectrum, good water solubility and thermal stability, effective over a wide range of pH values, friendly blend with other preservatives, improve cost efficiency due to low dosage rate, extend shelf life, and wide range of applications. (Jiang Hua, 2021)

Polymer nanocomposites are materials composed of a polymer matrix with nanoscale fillers dispersed throughout. These fillers, often nanoparticles or nanofibers, can be derived from various materials such as metals, ceramics, or carbon-based substances like carbon nanotubes or graphene. The addition of nanoscale fillers to polymers offers several advantages and enhanced properties compared to traditional polymer composites which are improved mechanical properties, enhanced thermal stability, can exhibit superior barrier properties, and improved optical properties (. Bratovcic, A, 2015)

1.2 Problem Statement

The problem with traditional petroleum-based plastics used for food packaging is that they are not biodegradable that will lead to pollution and other environmental problems. Furthermore, the use of these materials contributes to the depletion of finite resources and contributes to greenhouse gas emissions (. Jean Buzby, 2022). Around 4 per cent of world oil production is used as a feedstock to make plastics and a similar amount is used as energy in the process. Yet over a third of current production is used to make items of packaging, which are then rapidly discarded. From that, there was a lot of food waste happened in this world. Based on US Department of Agriculture, around 2.5 billion tons of food waste for each year. The food waste will go through landfill and make the gas of methane that will affect the people surrounding. (Jean Buzby,2022). So, from this research, it will save food and extend the shelf life of the food.

Therefore, there is a need to develop sustainable and eco-friendly alternatives to traditional food packaging materials. CNCs, PVA, and ε-Polylysine have the potential to provide such an alternative as they are biodegradable, non-toxic, and can be derived from renewable sources. If just only use PVA to make the biodegerable fdod packaging, it is not enough a good quality. From this research, ε-Polylysine will be add as a filler with PVA as a reinforcement to make it more quality as potential food packaging applications. However, to be effective in food packaging applications, the materials must possess sufficient mechanical strength, barrier properties, and antimicrobial activity to protect the food and extend its shelf life., extending the shelf life of food products and ensuring their safety and quality.

Thus, the problem statement for the application of CNCs, PVA, and ε-Polylysine in food packaging is to develop a bio-based and biodegradable packaging material that meets the necessary mechanical, barrier, and antimicrobial requirements to protect and preserve food products while also being sustainable and environmentally friendly.

1.3 Objective

To achieve the development goal, the objective is very important. Objectives of this research are:

1. to prepare and characterize PVA/CNC/ε-Polylysine nanocomposite thin films by using of solvent casting method.
2. to investigate the potential of biodegradable PVA/CNC/ ε-Polylysine nanocomposites thin film for fresh chili packaging application

1.4 Scope of Study

The study aimed to explore the preparation and characterization of a nanocomposite material consisting of three key components: polyvinyl alcohol (PVA), cellulose nanocrystals, and e-polylysine.

Polyvinyl alcohol (PVA), a synthetic polymer with excellent film-forming properties, is widely used in various applications, especially in food packaging (Jitendra Pratap Singh). Cellulose nanocrystals, derived from cellulose, are nanoscale particles that exhibit high strength, low density, and excellent barrier properties (Maria Paula Crioda, 2018). E-polylysine, a natural antimicrobial agent commonly used in the food industry to inhibit the growth of microorganisms (Jiang Hua Zhang, 2021).

The study involved several steps. Firstly, the PVA/cellulose nanocrystals/ε-Polylysine nanocomposite was prepared by incorporating cellulose nanocrystals and ε-Polylysine into a PVA matrix. The precise formulation and manufacturing process were optimized to achieve a uniform dispersion of the nanocrystals and ε-Polylysine within the PVA matrix.

Next, the prepared nanocomposite was characterized using various techniques. The physical and mechanical properties, such as tensile strength, flexibility, and barrier properties, were analyzed. Additionally, the thermal stability and morphological characteristics of the nanocomposite were examined using techniques such as scanning electron microscopy (SEM), X-ray diffraction (XRD), and thermogravimetric analysis (TGA) to determine their shape, crystallinity, and thermal stability. The nanocomposites were examined for their resistance to oxygen and water vapor using industry-standard techniques, including permeability testing. The applicability of the produced nanocomposites as food packaging materials was assessed based on their mechanical, thermal, and barrier properties.

1.5 Significant of Study

The significance of study for this topic, by using biodegradable nanocomposites in food packaging applications, offers several advantages for the consumers. The advantages are environmental sustainability, reduce waste, extend shelf life, health, and safety, and improve product performance.

First, the advantage is environmental sustainability. Biodegradable nanocomposites are designed to degrade naturally over time, reducing the environmental impact of packaging waste. They can be derived from renewable resources such as plant-based polymers, making them a more sustainable alternative to traditional non-biodegradable materials like plastics. After that, the advantage is reducing waste. Biodegradable nanocomposites can break down into non-toxic components, reducing the accumulation of non-biodegradable packaging in landfills and oceans. By utilizing biodegradable materials, food packaging waste can be minimized.

Next, the advantage is extending shelf life. It is because they provide an effective barrier against oxygen, moisture, and other contaminants, preserving the freshness and quality of the food for a longer period. This can help reduce food waste and improve overall food safety. Furthermore, the advantage is health and safety. It is because biodegradable nanocomposites can be designed to be non-toxic and free from harmful additives. This ensures that the packaging materials do not leach harmful substances into the food, maintaining food safety standards and minimizing health risks. Lastly, the advantage is improved product performance. It is because nanocomposite materials can be engineered to possess desirable properties like enhanced mechanical strength, flexibility, and thermal stability. These properties contribute to better protection and handling of food products, ensuring they remain intact and uncontaminated during storage and transportation.

CHAPTER 2

LITERATURE REVIEW

2.1 Polymer

2.1.1 Polymer Nanocomposite

A polymer nanocomposite refers to a composite material in which a polymer matrix is reinforced or filled with nanoscale particles, often referred to as nanofillers or nanoparticles. When nanofillers add to the polymer matrix, it can significantly enhance the properties and performance of the composite material. The nanofillers have a high surface area-to-volume ratio and unique physical and chemical properties at the nanoscale, which impart desirable characteristics to the polymer matrix.

One of the advantages of polymer nanocomposites is their improved mechanical properties. The presence of nanofillers can enhance the stiffness, strength, and toughness of the polymer matrix, resulting in materials with superior mechanical performance compared to pure polymers. The strong interaction between the nanofillers and the polymer matrix helps to distribute and transfer stress more effectively, improving load-bearing capacity and resistance to deformation.

2.1.2 Poly (Vinyl Alcohol)

Polyvinyl alcohol known as PVA is a synthetic polymer derived from vinyl acetate through a process called hydrolysis. It is a water-soluble and biodegradable polymer that has a range of useful properties, making it suitable for various applications, including food packaging. The characteristics of PVA are water solubility which is easy to process and allows for the formation of films, coatings, and fibers, film-forming ability which means it can be cast or extruded into thin films, barrier properties which is helps to preserve the freshness and quality of packaged products, and biodegradability which is environmentally friendly for surroundings.

2.1.3 ε-Polylysine

ε-Polylysine is a natural antimicrobial agent that is commonly used in various industries, including the food industry. It works by disrupting the integrity of microbial cell membranes and interfering with their essential metabolic processes. This disruption leads to the inhibition of microbial growth and, in some cases, the death of the microorganisms. (Liang Wang, 2021). It works by interacting and binds with the negatively charged microbial cell membrane and forms holes in membrane. After that, ε-Polylysine will enter the cell through the pores in the cell membrane, affects the structure and function of the cell membrane and ultimately leads to the death of bacterial cells.

2.1.4 Cellulose

2.1.4.1 Cellulose Nanocrystals

Cellulose nanocrystals (CNCs) are nanoscale particles derived from cellulose, which is an organic polymer found in nature. Cellulose is primarily extracted from renewable sources such as wood pulp, agricultural residues, and plants. By treating cellulose with powerful acids to disintegrate the cellulose fibers into tiny crystalline particles, a procedure known as acid hydrolysis produces CNCs.

The resulting CNCs have unique properties that make them attractive for a wide range of applications in various industries, including food packaging. The characteristics of CNCs are nanoscale size, which is have dimensions on the nanometer scale, high aspect ratio, which is improves the mechanical strength of composite materials, have crystalline structure which is contributes to the high stiffness, thermal stability, and barrier properties of CNC-based materials, and biodegradability which is leads to reducing the environmental impact associated with conventional materials.

2.2 Preparation of Polymer Nanocomposite

2.2.1 Solvent Casting Method

The solvent casting method is a technique used to fabricate thin films or membranes by dissolving a polymer in a suitable solvent and subsequently casting the polymer solution onto a substrate. As the solvent evaporates, it leaves behind a solid polymer film with desired properties. The solvent casting method is widely used in various industries, including biomedical, electronics, optics, and coatings. It allows for the fabrication of thin films or membranes with precise control over thickness, composition, and properties. These films find applications such as drug delivery systems, sensors, electronic devices, membranes for separation processes, and surface coatings.

2.2.2 Extrusion Technique

Extrusion is a widely used manufacturing technique for producing various products, including thin films for packaging food. The process involves forcing a material, typically a thermoplastic polymer, through a die to create a continuous shape with a consistent cross-section. The extrusion technique allows for high-volume production of continuous thin films with consistent thickness and properties. The process can be easily adjusted to accommodate different materials, die designs, and processing parameters, making it versatile and widely used in various industries, including food packaging.

2.3 Characterization Techniques of Polymer Nanocomposite

2.3.1 Visual Inspection

Visual inspection is a process that relies on human observation and visual perception to evaluate the quality, condition, or compliance of products, components, or systems. It involves carefully examining objects using the naked eye or aided by tools such as magnifying glasses, microscopes, or specialized inspection equipment. (Judi E See,2012).

2.3.2 Optical Inspection

Optical inspection, also known as vision inspection or machine vision, is a technology-driven process used for automated quality control and inspection in various industries. It involves the use of optical systems, cameras, image processing algorithms, and computer software to capture and analyse images or video footage of products or components in order to detect defects, measure dimensions, and ensure overall product quality. (Judi E See,2012)

2.3.3 X-RAY Diffraction (XRD)

X-ray Diffraction (XRD) is a technique used for determining the atomic and molecular structure of a crystalline material, in which the crystalline structure causes a beam of incident X-rays to diffract into many specific directions (ICDD, 2024). XRD is widely used in materials science, geology, chemistry, and various other fields to study the crystal structure, phase composition, and crystalline properties of materials. It plays a crucial role in the characterization of minerals, metals, ceramics, pharmaceuticals, and the development of new materials with desired properties (Jiang Hua, 2021).

2.3.4 Tensile Strength

Tensile strength is the maximum amount of tensile stress a material can withstand before it fails or breaks. It is a measure of the material's ability to resist deformation under tension or stretching forces (Raghvendra Gopal, 2023). It represents the material's ability to resist being pulled apart or stretched under tension. It is used to characterize and compare materials. It helps in selecting appropriate materials for structural applications, engineering designs, and manufacturing processes where tensile forces are expected. Different materials exhibit varying tensile strengths, with materials like steel and titanium generally possessing high tensile strengths, while materials like rubber and plastics typically have lower tensile strengths. Understanding the tensile strength of a material is crucial for ensuring its performance and safety in various industries, including construction, automotive, aerospace, and manufacturing (Jiang Hua, 2021).

2.4 Application of Polymer Nanocomposites as Fresh Fruit Packaging

2.4.1 Preparation of Fresh Fruit

The preparation of fresh fruit involves several steps to ensure that it is clean, ripe, and ready to be consumed. The fresh fruit will be selected in good condition, without any bruises, mold, or signs of spoilage. Look for fruits that have vibrant colors, firm texture, and a pleasant aroma.

2.4.2 Quality Assessment of Wrapped Food

2.4.2.1 Brix Analyzer

A brix analyzer, also known as a refractometer or digital refractometer, is a device used to measure the Brix value of a liquid. The Brix value is a measurement of the sugar content or total soluble solids in a liquid, primarily used in the food and beverage industry to determine the sweetness or concentration of a solution. Brix analyzers are portable, user-friendly, and provide quick measurements, making them valuable tools in various industries that deal with liquid concentrations. They help in maintaining product consistency, optimizing production processes, and ensuring customer satisfaction by accurately measuring the sugar content of liquids.

2.4.2.2 Texture Analysis

A texture analyzer is a specialized instrument used to evaluate and quantify the physical properties and characteristics of various materials, particularly in the food, pharmaceutical, cosmetics, and materials testing industries. It measures the mechanical properties of a sample, such as its hardness, compressibility, elasticity, adhesiveness, and brittleness, providing valuable insights into it texture and consistency.

2.4.2.3 Weight Loss

When referring to materials or substances, weight loss is the decrease in mass during a certain time or under a given set of circumstances. It's frequently expressed as a percentage of the starting mass. Weight loss analysis is useful in scientific research to assess the performance, stability, or deterioration of materials, including films, for thesis projects. For example, knowing how much mass a material loses with time in food packaging applications can reveal details about its resilience to environmental changes, longevity, and general appropriateness for maintaining the quality of the packed goods.

2.4.2.4 pH Testing

A pH test uses a scale of 0 to 14 to determine how acidic or alkaline a solution is; a pH of 7 is considered neutral. A pH of less than 7 indicates acidity, whereas a pH of greater than 7 indicates alkalinity. pH testing is useful in many applications, such as your food packaging thesis. For example, evaluating a product's pH or the atmosphere within its container might assist in ascertaining whether it is appropriate for a certain food. The impact of pH on flavor, texture, and shelf life is significant. To ensure the stability and safety of items, pH testing is usually done using pH indicators or meters. This is especially important when it comes to food packing, since maintaining the ideal pH level is crucial for retaining freshness and quality.

CHAPTER 3

MATERIALS AND METHOD

3.1 Overview

In this experiment, the raw material was used as powdered polyvinyl alcohol, powdered cellulose nanocrystals, and powdered polylysine. This raw material was weighed and mixed. Other than that, deionized water and distilled water are used in this experiment. This raw material was weighed and mixed by following its stoichiometric ratio. The amount of each raw material was determined. Shanghai Yuanye Bio-Technology Co., Ltd. supplied polyvinyl alcohol (PVA) that had a degree of polymerization of approximately 2500. (China). The Silver-Elephant Bio-engineering Company, Ltd. provided the e-PL that had a molecular weight of approximately 6 kDa (Zhejiang, China).

3.2 Materials

- Polyvinyl Alcohol (PVA) powder or pellets
- e-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)

- Oven or drying chamber

3.3 Methods

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

The CNC existed as a powder. For the raw materials' characterization technique, approximately 1.0 g of raw materials was 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 was poured into the petri dishes, where it was allowed to cure at room temperature. The CNC dispersion was added to the PVA solution first, and then the combination was 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 heated to 50 degrees Celsius while stirred constantly for an hour. Finally, the liquid was distributed among various petri dishes and allowed to air dry at ambient temperature. The films were dried at a temperature of 40 degrees Celsius for five hours before receiving a final rinse in deionized water.

PVA, CNC, and Polylysine solution samples were cast on glass Petri dishes and will be allowed 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 be created. The bio-composite thin film is then kept for characterization in a zip bag. 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. 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.

Content	Content of PVA(g)	Content of CNC(w%)	Content of ε-PL (wt%)
PVA	5.0	0.0	0.0
PVA and CNC	5.0	3.0	0.0
PVA/CNC/ ε-PL 1%	5.0	3.0	0.3
PVA/CNC/ ε-PL 2%	5.0	3.0	0.6
PVA/CNC/ ε-PL 3%	5.0	3.0	0.9

Table 3.1: Composition of Thin Film

3.4 Characterization of bio-nanocomposite films

3.4.1 X-RAY Diffraction (XRD)

Using an XRD diffractometer (D2 Phaser XRD Machine) 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.

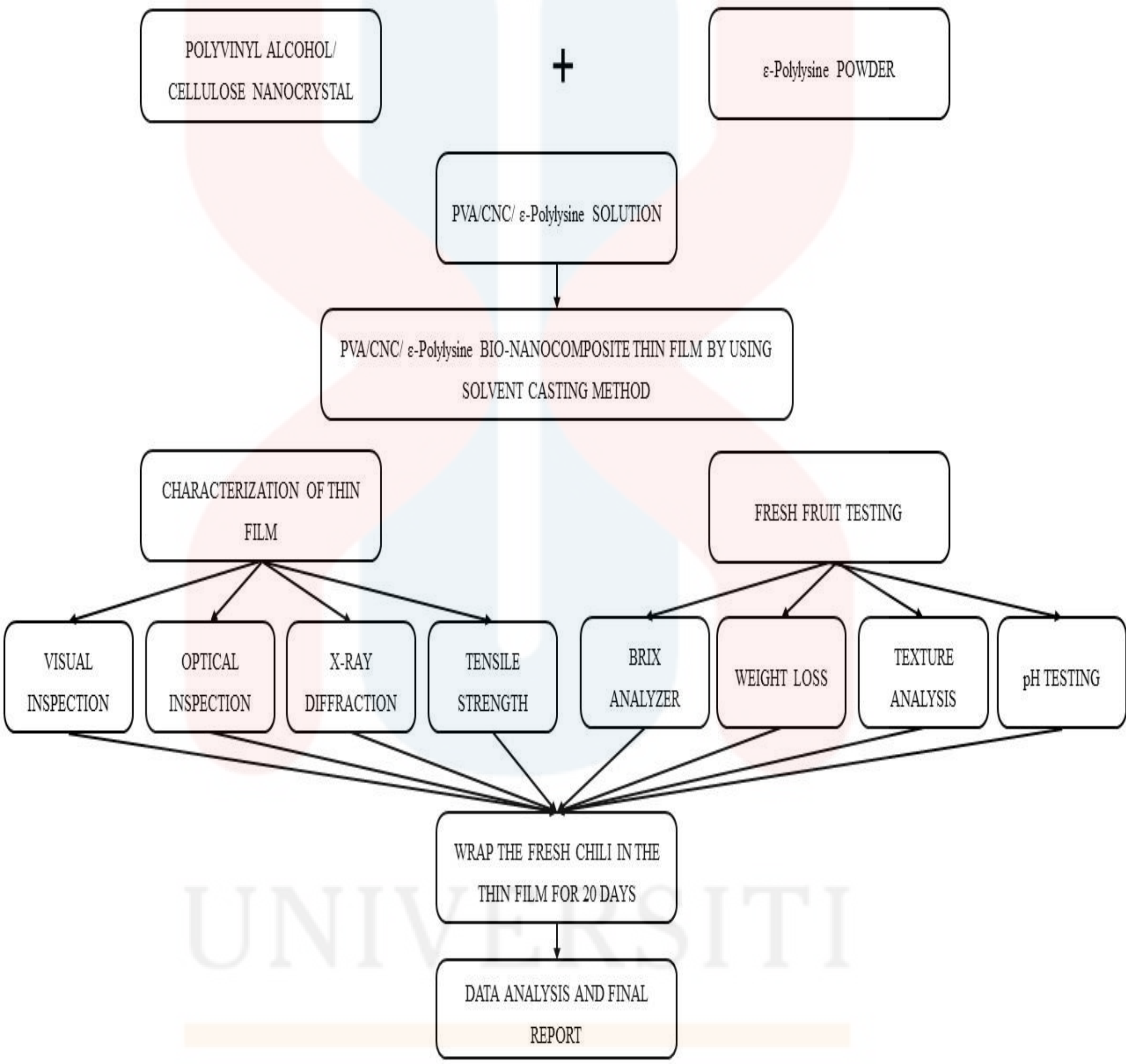
3.4.2 Film Thickness

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.

3.4.3 Tensile Strength

A universal testing device (TNUM-5900) was used to gauge the bio-nanocomposite films' tensile and elongation capabilities. Tensile and elongation tests were performed after the samples were divided into 300 x 100-mm rectangular strips. The values given are the meaning of three measurements.

3.5 Flow Chart



CHAPTER 4

RESULTS AND DISCUSSION

4.1 Overview

The intended outcome of the result will be detailed in this chapter. This section of the thesis covers visual inspection, solution mixture analysis, fresh food testing, weight loss and pH testing. This concise overview highlights the practical implications of the proposed solution for food preservation.

4.2 Visual Inspection

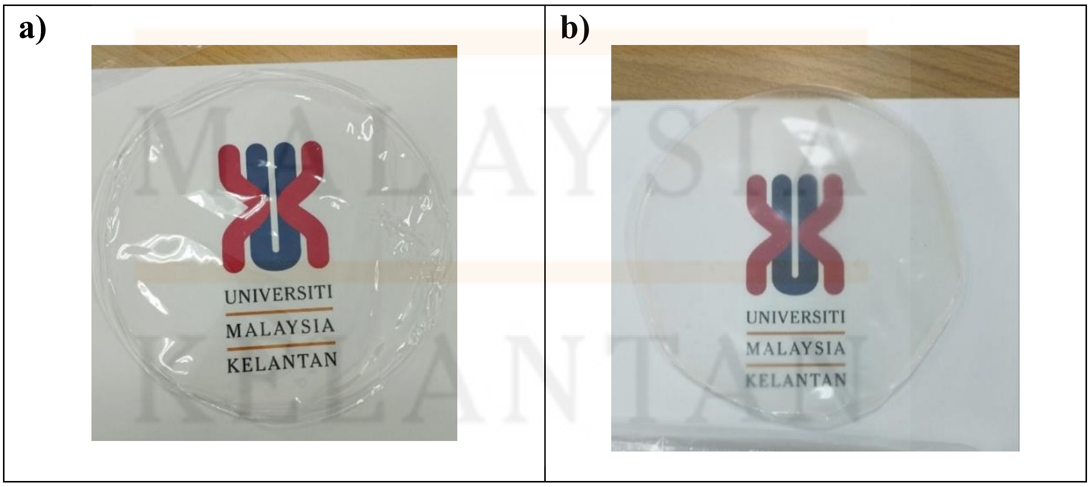


Figure 4.1: shown that a) PVA 5g thin film, b) PVA CNC 3g thin film.

c)	d)	e)
		

Figure 4.2: c) PVA CNC PL 0.3g thin film, d) PVA CNC PL 0.6g thin film, and e) PVA CNC PL 0.9g thin film.

As we can see from the result, the PVA thin film is more transparent compared to the PVA/ CNC 3g thin film, PVA/CNC/ PL 0.3g thin film, PVA/CNC/ PL 0.6g thin film, PVA/CNC/ PL 0.9g thin film. It is because when the solution was added on by polylysine, it will turn the solution to become cloudier.

4.3 Solution Mixture

4.3.1 Visual Inspection of Solution

Figure 4.3: From left, solution mixture of PVA 5g, PVA CNC 3g, PVA CNC PL 0.3g,

PVA CNC PL 0.6g and PVA CNC PL 0.9g

From this visual inspection, we can see that the solution of PVA was so clear compared to PVA CNC 3g, PVA CNC PL 0.3g, PVA CNC PL 0.6g, and PVA CNC PL 0.9g. It is because when we add the ε-Polylysine powder, it will change the solution to cloudy and more weight of ε-Polylysine powder, it will become cloudier and the concentrated of solution will be high.

4.3.2 Optical Inspection

Optical microscope was used for observed the structure of PVA/Cellulose Nanocrystal composite/ Polylysine thin film under 10x magnification polarized image.

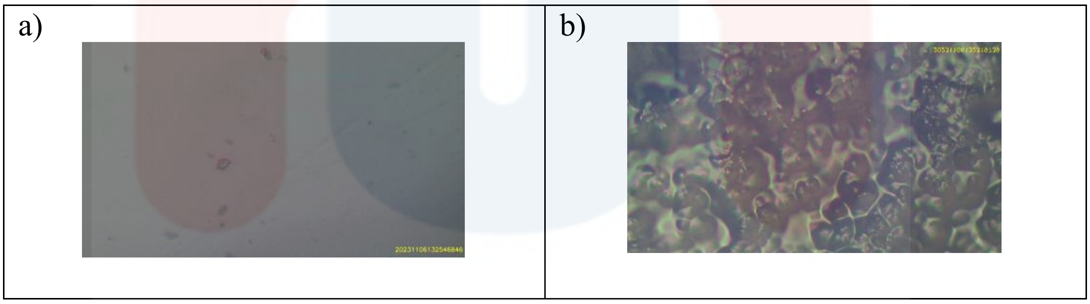


Figure4.4: OM image showed (a) is PVA 5g, (b) is PVA/CNC(3g)

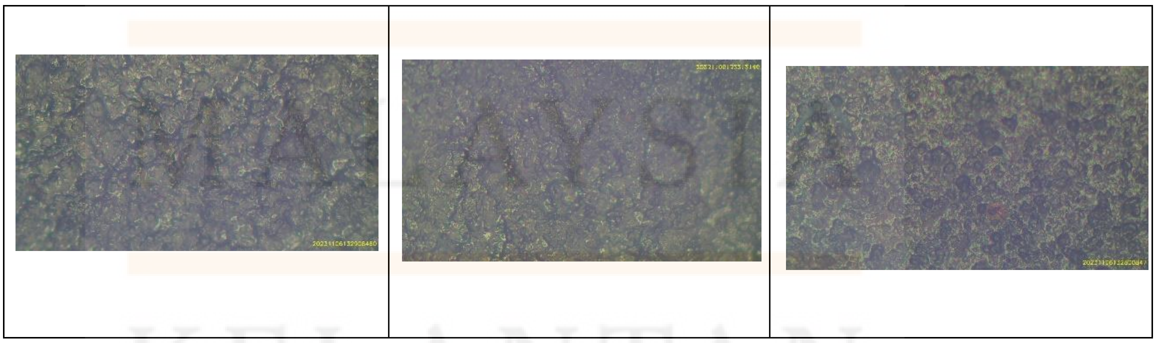


Figure 4.5: OM image showed (c) is PVA/CNC/PL0.3, (d) is PVA/CNC/PL0.6, and (e) is PVA/CNC/PL0.9

4.3.3 X-RAY Diffraction

Sample	Armophous %	Crystanallity index %
PVA 5g	70.3	29.7
PVA CNC 3g	59.1	40.9
PVA CNC PL 0.3g	61.6	38.4
PVA CNC PL 0.6g	54.8	45.2
PVA CNC PL 0.9g	65	35

Table 4.1: Percentage of amorphous and crystanallity index of CNC for each thin films

From the result, as we can see that PVA 5g have higher percentage of armophous which is 70.3% compared to the others composition of thin film. For crytanallity of cellulose nanocrystal, it shown that in the table which are PVA CNC 3g contains 40.9%, PVA CNC PL 0.3g contains 38.4%, PVA CNC PL 0.6g contains 45.2%, and PVA CNC PL 0.9g contains 35%.

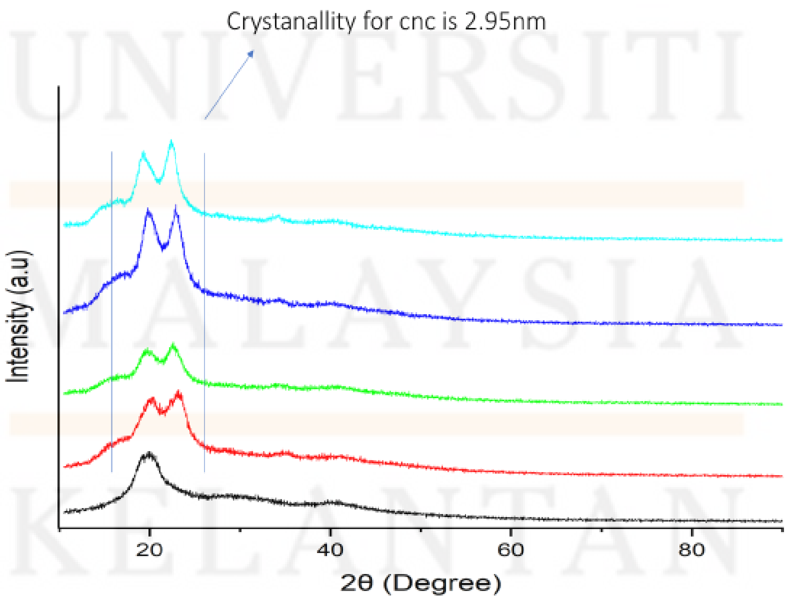


Figure 4.6: Graph XRD comparison for 5 difference thin film

From the graph, the (black line) is shown the armophous of PVA 5g thin film, for (red line) shown that crystanallity for thin film PVA CNC 3g, for (green line) shown that crystanallity for thin film PVA CNCN PL 0.3g, for (purple line) shown that crystanallity of thin film PVA CNC PL 0.6g, and for (blue line) shown that crystanallity for thin film PVA CNC PL 0.9g.

From the graph, we can see that the line of PVA 5g thin film do not have peak of crystanallity. It is because PVA is armophous. Compared to others thin film, it will show the peak of crystanallity in X- ray diffraction test. It is because the presence of cellulose nanocrystal that makes the graph show the peak in the others thin film.

4.3.4 Tensile Strength

SAMPLE	ModulusYoung (Mpa)	Stress at break (Mpa)	Elongationat break (Mpa)
PVA	**355.57±13.22**	**46.18±5.00**	**215.558±29.17**
PVA/CNC 3g	**191.37±23.00**	**35.91± 1.82**	**108.08±17.81**
PVA / CNC/ PL 0.3g	**1352.95±21.18**	**36.16± 2.27**	**110.90± 4.27**
PVA / CNC/ PL 0.6g	**1594.72± 24.56**	**28.89± 1.78**	**60.66± 9.52**
PVA / CNC/ PL 0.9g	**1016.27± 34.59**	**29.67± 2.28**	**130.63± 13.03**

Table 4.2: Modulus Young, Stress at break, and Elongation at break for each thin film

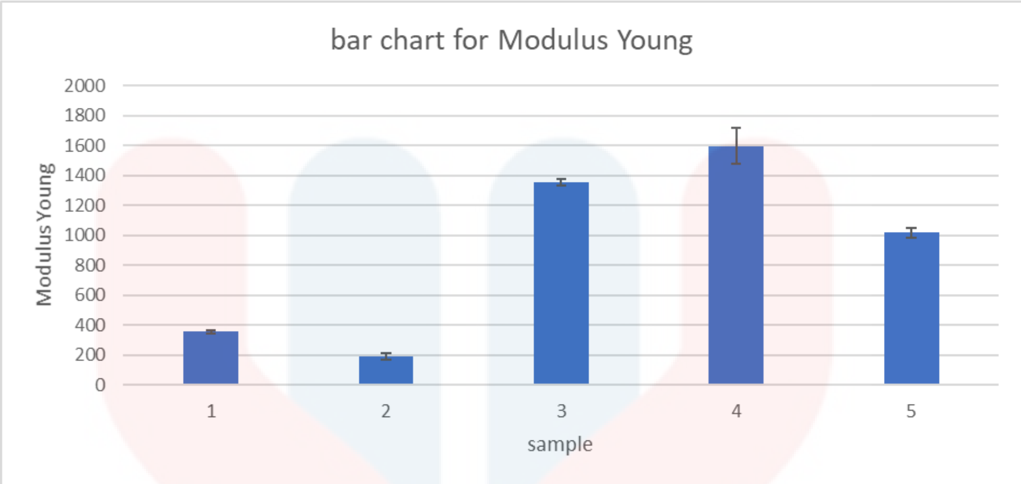


Figure 4.7: Bar chart for Modulus Young for thin films of PVA 5g (1), PVA CNC 3g (2), PVA CNC PL 0.3(3)g, PVA CNC PL 0.6g (4) and PVA CNC PL 0.9g (5).

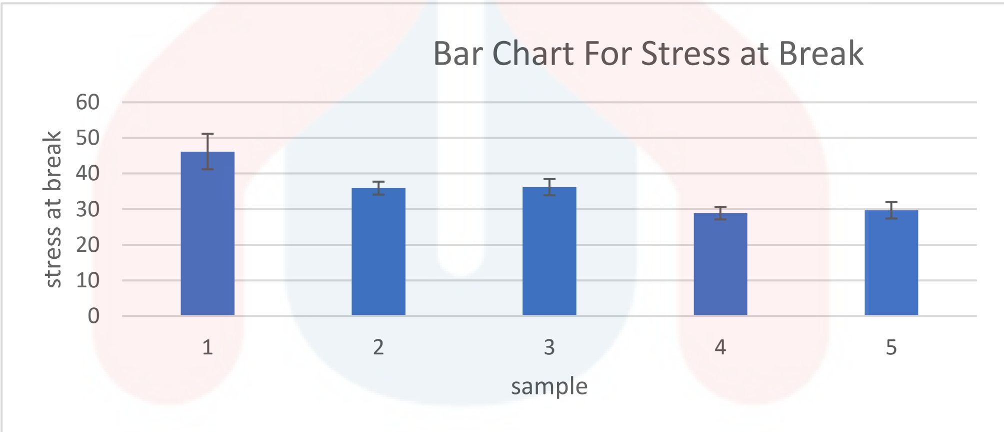


Figure 4.8: Bar chart for stress at break for thin films of PVA 5g (1), PVA CNC 3g (2), PVA CNC PL 0.3g (3), PVA CNC PL 0.6g (4) and PVA CNC PL 0.9g (5).

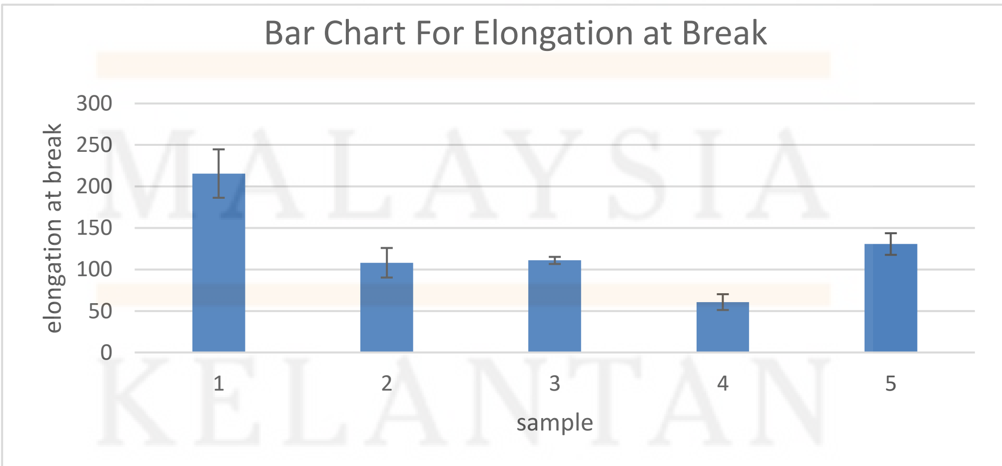


Figure 4.9: Bar chart for elongation at break for thin films of PVA 5g (1), PVA CNC 3g (2), PVA CNC PL 0.3g (3), PVA CNC PL 0.6g (4) and PVA CNC PL 0.9g (5)

From the result that has been shown at figure 8, figure 9, and figure 10, it shows that the comparison of Modulus Young, Stress at break, and Elongatio at break of 5 different composition of thin film. For Young's Modulus, we can see that 3 thin film compositions containing Polylysine powder get the highest results, which are 1352.95 MPa for Polylysine 0.3g, 1594.72 MPa for Polylysine 0.6g, and 1016.27 MPa for Polylysine 0.9g. From this result, we will know that the thin film that contains high Modulus Young have high stiffness compared of PVA and PVA/ CNC thin film.

After that, for stress at break, we can see that PVA thin film have high point which is 46.18 MPa compared than PVA CNC 3g thin film, PVA CNC PL 0.3g, PVA CNC PL 0.6g, and PVA CNC PL 0.9g thin film. From that we will know that the strength at failure of PVA is high compared to others thin film.

Next, for elongation at break, it shown that PVA thin film has more point which is 215.56 MPa compared of PVA CNC 3g, PVA CNC PL 0.3g, PVA CNC PL 0.6g, and PVA CNC PL 0.9g thin film. From this result, we can see that the ductility or flexibility of PVA 5g thin film is high compared to others thin film.

4.4 Fresh Food Testing

4.4.1 Brix Analyzer

4.4.1.1 Brix Analyzer at T0

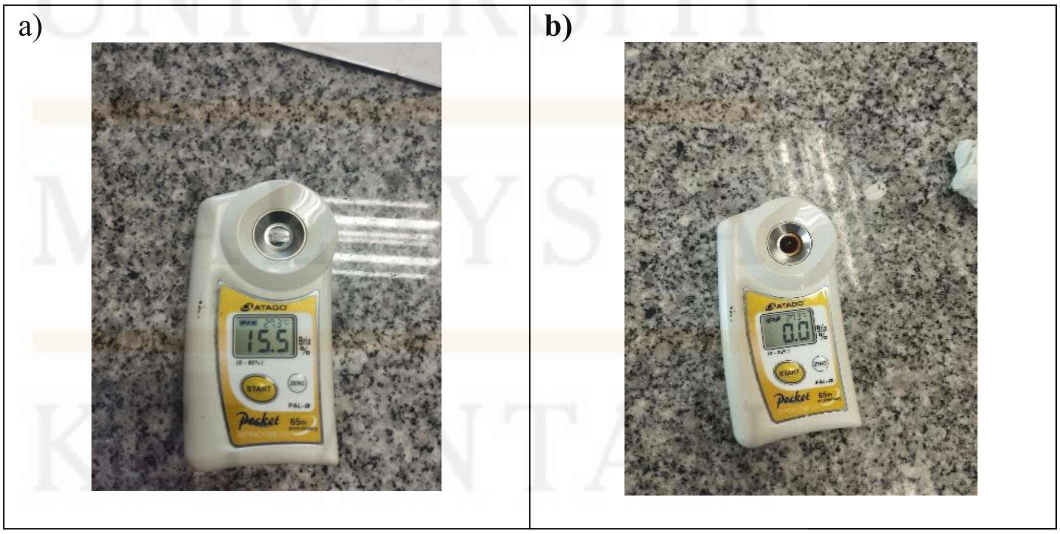


Figure4.10: It shown that a) brix analyzer for sample fresh chili, while for b) brix analyzer for mineral water

4.4.1.2 Brix Analyzer at T20

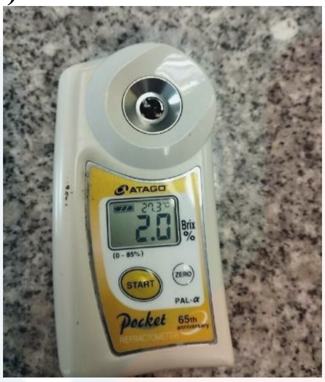
a)	b)	c)
		

Figure4.11: a) shown brix analyser for PVA CNC PL 0.9g thin film of chili, b) shown that PVA CNC PL 0.6g thin film of chili,

Figure 4.12: c) shown that PVA CNC PL 0.3g thin film of chili, d) shown that PVA CNC 3g thin film of chili, and d) shown that PVA 5g thin film of chili.

From the result, we can see that PVA/CNC/PL 0.9g have high point of chili for brix analyzer which is 2.6% compared PVA 5g (0.0%), PVA CNC 3g (0.7%), PVA CNC PL 0.3g(1.2%), PVA CNC PL 0.6g(2.0%). It is because at high composition of polylysine in the thin film, it will avoid decreasing the contain of sugar in chili and keep it fresh for a long time until last day of the experiment. The presence of Polylysine also give the protection to thin film from bacteria from outside layer to attach to the fresh chili because Polylysine have characteristic such as antimicrobial and extend the shelf life in the thin film as a packaging food application.

4.4.2 Texture Analysis

4.4.2.1 Hardness Test

	HARDNESS TEST (g)				
	SAMPLE 1	SAMPLE 2	SAMPLE 3	SAMPLE 4	SAMPLE 5
	1146	895	881	1005	1116
	1359	942	1153	861	989
	1263	735	726	1294	1124
	1247	609	1036	1619	786
	1182	749	1132	1621	1304
	1243	1182	905	1502	1021
MEANS	1240	852	972.1667	1317	1056.667
STD	73.41389514	201.095002	164.7682	323.6214	172.2436

Table 4.3: hardness of chili before the fresh chili has been wrapped in 5 different of thin film

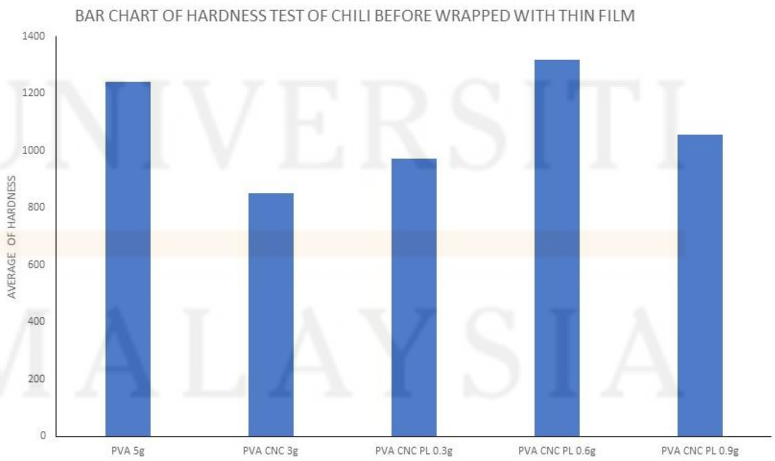


Figure 4.13: Bar chart of average hardness of fresh chili before wrapped in 5 different thin films

As we know from the result, we can see the hardness of fresh chili for 5 different samples were in at high trend. It is because the condition of the chili was still in fresh condition before we ran the experiment packaging the fresh chili into 5 different nanocomposite thin film. From the test, the hardness test shows that the water content in fresh chili is in high composition to make sure the fresh chili does not contribute to dehydration process. The hardness test also showed the point 500g above for different 5 samples of fresh chili.

Furthermore, the springiness and cohesiveness of fresh chili on the first day are also at high point which are above 3 mm and 0.5. It is shown that the shape of chili is of maximum size to be matured. So, based on the result on the first day, we know that all the chili samples were fresh and suitable for our experiment.

HARDNESS TEST (g)

	SAMPLE 1	SAMPLE 2	SAMPLE 3	SAMPLE4	SAMPLE 5
	1223	1302	1326	1126	1126
	1100	1072	1176	943	974
	839	898	783	876	1356
	413	745	349	560	1201
	727	301	1201	1011	1212
	301	339	984	786	1066
MEANS	767.1666667	776.1667	969.8333	883.6667	1155.833
STD	365.535452	399.1819	358.7029	196.3758	132.1248

Table 4.4: hardness of chili after the fresh chili has been wrapped for 20 days from each thin film

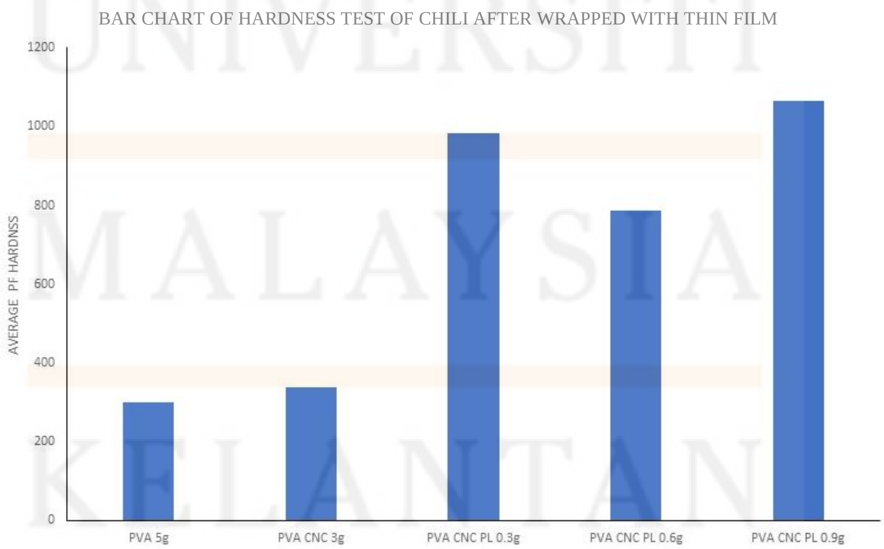


Figure 4.14: Bar chart of average hardness of fresh chili after wrapped for 20 days from each thin film.

After 20 days we packed the chili in 5 different thin film of nanocomposite, we can see that from the result that shown the hardness test for the chili. Based on the average hardness of fresh chili, it showed the hardness of chili that was packed in PVA thin film was decrease compared to bar chart of average of fresh chili from others thin film. It is because the hardness of chili in thin film that have PVA only can easily dehydrated because it cannot avoid microorganism, which can grow up outside layer of chili surface. Compared to thin film that have PVA CNC and ε-Polylysine that can avoid the bacteria or microorganism to grow up in the thin film. It is because ε-Polylysine powder has a broad spectrum of antimicrobial activity against bacteria, yeasts, and molds, and is used as a food preservative. It also can extend the shelf life of chili to keep it still fresh for a long time until the last day of experiment. So, from that the chili in the thin film that contain high composition of ε-Polylysine, have higher point of hardness surface of chili compared to thin film that have contain PVA only.

Other than that, the springiness and cohesiveness of chili in the thin film that contain high composition of ε-Polylysine, have more high point for the freshness test of chili compared to other thin film. It keeps the shape of chili, so it does not rupture during deformation process. So that it will keep the shape of chili always at maximum size and keep it fresh until the last day of experiment.

4.4.2.2 Springiness and Cohesiveness of Chili before Wrap with Thin Film

	SPRINGINESS (mm)				
	SAMPLE 1	SAMPLE 2	SAMPLE 3	SAMPLE 4	SAMPLE 5
	5.16	4.04	3.56	5.06	5.15
	3.97	4.17	4.76	4.05	4.32
	4.86	4.21	4.65	4.14	3.23
MEANS	4.6633333	4.14	4.323333	4.416667	4.233333
STD	0.6188969	0.088882	0.66335	0.558957	0.96293

Table 4.5: Springiness of fresh chili before has been wrapped with thin film.

	COHESIVENESS				
	SAMPLE 1	SAMPLE 2	SAMPLE 3	SAMPLE 4	SAMPLE 5
	1.16	1.08	1.28	0.69	1.32
	0.01	0.95	1.27	1.21	1.24
	0.82	2	0.75	1.11	0.98
MEANS	0.663333333	1.34333333	1.1	1.003333	1.18
STD	0.590790431	0.57239264	0.30315	0.275923	0.177764

Table 4.6: Cohesiveness of fresh chili before has been wrapped with thin films.

4.4.2.3 Springiness and Cohesiveness of Chili after Wrap with Thin Film

	SPRINGINESS (mm)				
	SAMPLE 1	SAMPLE 2	SAMPLE 3	SAMPLE4	SAMPLE 5
	4.3	4.5	4.51	4.52	4.78
	3.84	4.72	4.32	4.07	4.56
	3.25	4.51	4.01	3.83	4.39
MEANS	3.796666667	4.576667	4.28	4.14	4.576667
STD	0.526339561	0.124231	0.252389	0.350286	0.195533

Table 4.7: Springiness of fresh chili after has been wrapped with thin films.

	COHESIVENESS				
	SAMPLE 1	SAMPLE 2	SAMPLE 3	SAMPLE4	SAMPLE 5
	0.9	1.2	1.32	1.43	1.51
	0.44	0.21	0.56	0.78	0.89
	0.31	0.45	0.73	0.63	0.75
MEANS	0.55	0.62	0.87	0.946667	1.05
STD	0.31	0.51643	0.398873	0.425245	0.404475

Table 4.8: cohesiveness of fresh chili after has been wrapped with thin films.

As we know from the result, from figure 4.13, we can see the hardness of fresh chili for 5 different samples were in at high trend. It is because the condition of the chili was still in fresh condition before we ran the experiment packaging the fresh chili into 5 different nanocomposite thin film. From the test, the hardness test shows that the water content in fresh chili is in high composition to make sure the fresh chili does not contribute to dehydration process. The hardness test also showed the point 500g above for different 5 samples of fresh chili.

Furthermore, the springiness and cohesiveness of fresh chili on the first day are also at high point which are above 3 mm and 0.5. It is shown that the shape of chili is in maximum size to be matured. So, based on the result on the first day, we know that all the chili samples were fresh and suitable for our experiment.

After 20 days we packed the chili in 5 different thin films of nanocomposite, we can see that from the result that shown on figure 4.14, the hardens test for the chili. Based on figure 10, it showed the hardness of chili that was packed in PVA thin film was decreased compared to figure other thin film. It is because the hardness of chili in thin film that have PVA only can easily dehydrated because it cannot avoid microorganism, which can grow up outside layer of chili surface. Compared to thin film that have PVA CNC and ε-Polylysine that can avoid the bacteria or microorganism to grow up in the thin film. It is because ε-Polylysine powder has a broad spectrum of antimicrobial activity against bacteria, yeasts, and molds, and is used as a food preservative. It also can extend the shelf life of chili to keep it still fresh for a long time until the last day of experiment. So, from that the chili in the thin film that contain high composition of ε-Polylysine, have higher point of hardness surface of chili compared to thin film that have contain PVA only.

Other than that, the springiness and cohesiveness of chili in the thin film that contain high composition of ε-Polylysine, have more high point for the freshness test of chili compared to other thin film. It keeps the shape of chili to not ruptured during deformation process. So that it will keep the shape of chili always at maximum size and keep it fresh until last day of experiment.

4.5 Weight Loss

PVA/CNC/PL (0.3%, 0.6% & 0.9%)		PVA 5g	PVA/ CNC 3g	PVA / CNC / PL 0.3g	PVA / CNC / PL 0.6g	PVA / CNC / PL 0.9g
Weight chili before wrap(g)		17.01	17.39	18.47	16.66	18.42
After wrap in thin film(g)	**7/12/2023**	16.05	16.47	18.31	16.41	17.98
	12/12/2023	14.74	15.60	18.20	15.98	17.01
	17/12/2023	**14.05**	**14.76**	**16.75**	**14.37**	**16.88**
	22/12/2023	**13.98**	**13.23**	**15.66**	**13.29**	**16.25**
	3/1/2024	**13.03**	**12.60**	**14.77**	**12.79**	**15.99**

Table 4.9: weight loss of fresh chili for 20 days from each thin film.

sample	Sample 1	Sample 2	Sample 3	Sample4	Sample 5
	17.01	**17.39**	**18.47**	**16.66**	**18.42**
	16.05	**16.47**	**18.31**	**16.41**	**17.98**
	14.74	**15.6**	**18.2**	**15.98**	**17.01**
	14.05	**14.76**	**16.75**	**14.37**	**16.88**
	13.98	**13.23**	**15.66**	**13.29**	**16.25**
	13.03	**12.6**	**14.77**	**12.79**	**15.99**
difference	**3.98**	**4.79**	**3.7**	**3.87**	**2.43**
divide	**0.233980012**	**0.27544566**	**0.20032485**	**0.23229292**	**0.13192182**
percentage %	**23.39800118**	**27.5445658**	**20.0324851**	**23.2292917**	**13.1921824**

Table 4.10: Difference of weight loss for fresh chili that has been wrapped for 20 days from each thin film.

SAMPLE	PERCENTAGE
PVA 5g	**23.40%**
PVA/ CNC 3g	**27.50%**
PVA / CNC / PL 0.3g	**20.00%**

PVA / CNC / PL 0.6g	**23.20%**
PVA / CNC / PL 0.9g	**13.20%**

Table 4.11: Percentage of weight loss for fresh chili that has been wrapped for 20 days from each thin film.

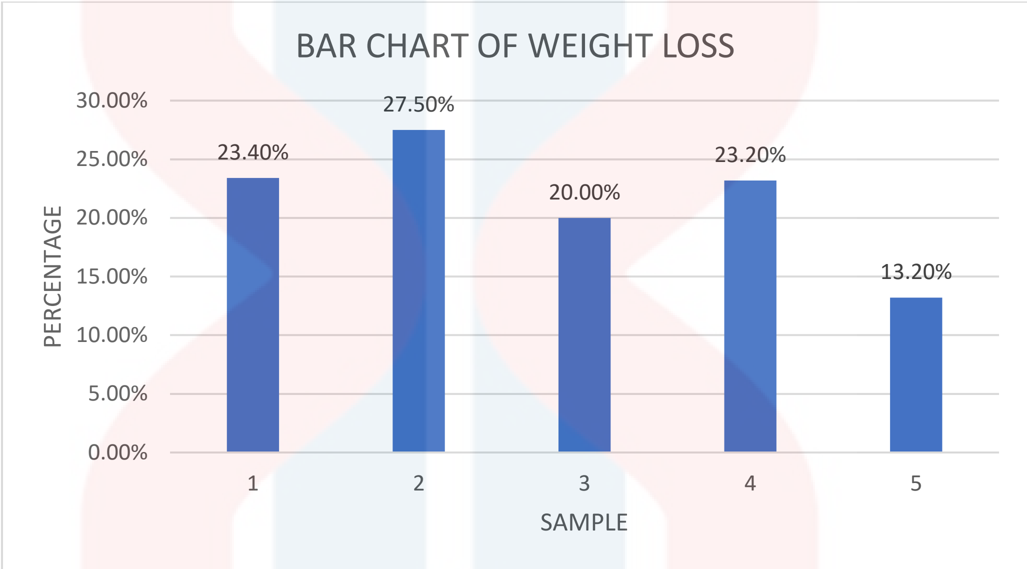


Figure 4.15: Bar chat of percentage weight loss of fresh chili for each thin film which are PVA 5g (1), PVA CNC 3g (2), PVA CNC PL 0.3g(3), PVA CNC PL 0.6g(4), and PVA CNC PL 0.9g(5).

From the result of Table A, we will know that the difference weight loss from T0 of we test to wrap the fresh chili of 5 difference composition of thin film which is PVA 5g thin film, PVA CNC 3g thin film, PVA CNC PL 0.3g thin film, PVA CNC PL 0.6g thin film, and PVA CNC PL 0.9g thin film. It is because we take the data of weight loss for 3 days until last day, T final. From that, we will know that PVA CNC PL 0.9g of thin film get less point in differentiate of weight loss data from Table A which is 2.43, while other thin film lika PVA 5g get 3.98, PVA CNC 3g get 4.79, PVA CNC PL 0.3g get 3.70 and PVA CNC PL 0.6g get 3.87. So, from this result, we can calculate the percentage of weight loss for fresh chili that has been wrapped in 5 different compositions of thin film.

As the graph bar of percentage weight loss of fresh chili, we can see that PVA CNC PL 0.9g of thin film get lower percentage of weight loss which is 13.20% compared to others 4 thin film. It is because this thin film contains the highest ε-Polylysine which is 0.9g. As we know that ε-Polylysine can help to extend the shelf life of food in the thin film application. ε-Polylysine is a natural antimicrobial agent that is commonly used in various industries, including the food industry. It works by disrupting the integrity of microbial cell membranes and interfering with their essential metabolic processes. This disruption leads to the inhibition of microbial growth and, in some cases, the death of the microorganisms. (Liang Wang, 2021). It works by interacting and binds with the negatively charged microbial cell membrane and forms holes in membrane. After that, ε-Polylysine will enter the cell through the pores in the cell membrane, affects the structure and function of the cell membrane and ultimately leads to the death of bacterial cells.

So, from this analysis, we can conclude that thin film that contains more ε-Polylysine powder in the process to make it as a thin film can get less percentage of weigth loss for fresh chili after wrapped for 20 days.

4.6 pH Testing

4.6.1 pH Testing of Fresh Chili at T0

a)

b)

Figure 4.16: shown that pH testing a) pH of chili, b) pH of mineral water

Terms	Result
pH of chili	6.6
pH of mineral water	7.5

Table 4.12: pH testing for first batch of fresh chili and mineral water

4.6.2 pH Testing of Fresh Chili at T20

a)

b)

c)

Figure 4.17: shown that pH testing a) PVA 5g thin film of chili,b) PVA CNC 3g thin film of chili

d)

e)

Figure 12: shown that pH testing c) shown that PVA CNC PL 0.3g thin film of chili,

d) shown that PVA CNC PL 0.6g thin film of chili, e) shown that PVA CNC PL 0.9g thin film of chili.

From the result of pH testing, we can see that pH of fresh chili in PVA CNC PL 0.9g get the highest point which is 7.8 compared to other fresh chili in 4 difference thin film which are PVA 5g, PVA CNC 3g, PVA CNC PL 0.3g and PVA CNC PL 0.6g. It is because this thin film has high composition of Polylysine. This composition helps to extend the shelf life of fresh chili in the thin film of PVA CNC PL 0.9g.

When PVA CNC PL 0.9g thin film is applied to fresh chili peppers, the two materials can interact with each other. The acidity of the chili peppers can cause the PVA to degrade, which can lead to the film losing its effectiveness. To prevent this from happening, it is important to adjust the pH of the chili peppers before applying the thin film.

From this result, the chili from PVA CNC PL 0.9g thin film will always be fresh until the last day of wrapping the fresh chili into the thin film.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

As a conclusion for the thesis, the fabrication of PVA/CNC/ε-Polylysine nanocomposite thin films by solvent casting method has made it possible to fully comprehend their mechanical and structural properties, which has helped achieve the stated goals. The effective combination of ε-Polylysine, cellulose nanocrystals (CNC), and polyvinyl alcohol (PVA) in the nanocomposite has produced films with special qualities appropriate for possible uses in the food packaging industry.

The characterization procedure has illuminated the modulus, tensile strength, and other pertinent characteristics of the nanocomposite films, offering crucial information for assessing their efficacy and appropriateness for real-world uses. Promising results have been found from a thorough examination into the possibility of these biodegradable sheets for packaging fresh chilli. The nanocomposite films exhibit characteristics that are in line with those needed for efficient food packaging, providing a harmony of biodegradability, flexibility, and strength.

This discovery is important because it adds to the continuing attempts to create environmentally friendly packaging materials. In addition to providing environmentally friendly options, the application of PVA/CNC/ε-Polylysine nanocomposite thin films highlights the possibility of utilizing biobased and biodegradable materials in the food packaging industry. These results offer a strong basis for future developments and applications in the field of green packaging solutions, since environmental sustainability remains a top priority in society.

5.2 Recommendations

Several recommendations are made to direct future research and practical uses of PVA/CNC/ε-Polylysine nanocomposite thin films for fresh chilly packaging considering the goals and results provided in this thesis. First, by adjusting the PVA, CNC, and ε-Polylysine ratios, the nanocomposite film formulation must be optimized. Certain qualities, such mechanical strength and flexibility, might be improved by this optimization, meeting the complex needs of fresh chilly packing. It's also advisable to investigate different processing methods, like electrospinning or extrusion, to see how they affect the properties of the film and whether they have any advantages over solvent casting.

Moreover, a thorough examination of the nanocomposite films' barrier characteristics is advised. This analysis ought to explore how well they protect fresh chilly from outside elements such as light, moisture, and gases. It is essential to comprehend these barrier qualities to make sure the films fulfil or surpass the requirements for food packing.

It is also advised to carry out a life cycle assessment (LCA) to examine the nanocomposite films' environmental impact over the course of their whole life cycle. When compared to traditional packaging materials, the films' sustainability and environmental friendliness would be more understood thanks to this all-encompassing approach.

It is advisable to investigate the nanocomposite films' compatibility with different kinds of fruits, vegetables, or perishable commodities to increase the range of applications. This diversification would help to increase the number of possible uses for them in the food sector. To close the gap between academic research and real-world application, industry stakeholders such as food and packaging makers must work together.

Furthermore, investigations on consumer perception are necessary to find out how customers view and adopt PVA/CNC/ε-Polylysine nanocomposite packaging. This includes assessing elements that are critical to a successful market adoption, like appearance, safety, and the perceived environmental impact. It is crucial to ensure regulatory compliance. The created nanocomposite films must abide with the applicable legal requirements and food packaging recommendations. This covers the safety, hygiene, and regulatory aspects of using biodegradable materials in applications that meet food.

To sum up, these suggestions work together to further the study and use of PVA/CNC/ε-Polylysine nanocomposite thin films. They also offer a path forward for future advancements in efficient and sustainable food packaging, especially regarding fresh chilly.

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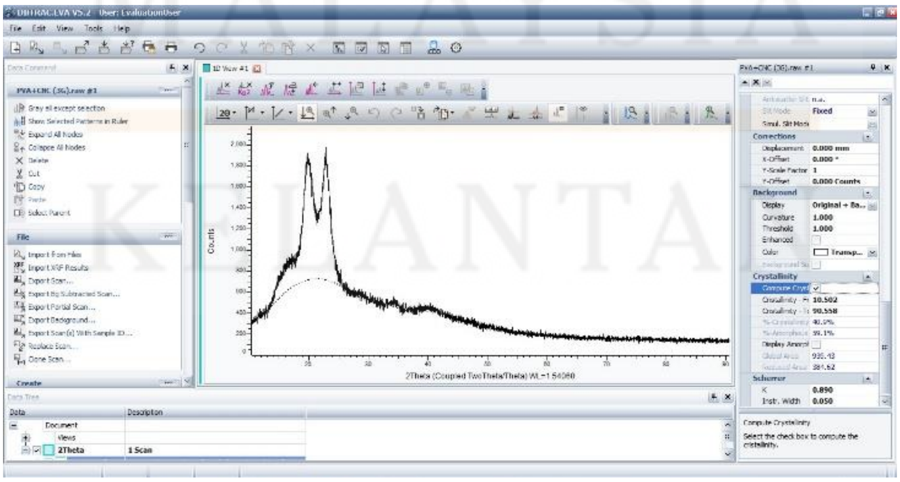
APPENDICES

			
Beaker	Petri Dish	**PVC Powder**	ε-Polylysine powder

MACHINE OF TECHNIQUE

	
TGA Machine	D2 Phase XRD

XRD IMAGE



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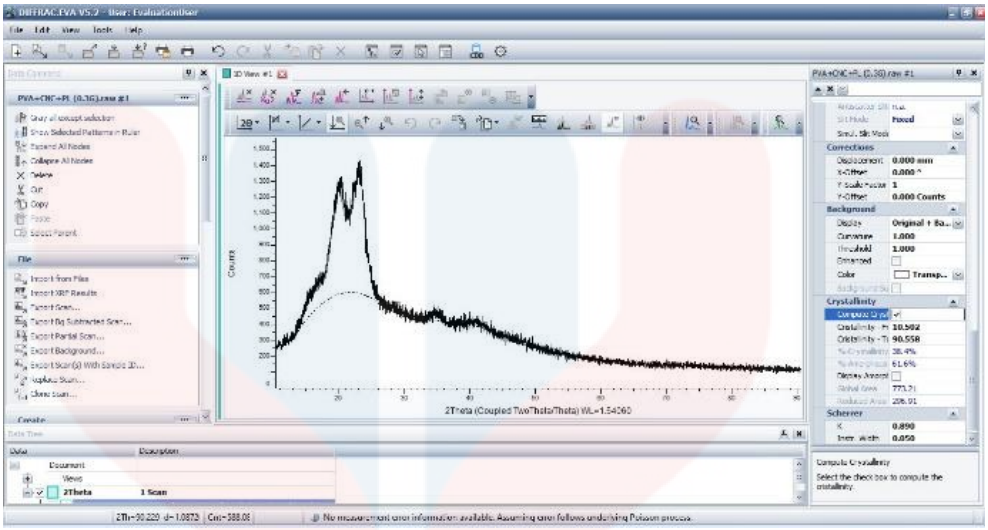

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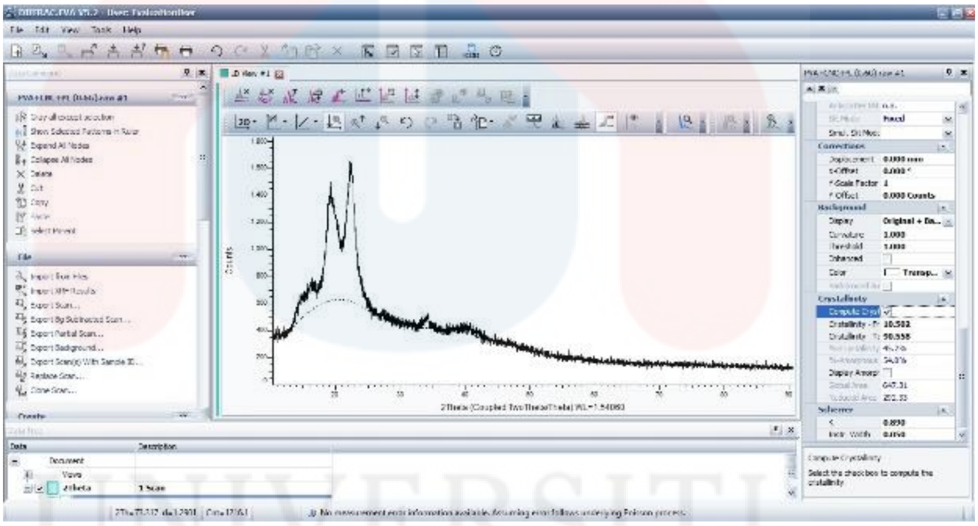

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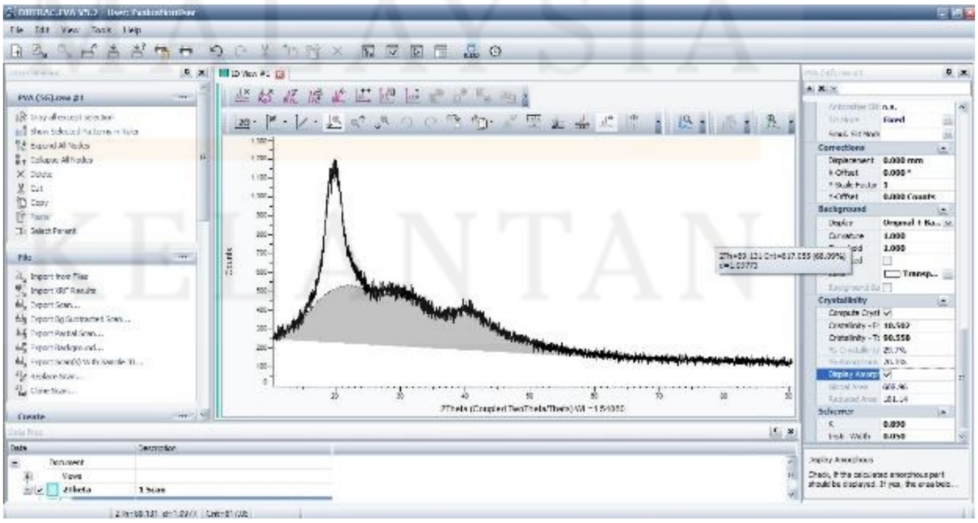

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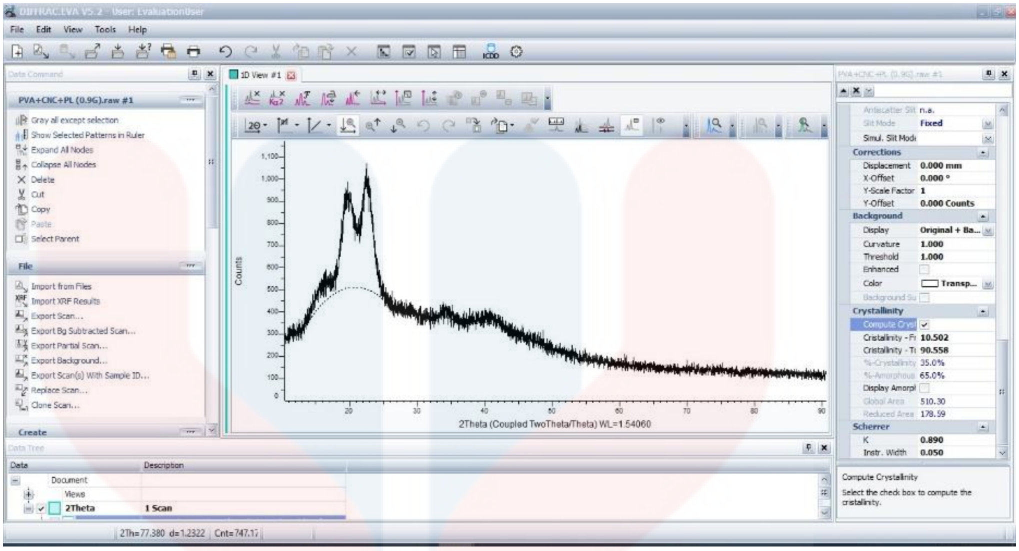

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