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EVALUATION OF PHYSICOCHEMICAL AND THERMAL PROPERTIES OF PECTIN AND STARCH AEROGELS AND THEIR ABSORPTION CAPACITY OF METHYLENE BLUE

WAN FARAHIYAH IFFAH BINTI TUN SULONG
J20A0703

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FACULTY OF BIOENGINEERING AND TECHNOLOGY
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DECLARATION

I declare that this thesis entitled "Evaluation of Physicochemical and Thermal Properties of Pectin and Starch and Their Absorption Capacity of Methylene Blue" is the results of my own research except as cited in the references.

Signature :
Student's Name : WAN FARAHIYAH IFFAH BINTI TUN SULONG
Date : 7 JANUARY 2024

Verified by:

Signature : ____________________________
Supervisor's Name : DR. ASANAH BINTI RADHI

Stamp : ____________________________
Date : ____________________________

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ABSTRAK

Dalam kajian ini, potensi bio aerogel yang diperbuat daripada kanji dan pektin dalam rawatan air sisa telah dikaji. Kesan penggabungan graphene oxide ke dalam bio airgel ini juga telah dikaji. Objektif kajian ini adalah sifat fizikokimia dan haba bagi aerogel kanji dan pektin yang digabungkan dengan graphene oxide (GO) dan kapasiti penjerapan kanji dan aerogel pektin pada penyingkiran metilena biru. Aerogel pektin dan kanji telah dicirikan menggunakan Scanning Electron Microscopy (SEM), Fourier Transform Infrared Analysis (FTIR), dan Thermogravimetric analysis (TGA). Keputusan menunjukkan bahawa menggabungkan GO menghasilkan lebih banyak struktur mikro airgel berliang kerana ikatan hidrogen antara polimer dan kumpulan berfungsi GO. Penambahan GO juga meningkatkan kestabilan terma dan pengambilan metilena biru dengan menyediakan tapak penjerapan yang lebih aktif. Pemuatan GO yang optimum meningkatkan penjerapan pewarna secara sinergistik dengan meningkatkan luas permukaan dan interaksi yang menggalakkan dengan metilena biru. Secara keseluruhan, gabungan pektin/kanji dengan GO menunjukkan janji untuk membangunkan penjerap aerogel berasaskan bio yang mampan dan berkesan untuk penyingkiran pewarna daripada air sisa.

Kata kunci: Aerogel, graphene oksida, kapasiti penyerapan, biru metilena, penyingkiran pewarna

ABSTRACT

In this study, the potential of bio aerogel made from starch and pectin in wastewater treatment has been investigated. The effect of graphene oxide incorporation into this bio aerogel was also studied. The objectives of this study are physicochemical and thermal properties of starch and pectin aerogels incorporated with graphene oxide (GO) and their adsorption capacity of starch and pectin aerogels on methylene blue removal. The pectin and starch aerogels were characterized using Scanning Electron Microscopy (SEM), Fourier Transform Infrared Analysis (FTIR), and Thermogravimetric analysis (TGA). Results showed that incorporating GO produces more porous aerogel microstructures due to hydrogen bonding between the polymers and GO functional groups. GO addition also improved thermal stability and methylene blue uptake by providing more active adsorption sites. The optimal GO loading enhanced dye adsorption synergistically by increasing surface area and favorable interactions with methylene blue.Overall, the combination of pectin/starch with GO shows promise for developing sustainable and effective bio-based aerogel adsorbents for dye removal from wastewater.

Keywords: Aerogels, graphene oxide, absorption capacity, methylene blue, dye removal

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LIST OF ABBREVIATION

GO	Graphene Oxide
SEM	Scanning Electron Microscopy
Uv-Vis	Ultraviolet–visible spectroscopy
CS-R-P	Chitosan-resole-pectin.
FTIR	Fourier Transform Infrared Spectroscopy

LIST OF SYMBOLS

CO_2	Carbon Dioxide
Pb^{2+}	Plumbum
pH	Potential of hydrogen
MPa	Mega Pascal
wt%.	Weight Percentage
g/cm^3	Grams per cubic centimetre
m^2/g	square meters per gram
pKa	Negative log of acid dissociation constant
°C	Degree Celsius
ml	Millilitres
ml/mg	Millilitres per milligram
g	Grams
µL	microliters
cm^{-1}	inverse centimetre

CHAPTER 1

INTRODUCTION

1.0 Background of study

Bio-aerogels are pliable new polysaccharide-based polymers. The latter are already available, renewable, non-toxic, and biocompatible cause s large number of hydroxyl groups on the polymer backbone also be easily functionalized. Aerogels have several advantages over hydrogels as adsorbents due to lightest and most moisture-free properties. Also, easy to carry and store. A single aerogel cannot directly adsorb heavy metals has a limited adsorption capacity. Because of the absence of particular functional groups (Groult et al., 2021).

Aerogels are classified into three categories based on their appearance, monolith, powder, and film. Depending on how it is made, it is known as an aerogel, xerogel and cryogel. Microporous aerogel range between 2-50 and has mixed porous aerogel. Bio-aerogels are suitable for various study field of life science applications. The application determines the chemical, mechanical, and structural characteristics of aerogels. Understanding how formulation and environmental variety of polymer ionisation and concentration, solution pH, ion presence affects aerogel properties (Abdul Moheman al., 2021).

Pectin aerogel is a lightweight, porous substance made from pectin; a natural polysaccharide presents in plant cell walls. Pectin is widely employed in the food sector as a gelling agent, but it also has applications in materials science, such as aerogel manufacture.

Pectin aerogels are commonly made via a sol-gel technique, like that used for chitosan-resole-pectin (CS-R-P) aerogels. This method involves making a sol or gel from a pectin solution, then exchanging solvents and supercritical drying to produce a highly porous, low-density material with a large surface area.(Flores-Gómez et al., 2023)

Due to their biocompatibility and biodegradability, pectin aerogels have demonstrated potential for usage in a variety of applications, including medication delivery systems, adsorbents for environmental cleanup, and components in food packaging and preservation. The application of pectin in aerogel manufacturing provides a sustainable and ecologically friendly way to create functional materials with a wide range of uses.(Flores-Gómez et al., 2023)

Starch aerogel is an aerogel material derived from starch, a natural and renewable polymer. It is a highly porous and lightweight material with a three-dimensional network structure, resulting in a large surface area and outstanding adsorption capabilities. Starch aerogels have been investigated for their possible applications in a variety of fields, including water treatment, environmental remediation, and energy storage. They are regarded as a promising alternative to conventional adsorbents due to their biodegradability, low cost, and abundance.(Khoo et al., 2023)

Aerogels have multiple features that make them suitable for a variety of applications, including water treatment and environmental remediation. Starch aerogels have several critical features, including high porosity. Aerogels have a highly porous structure, giving them a wide surface area for adsorption of pollutants from water and other fluids. Since aerogels are lightweight materials, it makes them easy to handle and excellent for applications requiring weight control. Many aerogels, notably starch aerogels, are biodegradable, providing an environmentally acceptable alternative to water treatment techniques.(Khoo et al., 2023)

Starch and pectin aerogels, made from natural starch which renewable and abundant, helping to ensure the sustainability of water treatment procedures. Aerogels can be changed and functionalized to improve their adsorption capabilities and selectivity for certain impurities, making them suitable for a variety of water treatment applications. Aerogels in water treatment have the potential to reduce waste generation when compared to typical treatment processes, hence contributing to more sustainable practices. This make pectin and starch aerogel is one of the excellent materials for water treatment.(Khoo et al., 2023)

The introduction of nanoparticles into aerogels has been proven to improve their absorption capacity. Since nanomaterials is nanoscale dimensions, it makes them have unique properties such as nanomaterials have a large surface area compared to volume, which can contribute to improved reactivity and absorption capacity. Improved mechanical characteristics. (Chen et al., 2023)

Graphene oxide is one of the suggested nanomaterials to incorporate with aerogel. This is because adding graphene oxide (GO) to aerogels has been found to improve their absorption capacity. When coupled with other materials, GO's vast surface area and good conductivity can provide a synergistic effect that results in increased mechanical properties and higher sensing sensitivity. Furthermore, the usage of GO in composite aerogels has been shown to alter the microstructure of the aerogel backbone, resulting in increased elasticity, toughness, and long-term durability following multiple compression cycles. As a result, including graphene oxide into aerogels shows potential for increasing their absorption capacity and overall performance. (Chen et al., 2023)

Graphene oxide (GO) is a graphene derivative that is a single layer of hexagonally arranged carbon atoms. GO is just graphene with the basal plane and edges chemically altered by the addition of oxygen-containing functional groups. Its composition consists of carbon, oxygen, and hydrogen atoms. Individual GO layers are made up of a single graphene sheet with oxygen functional groups attached to its basal plane and edges. GO is hydrophilic due to the presence of hydrophilic oxygen functional groups, which means it is easily soluble in water and other polar solvents. The mechanical properties of GO are poorer than those of pristine graphene due to the presence of defects and functional groups (Pinelli et al., 2021).

1.1 Problem statement

Synthetic dyes have a substantial impact on water contamination. These dyes are recalcitrant in aerobic environments, so they accumulate in sediments and soil. Once discharged into the aquatic environment, heavy metal cations in these dyes can be digested by fish gills and accumulate in specific tissues, endangering aquatic creatures and potentially reaching human organs via the food chain, causing a variety of ailments. The colour associated with textile dyes not only causes aesthetic damage to water bodies, but it also inhibits light penetration through water, altering photosynthesis and dissolved oxygen levels, so hurting the entire aquatic ecosystem. Moreover, textile dyes are poisonous, mutagenic, and carcinogenic agents that stay in the environment and can traverse entire food chains, resulting in biomagnification.(Lellis et al., 2019)

Bio aerogels and aerogels have potential use for water and remove dyes. They have a large surface area and porosity, making them efficient at adsorbing pollutants from water. Aerogels, in specifically, have been studied for their ability to remove colours from wastewater due to their large surface area and unique characteristics. These materials can effectively adsorb dye molecules, helping to remediate dye-contaminated water. Moreover, bio aerogels and aerogels are biodegradable which is safe to use without harmed the environment.(Lellis et al., 2019)

The incorporation of graphene oxide (GO) into bio aerogel for dye removal has various advantages. First, GO has a wide surface area and a high concentration of functional groups, which improves its ability to adsorb dyes and other pollutants. This improves dye effectiveness in removing from wastewater. Furthermore, GO has high mechanical strength and stability, which can improve the structural integrity of the bio aerogel and make it more suitable for practical applications. (Muhammad et al., 2023)

Additionally, the presence of GO in the bio aerogel can improve its dispersibility and stability in water, solving the issue of limited dispersion caused by activated carbon. The addition of GO into bio aerogel increases the materials' potential for wastewater treatment applications by enhancing their adsorption capacity, mechanical characteristics, and water stability. This modification improves the bio aerogel's ability to remove colours and other impurities from wastewater, boosting its usefulness and efficiency in water treatment procedures.(Muhammad et al., 2023)

Overall, GO/biopolymer-based nanocomposites are expected to present a considerable opportunity for academic and industry researchers, as well as governments for absorbent for heavy metal removal (Elella, 2023).

1.2 Objectives

The objectives of this study are:

I. To prepare starch and pectin aerogels incorporated with graphene oxide (GO) nanoparticles.
II. To characterize the morphology, physicochemical and thermal properties and their adsorption capacity of starch and pectin aerogels on methylene blue removal

1.3 Scope of study

The scope of this study is to study the evaluation of physicochemical and thermal properties of pectin and starch and their absorption capacity of methylene blue. Pectin and tapioca starch aerogel were use as a bio aerogel and incorporated with nanomaterials which is graphene oxide. 12 sample were prepared which are pure pectin and starch 0, 0.2, 0.4, 0.6, 0.8 and 1.0 mg/ml GO loaded in both pectin and starch sample. Then, Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR) and Ultraviolet–visible spectroscopy (UV-VIS) was used to analyse. For SEM tested to identify the microstructure of tapioca starch and pectin while FTIR to identify the functional groups that present in the sample and lastly UV-VIS to identify the absorption capacity of methylene blue by the samples.

1.4 Significance of study

The research of bio-aerogels is extremely important in the field of science and materials. For instance, water treatment. The incorporation of GO nanolayers into biopolymer chains has shown great promise and potential as an absorbent for heavy metal removal nanocomposite materials with exceptional mechanical and thermal stability, antimicrobial activity, excellent water resistance, and UV-shielding properties. Because of its porous structure, strong adsorption capabilities, and ease of modification, graphene-based aerogels are commonly used to remove heavy metal ions from aqueous solutions.

CHAPTER 2

LITERATURE REVIEW

2.1 Pectin

Pectin is a naturally generated polysaccharide that exists in plant cell walls, especially in fruits like apples, citrus fruits, and berries. It is a complex heteropolysaccharide made up primarily of partly methylated poly galacturonic acid units. The density of pectin aerogels varied from 0.05 to 0.15 g/cm^3, and the specific surface area ranged from 270 to 600 m^2/g, demonstrating the ability to alter these properties depending on the synthesis circumstances. Pectin is commonly used in the food business as a gelling agent, thickener, and stabiliser in a wide range of goods such as jams, jellies, and confectionery. (Groult et al., 2021).

Pectin is an anionic polymer derived from starch and cellulose that occurs naturally. Pectin has functional carboxyl groups and a high capacity for heavy metal removal. Pectin has excellent properties such as exceptional biological compatibility, biodegradability, and abundance. Recent study showed, the pectin and pectin-based hydrogels were developed for the adsorption of $Pb^{2+}$from wastewater (Wang et al., 2021).

Pectin is a structural polysaccharide found in many higher plant cells. It might be isolated and used as an anionic biopolymer such as water soluble. A slew of recent articles has emphasised the benefits of employing pectin over traditional polymers. Lastly, pectin is become increasingly important in food packaging applications. Due to gelling agents, colloidal stabilisers, texturizers, emulsifiers, coatings on fresh also cut fruits and vegetables. Other than that, micro and nano-encapsulating agents for the controlled release of active principles with various functionalities (Groult et al., 2021).

Parameters will influence the gelation of pectin solutions that is pectin content, temperature, pH, and metal ion concentration. Physical gelation can occur at room temperature below pectin's pKa (3.0-3.5) and depends on polymer concentration owing to hydrogen bonds between hydroxyls. In protonated carboxyl groups, hydrophobic able to interact between methyl ester groups. At the end, enables for the creation of a diverse spectrum of aerogel shapes and properties.

2.2 Pectin Aerogel

Supercritical dissolution solvent exchange drying CO_2 was used to create highly porous and nanostructured pectin aerogels. The purpose was to demonstrate why and how aerogel form change in response to external factors and characteristics. The concentrations of polymers, pH, and divalent metal salts also changed. Structure stability of gelation process or direct non-solvent driven phase separation and solvent exchange gel, or solution. This process is one of the primary criteria influencing aerogel shape and density (Groult et al., 2021).

Pectin aerogels created from polymer dissolved in water this is means the exchange of water to a fluid miscible and supercritical dry with CO_2. Besides, we focused at the relationship between pectin aerogel thermal conductivity and shape. The current study investigates how and why aerogel structure and characteristics change because of external inputs. The latter are thoroughly studied by varying pectin content, solution pH, metal salt type and concentration of $CaCl_2$ and NaCl, and non-solvent nature (Groult et al., 2021).

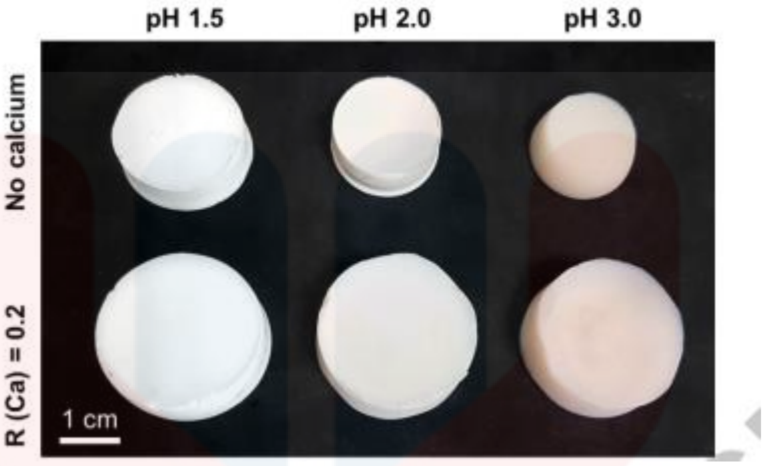

Figure 2.1: Photos of pectin aerogels were made from 3 wt% solutions at pH 1.5, 2.0, and 3.0 (Groult et al., 2021)

Images of pectin aerogels made from 3 wt.% solutions at pH 1.5, 2.0, and 3.0, without salts, and with calcium at R(Ca) = 0.2. Prior to solvent exchange, the sample was a weak acid gel at pH 1.5, a high viscosity solution at pH 2.0, and a low viscosity solution at pH 3.0 (without calcium). When R(Ca) = 0.2, strong ionic polymers formed at all pH values. Alcohol wasn't a solvent.

The development network an aerogel precursor has been studied using solution gelatin. This research will investigate how to control the structure of formation in pectin-based aerogels and possibly. After, gelling polyelectrolyte polysaccharides such as alginates and carrageenan’s, how multiscale linkages between dissolved polymer characteristics also to analyse the final structure and properties of pectin aerogels are emphasised.

2.3 Tapioca Starch

Tapioca starch, also called cassava starch, is a form of starch derived from cassava root (Manihot esculenta). It is a fine, powdered substance that is extensively employed as a thickening, stabilising, and texturizing agent in a variety of food and industrial applications. Tapioca starch is prized for its neutral flavour, high purity, and varied functional qualities, making it a popular ingredient in a variety of goods. The extraction process includes washing, peeling, grating, and separating the starch from the fibrous residue of the cassava root. After extraction, the starch is dried and ground to a fine powder. Tapioca starch can be modified further using physical, chemical, enzymatic, and genetic approaches to improve its characteristics for specific uses.(Breuninger et al., 2009)

Tapioca starch is used in a variety of food products, including soups, sauces, baked goods, and desserts, as well as a crucial ingredient in the manufacturing of tapioca pearls for bubble tea and other beverages. Its neutral flavour and hypoallergenic qualities make it suitable for use in gluten-free and allergen-free food compositions. Tapioca starch is used in paper manufacture, corrugated adhesives, sizing, and coating preparations, and as a component in modified starch products. Its distinct properties, such as low residual materials and high molecular weight components, make it ideal for a variety of industrial operations.(Breuninger et al., 2009)

2.4 Graphene Oxide

Graphene oxide (GO) is a graphene derivative, where two-dimensional material composed of a single sheet of organised carbon atoms in a hexagonal lattice. Chemically altered its basal plane and edges. The inclusion of these functional groups' renders GO hydrophilic, which means it dissolves easily in water and other polar solvents whereas pristine graphene is hydrophobic. GO has are different physical and chemical properties than pure graphene it is valuable in a various application (Elella, 2023).

These functional groups make GO more reactive than pristine graphene and allow it to undergo numerous chemical reactions such as reduction, oxidation, and functionalization. GO has potential uses in a wide in sectors. For example, electronics, energy storage, water treatment, biomedicine, catalysis, coatings, composites, and sensors. GO its unique features cause large surface area, electrical conductivity, and optical qualities (Elella, 2023).

2.5 Structure of Graphene Oxide

Graphene Oxide (GO) with oxygen groups (carboxyl, hydroxyl, and epoxy groups) may form complex with organic contaminants and metal ions by electrostatic contact, hydrogen bonding, and coordination which is suitable for prospective use in environmental pollution, clean-up, and control. In general, graphite was oxidised in acidic environments with different oxidants to produce GO. These oxygen-containing functional groups can provide an abundance of adsorption sites for radionuclide removal. Furthermore, the oxygen functional groups in GO rendered it more hydrophilic than graphene, which may lead to the development of stable complexes between GO and pollutants. Numerous research publications have focused on the removal of pollutants by GO, including tiny molecules, heavy metal ions, radionuclides, and organic compounds (Lu et al., 2019).

In comparison to low-cost adsorbents for example oxides and clay minerals. GO-based materials have a low density, a high surface area, and numerous surface functional groups. GO-based materials have great contaminant adsorption capability. Because graphene oxide (GO) strong mechanical strength, and oxygen-containing functional groups. GO also high potential for applications that involve changing its surface structures, such as by attaching functional peptides, antibodies, and enzymes (Lu et al., 2019).

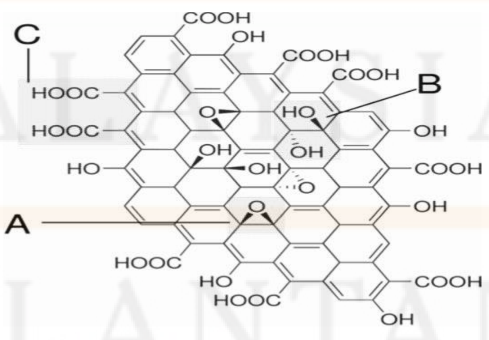

Figure 2.2: Structure of graphene oxide with functional groups, A: Epoxy bridges, B: hydroxy groups, C: pairwise carboxy groups.(Krasilnikov et al., 2005)

2.6 Properties of pectin and starch

2.6.1 Physical Properties

Pectin is biodegradability and biocompatibility. This is because they are generated from natural polysaccharides. Suitable for various application. One of it is used in food packaging application. It also water absorption capacity. Pectin aerogels potentially to absorb and retain large volumes of water. The porosity, degree of esterification, and presence of hydrophilic groups within the pectin structure all affect water absorption capacity. This feature makes pectin aerogels appropriate for applications such as humidity management, moisture absorption, and water purification.

The sample morphologies were characterised by SEM, and the results shown the aerogels were typical microporous materials with 3D network topologies. Pectin morphology has differed substantially. A network of pectin according to their mass fraction which is pectin 0.05 was shown a fibrillated and lamellar structure with the fibril structure predominating. As the EGDE content grew, the structure of aerogel became more regular, and made the pores smaller. Pectin 0.1 and pectin 0.02 featured "honeycomb" morphologies, whereas pectin 0.3 had a "sponge" morphology (Wang et al., 2021).

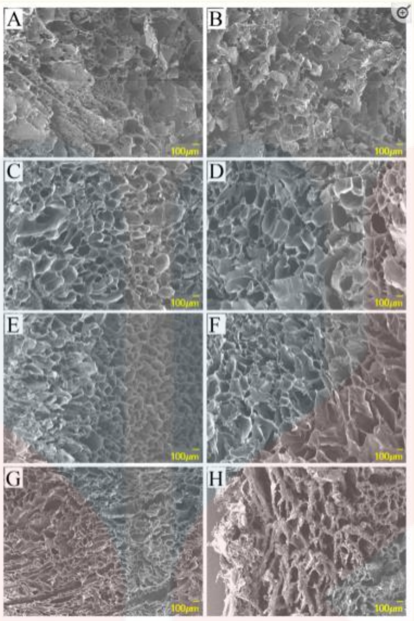

Figure 2.3: SEM images of pectin according to their mass fraction (Wang et al., 2021)

Starch is made from natural polymer with several essential physical properties including the size and form of starch granules vary depending on the botanical source. For example, potato starch granules are large and oval, but maize starch granules are smaller and more angular. Starch is insoluble in cold water, but it can swell and absorb water when heated, resulting in gel formation. This feature is useful in many food and industrial applications. When starch is disseminated in water and heated, it becomes viscous and forms a gel. The viscosity of starch solutions varies according to concentration and temperature.(Khoo et al., 2023)

When starch is heated in the presence of water, it forms gels, which provide thickening and gelling qualities used in both food and non-food applications.

After production, starch gels can undergo retrogradation, a process in which the gel structure reorganises and becomes stiffer with time. Moreover, starch is swelling, it granules can swell and absorb water, causing an increase in volume. The swelling power of starch varies based on the botanical source and processing.(Khoo et al., 2023)

2.6.2 Thermal properties

Aerogels are lightweight nanostructured materials having an open porosity network. Pectin aerogel is a lightweight, porous material created from pectin, a naturally occurring carbohydrate found in fruits and vegetables. Because of its unique structure and characteristics, pectin aerogel offers excellent thermal qualities. Aerogels, particularly pectin aerogels, are well known for their low heat conductivity. Pectin aerogel's porous structure is made up of connected nanoscale particles that trap air molecules within the material. This trapped air acts as an insulator, significantly reducing heat energy transmission (Wang et al., 2021).

Pectin aerogel is a lightweight, low-density material. Because it reduces the amount of solid material through which heat may conduct, its low density contributes to its exceptional thermal insulation capabilities. Resistance to high temperatures. Pectin aerogels can withstand quite high temperatures without deteriorating structurally. The precise thermal stability of pectin aerogel is determined by the manufacturing process and the purity of the starting materials.

For starch including melting point. Starch does not have a definite melting point since it is thermally degraded before melting. The thermal decomposition temperature of starch varies based on the botanical source and processing. It also thermal stability. Starch's heat stability varies depending on the botanical source and processing. Some forms of starch, such as high-amylose starch, have higher heat stability than others. Starch also can be pyrolyzed, which is the process of decomposing it with heat in the absence of oxygen. This procedure can be used to produce biofuels and other sustainable energy sources.(Khoo et al., 2023)

Moreover, starch can form a gel. When heated, starch gelatinizes and forms viscous solutions or gels, which is used in water treatment procedures including flocculation and sedimentation. Starch-based flocculants can aid in the removal of suspended particles and pollutants from water by causing them to aggregate and settle. Starch is also biodegradability. The heat stability and breakdown behaviour of starch are critical concerns in the development of biodegradable water treatment technologies. Starch-based polymers with certain thermal properties can be customised for use as biodegradable adsorbents and filtration media, helping to promote sustainable water treatment methods.(Khoo et al., 2023)

2.6.3 Mechanical Properties

The mechanical properties of pectin aerogels are significantly impacted by the changes in network shape, produced by the original pectin solution's ph. Increasing the pH from 1.5 to 3.0 in 10 times higher Young's moduli and stress at 85% deformation which means this correlated with increased bulk density. Coulombic repulsions were reduced. As a result, in weaker acid-gels at "high" pectin concentrations of 5% and even blocking acid gelation at lower pectin concentrations of 4.5 wt%. The aerogels has a maximum Pb2+ adsorption capacity 373.7 mg/g, measured at pH 5.0, lightweight low is 63.4 mg/cm3, has a mechanical strength is stress over 0.24 MPa at 50% strain, and were easily recyclable (Wang et al., 2021)

While for starch, starch's mechanical properties play an important role in water treatment processes that remove dyes from wastewater. Starch-based adsorbents with appropriate mechanical qualities, such as high surface area, porosity, and mechanical strength, can successfully remove dyes from wastewater by sorption. The mechanical properties of starch-based adsorbents is critical for maintaining structural integrity during the adsorption process and preventing absorbed colours from being released back into the water. (Khoo et al., 2023)

Starch gels' viscoelastic qualities can also help them remove pigments from wastewater via mechanisms like entrapment and diffusion. The film-forming characteristics of starch can also be used to create starch-based membranes for colour removal using methods like ultrafiltration and reverse osmosis. Overall, starch-based materials' mechanical qualities are key factors to consider when designing and optimising water treatment procedures for dye removal from wastewater.(Khoo et al., 2023)

2.7 Potential use of aerogel for heavy metal removal

Aerogels have a wide range of uses, including thermal insulation, acoustic insulation, capacitor electrodes, carriers for different materials catalysts, solar cells, and adsorbents for the removal of organic and inorganic contaminants from wastewater. Adsorption is the process by which a liquid (adsorbate) collects on the surface of a solid (adsorbent) and creates a molecular or atomic layer (Meena et al., 2005).

The carbon aerogels have lately grabbed researchers' interest. This is because of broad applicability in sectors such as solar cell, super-capacitor, elector-chemical, and environmental science. Furthermore, carbon aerogels, due to their unique qualities such as low density, three-dimensional (3D) interconnected porosity, large surface area, and extraordinary wettability, may be utilised to extract organic and inorganic pollutants from water/wastewater (Meena et al., 2005).

Adsorption is gaining popularity among researchers due to its simplicity, low cost, and success in the removal of heavy metal ions. Contact length, solution pH, ionic medium and beginning metal concentration, adsorbent dosage, temperature, and coexisting ions all had a significant impact on the removal of contaminants using aerogels. The highest adsorption capability for heavy metal ions of the three groups investigated shifted from organic-inorganic hybrid to organic and inorganic. Because of their porous structure, good adsorption capabilities, and simplicity of modification, graphene-based aerogels are frequently employed for heavy metal ion removal from aqueous solutions (Meena et al., 2005).

2.8 Methylene blue structure

Methylene blue, a heterocyclic aromatic chemical molecule, has the molecular formula $C_{16}H_{18}N_3SCl$. Its chemical structure is a thiazine ring system with a methylamino group and a sulphur atom. The molecule can exist in several forms depending on the pH of the environment, as stated in the accompanying text. In a neutral environment, methylene blue is presented in a form with one positive charge localised on the sulphur atom. In the acidic solution, one of the dimethylammonium groups is protonated, resulting in the presence of two positive charges.(Charmas et al., 2023)

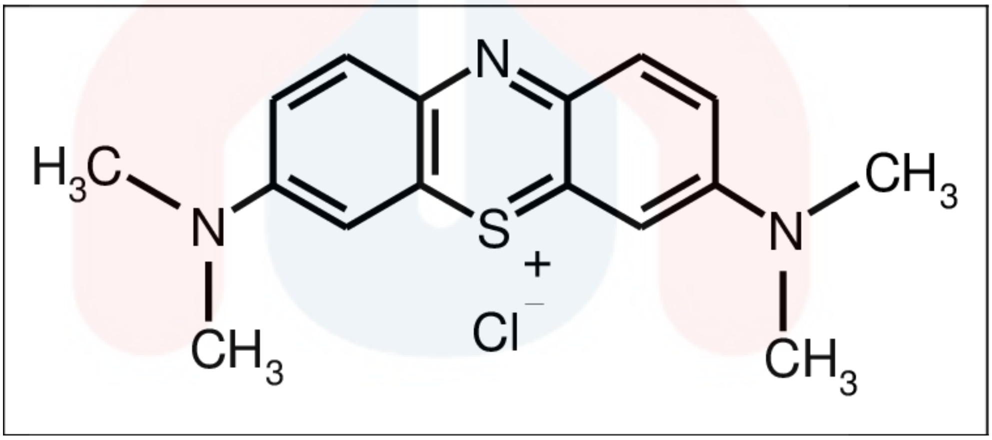

Figure 2.4: Chemical structure of methylene blue (Ghosh et al., 2019).

Methylene blue is widely utilised in a variety of applications, including biological staining, redox indicators, and methemoglobinemia treatment. It has also been investigated for its possible application in wastewater treatment due to its ability to adsorb onto diverse substrates.(Charmas et al., 2023)

CHAPTER 3

MATERIALS AND METHODS

3.1 Materials

Pectin powder was supplied from R&M chemicals. Tapioca starch was purchased from mart. Graphene oxide was supplied from Alorich Chemistry.

3.2 Methods

3.2.1 Preparation of Pectin

The pectin preparation process was initiated through a series of steps, including dissolution, retrogradation, solvent exchange, and freeze drying. Initially, pectin powder was weighed (5g) using a scale and placed in a beaker. Distilled water (95ml) was prepared and combined with the previously weighed pectin powder to generate a 5% wt concentration of the pectin solution. The solution was stirred for approximately 20 minutes until homogeneity was achieved, utilizing a magnetic stir. Subsequently, each solution was introduced to graphene oxide in varying concentrations 0.2, 0.4, 0.6, 0.8, 1.0 mg/ml, and the mixture was stirred thoroughly for about 10 minutes using a glass rod.

Following this, the solution was subjected to heating at 90°C for 45 minutes in a thermostatic bath under stirring to induce starch gelatinization. The resultant solution was then poured into a silica mould for pre-freezing, allowing it to cool for 10 to 15 minutes. The samples were subsequently placed in a -80℃ freezer and left for 24 hours to undergo the retrogradation process, aimed at minimizing the potential for moisture absorption. Upon completion of retrogradation, the drying step was executed over a period of 3 days. The culmination of the drying process led to the formation of starch aerogel.

The figure below shows the pectin-prepared samples with different concentrations of graphene oxide.

Sample	**Concentration Of GO (mg/ml)**
P0.0	0.0
P0.2	0.2
P0.4	0.4
P0.6	0.6
P0.8	0.8
P1.0	1.0

Table 3.1: Pectin-prepared samples with different concentrations of graphene oxide.

3.2.2 Preparation of Starch

The starch preparation process was initiated through a series of steps, including dissolution, retrogradation, solvent exchange, and freeze drying. Initially, tapioca starch was weighed (5g) using a scale and placed in a beaker. Distilled water (95ml) was prepared and combined with the previously weighed starch to generate a 5% wt concentration of the starch solution. The solution was stirred for approximately 20 minutes until homogeneity was achieved, utilizing a magnetic stir. Subsequently, each solution was introduced to graphene oxide in varying concentrations 0.2, 0.4, 0.6, 0.8, 1.0 mg/ml, and the mixture was stirred thoroughly for about 10 minutes using a glass rod. Following this, the solution was subjected to heating at 90℃ for 45 minutes in a thermostatic bath under stirring to induce starch gelatinization.

The resultant solution was then poured into a silica mould for pre-freezing, allowing it to cool for 10 to 15 minutes. The samples were subsequently placed in a -80℃ freezer and left for 24 hours to undergo the retrogradation process, aimed at minimizing the potential for moisture absorption. Upon completion of retrogradation, the drying step was executed over a period of 3 days. The culmination of the drying process led to the formation of starch aerogel. Figure 3.1 depicts the starch-prepared samples with different concentrations of graphene oxide, illustrating the variations in the aerogel structure or properties.

Sample	**Concentration of GO (mg/ml)**
S0.0	0.0
S0.2	0.2
S0.4	0.4
S0.6	0.6
S0.8	0.8
S1.0	1.0

Table 3.2: Starch-prepared samples with different concentrations of graphene oxide

3.2.3 Preparation of Graphene oxide

The graphene oxide solution was dilute using formula M1V1 = M2V2. M1 and V1 represent the beginning concentration and volume, while M2 and V2 represent the ultimate concentration and volume, respectively. GO solution was diluted in different concentrations (0.2,0.4,0.6,0.8,1.0) using the formula. Each solution concentration was diluted using a micropipette 1000µL.

Each concentration was then added into each pectin and starch solution. For example, 0.2 (200 µL), 0.4 (400 µL),0.6(600 µL), 0.8 (800 µL),1.0(1000 µL). After that, the mixture was stirred thoroughly for about 10 minutes using a glass rod until it become homogenous with each pectin and starch solution.

3.2.4 Preparation of synthetic dye solution

Methylene blue (MB) was obtained from R&M Chemicals. Methylene blue solution (1000 mg/L) was prepared through dissolving 1.000 g methylene blue with distilled water in the volumetric flask of 1 L. The prepared solution was diluted with distilled water to obtain the required concentrations for each experiment.

3.3 Characterization of aerogel

3.3.1 Characterization method

The characterization of pectin and starch aerogel were examined and carried out by using 3 main machines which is Scanning electron microscopy (SEM), Fourier transform infrared analysis (FTIR), Ultraviolet-visible spectroscopy (UV-Vis) and Thermogravimetric analysis (TGA). All this characterization was done to characterize the morphology and the structural change of pectin and starch aerogel.

3.3.2 Scanning Electron Microscopy (SEM)

Scanning electron microscopy JSM-IT100 enhanced function by JOEI ltd was used to study the surface morphology of pectin and starch aerogel. The pectin and starch pure 0 mg/ml, pectin, and starch with 0.6, 1.0 mg/ml graphene oxide loaded were examined using this machine. The magnification used is 200x, 500x and 1000x to examine the morphology of aerogel.

3.3.3 Fourier transform infrared analysis (FTIR)

Utilizing a Thermo Scientific Nicolet iZ10 FTIR was used to study the different functional groups of pectin and starch aerogel. Range wavenumber is between 500cm^{-1}. – 4000 cm^{-1} with resolution 4 cm^{-1}.

3.3.4 Thermogravimetric analysis (TGA)

TGA measures the thermal stability of compounds at high temperatures. Thermogravimetric Analysis (TGA) entails heating a sample under controlled conditions and measuring weight loss over time or at a specific temperature. The weight reduction after furnace heating and crucible insertion is assessed using a balance. The material under investigation impacts temperatures ranging from 25°C to 600°C, heating rates of 20°C/min, and nitrogen atmosphere gas flow at a rate of 50 ml min-1. Materials science uses TGA to determine material composition, purity, and heat stability. Universiti Malaysia Kelantan used EW-25753-44. Mettler Toledo TGA2 Thermogravimetric Analyser with Small Furnace, XP1 Balance, and TGA Sensor.

3.3.5 Adsorption Capacity

In experiments of batch equilibrium adsorption, 2.0 g of starch-GO aerogel samples were weighed and taken in several conical flasks. Methylene blue solution (100 mL) at 10 mg/L was added. Then, the flasks were placed in a covered area for 24 hours at room temperature (25±1°C). The solution was filtered, and the residual concentration of methylene blue was determined at 660 nm using a UV/vis spectrophotometer. The difference between methylene blue concentration before and after adsorption (Ci−Cf) to the initial concentration of methylene blue in the aqueous solution (Ci) is known as the percentage of methylene blue uptake, and it may be estimated using the following the equation:

$$Removal\,(\%) = \frac{(c_f - c_i)}{c_i}\, x\, 100$$

Figure 3.1: Equation of difference between methylene blue concentration

Unfortunately, only starch remained the same, but all pectin samples were dissolved in methylene blue solution. This is because pectin is hydrophilic and ionization which is acidic groups, such as carboxyl groups, may also appear in pectin molecules. These acidic groups can ionise in the presence of water, resulting in charged particles (ions) that contribute to pectin's solubility in water. Then, only starch was weighed after.

3.4 RESEARCH FLOWCHART

3.5 Research Flowchart

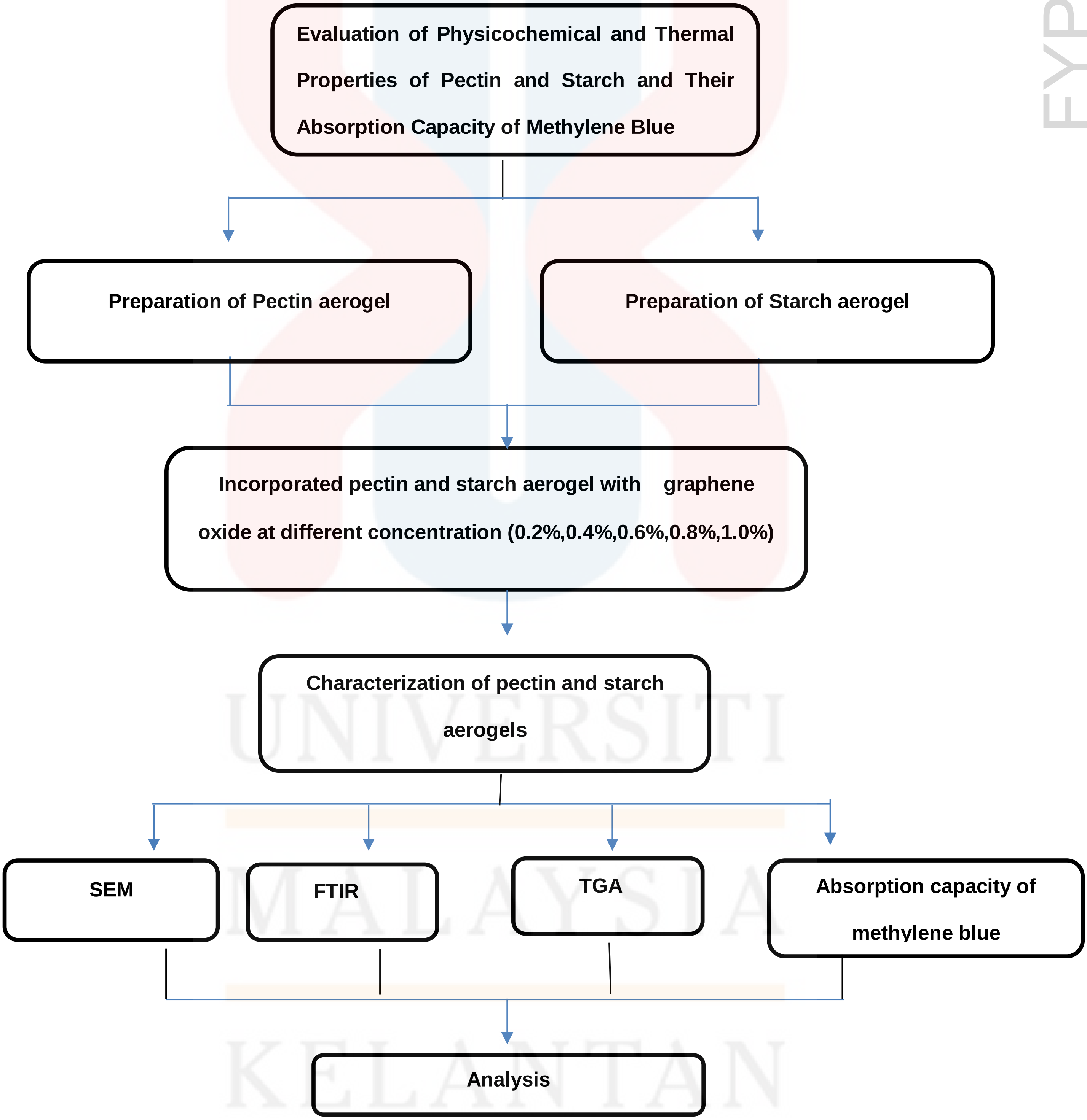

Figure 3.2: Research flow for aerogel.

CHAPTER 4

RESULT AND DISCUSSION

Characterization of all samples that are the pectin pure, pectin with 0.6 mg/ml GO, pectin with 1.0 mg/ml GO, tapioca pure, tapioca with 0.6 mg/ml GO and tapioca 1.0 mg/ml GO were analysed by Scanning electron microscopy (SEM), Fourier transform infrared analysis (FTIR), Ultraviolet-visible spectroscopy (UV-VIS) and Thermogravimetric analysis (TGA).

SEM was used to investigate the microstructure of tapioca starch and pectin. SEM analysis of starch and pectin microstructures is valuable for a wide range of applications, including food industry optimisation and the development of innovative materials for various industries. It contributes to the understanding of structure-property relationships, enabling for better control and use of these polymers in several applications.

FTIR graph shows the transmission of infrared light through a sample at different wavelengths. The sample's functional groups absorb infrared light at different wavelengths, resulting in spectral dips or peaks. By examining the peaks and their intensities, we may identify and comprehend the functional groupings represented in the sample.

UV-VIS spectroscopy with standard curves is used to evaluate methylene blue's absorption capacity and develop a quantitative link between absorbance and concentration.

TGA measures the thermal stability of compounds at high temperatures. Thermogravimetric Analysis (TGA) entails heating a sample under controlled conditions and measuring weight loss over time or at a specific temperature. The weight reduction after furnace heating and crucible insertion is assessed using a balance.

3.6 Surface Morphology

3.6.1 SEM

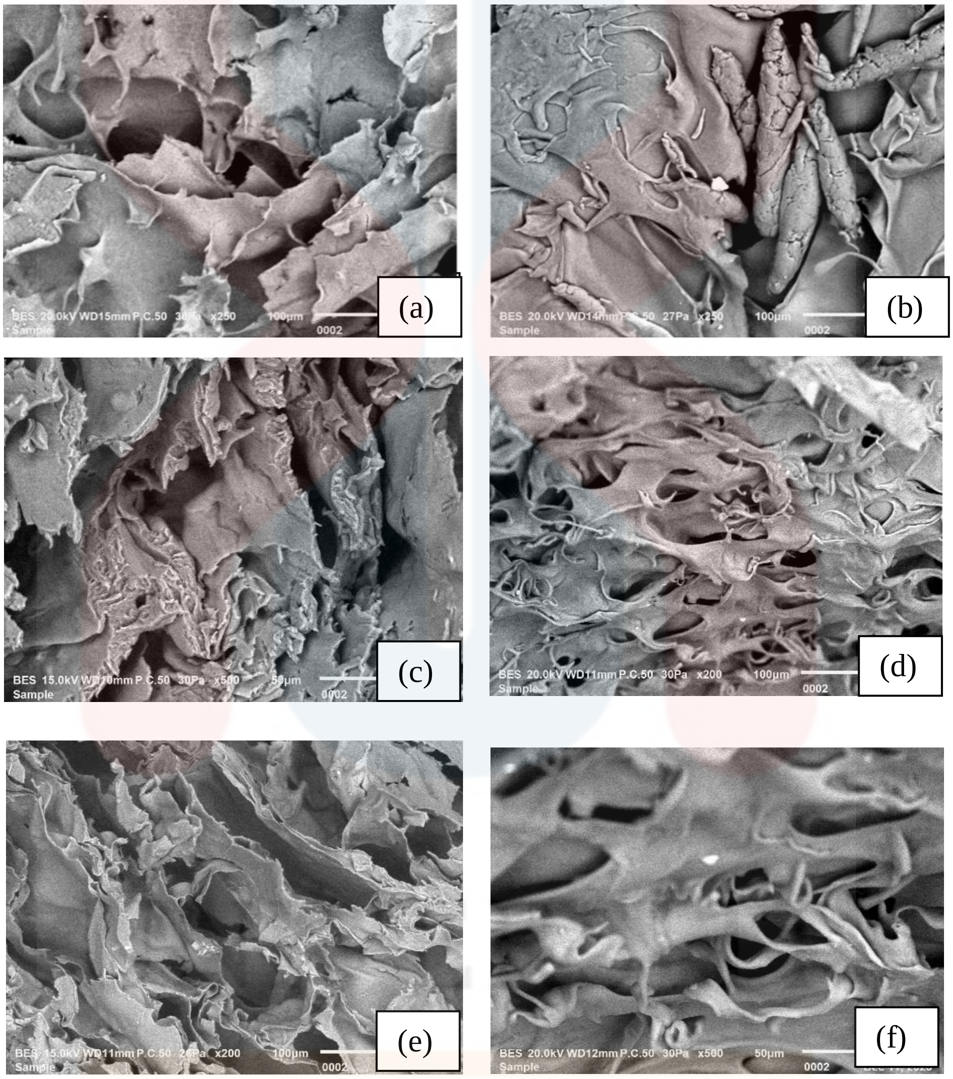

Figure 4.1: SEM images of (a) S0.0, (b) P0.0, (c) S0.6, (d) P0.6, (e) S1.0, (f) P1.0.

Figure 4.1 shows the SEM images of (a) S0.0, which is starch without GO loaded, (b) P0.0, pectin without GO loaded, (c) S0.6, starch with 0.6 mg/ml GO loaded, (d) P0.6, pectin with 0.6 mg/ml GO loaded, (e) S1.0, starch with 1.0 mg/ml GO loaded and (f) P1.0, pectin with 1.0 mg/ml GO loaded.

SEM analysis was done to examine the microstructure of tapioca starch and pectin. SEM study of starch and pectin microstructures provides useful information for a variety of applications, including food industry optimization and the development of novel materials for diverse sectors. It aids in the knowledge of structure-property correlations, allowing for improved control and utilization of these polymers in a variety of applications.

From the SEM result that shows in figure, between the pure pectin and with graphene oxide (GO) added, it showed more porous microstructure with GO added in it as well as starch. This is because graphene oxide incorporated with both pectin and starch will enhance the properties of both pectin and starch since oxygen functional group in GO will allow the formation of hydrogen bond with polysaccharide. Since starch and pectin is a polysaccharide, the porous structure was created. This will make the starch and pectin incorporated with GO is an excellent for water absorption.

3.6.2 FTIR

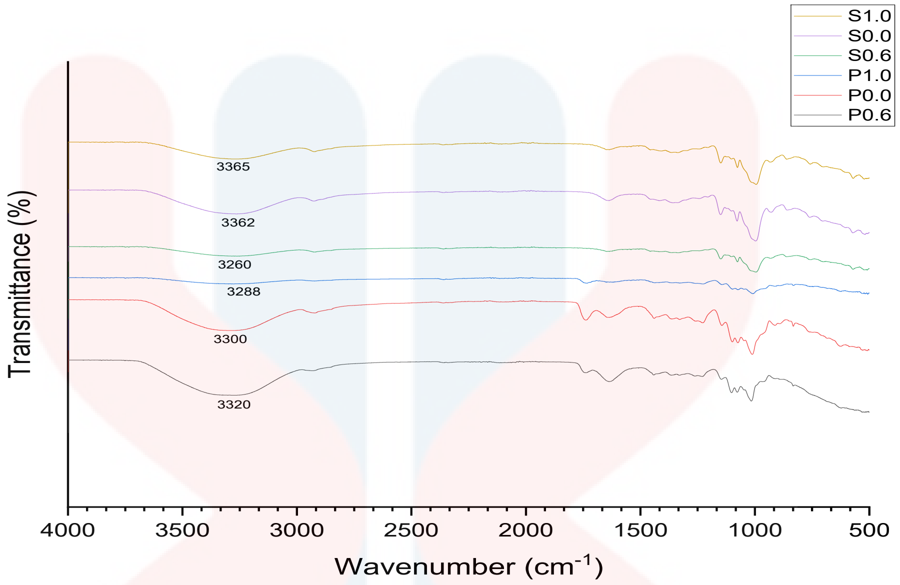

Figure 4.2: FTIR graph of (a) S1.0, (b)S0.0, (c)S0.6, (d)P1.0, (e)P0.0 and (f)P0.6

Figure 4.2 shows the graph of (a) S0.1 which is starch with 1.0 GO loaded, (b) S0.0,starch without GO loaded, (c) S0.6 starch with 0.6 GO loaded, (d) P1.0, pectin with 1.0 GO loaded, (e) P0.05, pectin without GO loaded and (f)P0.6, pectin with 0.6 GO loaded.

The FTIR graph depicts the transmission of infrared light through a sample at various wavelengths. The functional groups in the sample absorb infrared light at various wavelengths, causing dips or peaks in the spectrum. By analysing the peaks and their intensities, we may identify and comprehend the functional groups present in the sample. Comparing FT-IR plots for different concentrations of the same material allows you to see how the positions and intensities of the peaks change with concentration.

For S1.0 at 3311 cm^{-1} ,O-H stretching vibrations in hydroxyl groups of tapioca. At 2926 cm^{-1} C-H stretching vibrations in aliphatic hydrocarbon chains of tapioca. At 1637cm^{-1} C=O stretching vibrations in carbonyl groups of tapioca. At 1022 cm^{-1} C-O stretching vibrations in alkoxy groups of tapioca and 858 cm^{-1} α-glucosidic linkage between glucose units in tapioca starch.

For S0.6,O-H stretching vibrations are seen around 3300 cm^{-1} and are due to the presence of hydroxyl groups in the tapioca, pectin, and water molecules.C-H stretching vibrations are seen around 2900 cm^{-1} and are due to the presence of hydrocarbon chains in the tapioca and pectin molecules =O stretching vibrations are seen around 1600 cm^{-1} and are due to the presence of carbonyl groups in the tapioca and pectin molecules- O stretching vibrations are seen around 1050 cm^{-1} and are due to the presence of alkoxy groups in the tapioca.

For P1.0 at 3373 cm^{-1} O-H stretching vibrations in hydroxyl groups of pectin. At 2924 cm^{-1} C-H stretching vibrations in aliphatic hydrocarbon chains of pectin. At 1743 cm^{-1} C=O stretching vibrations in carboxylic acid groups of pectin. At 1608 cm^{-1} C=O stretching vibrations in esterified carboxylic acid groups of pectin. At 1036 cm^{-1} C-O stretching vibrations in alkoxy groups of pectin.848 cm^{-1} α-1,4-glycosidic linkage between galacturonic acid units in pectin.

For P0.0, 3324 cm^{-1} O-H stretching vibrations in hydroxyl groups of water. 2924 cm^{-1} C-H stretching vibrations in aliphatic hydrocarbon chains from the cuvette or background.1640 cm^{-1} C=O stretching vibrations in carbonyl groups from the cuvette or background.1050 cm^{-1} broad peak due to various C-O stretching vibrations.

For P0.6, its similar with pectin but with weaker peak since it has low concentration than P1.0.

3.7 Water Absorption (UV-VIS)

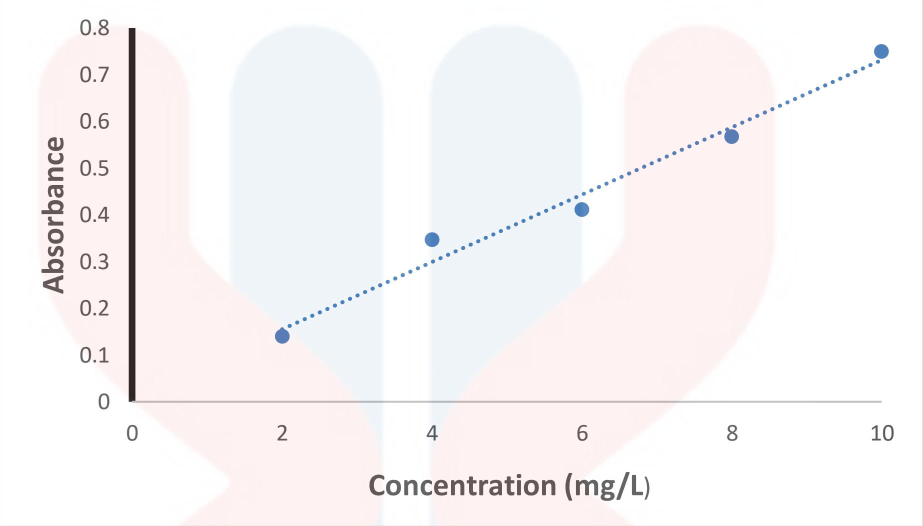

Figure 4.3: Standard curve of methylene blue solution

Figure 4.3 shows the standard curve of methylene blue solution in UV-VIS testing. The purpose of making this standard is to link a substance's absorbance to its concentration in a solution. This is because they establish a quantifiable link between a substance's absorbance and concentration. This connection is based on the Beer-Lambert Law, which asserts that a solution's absorbance is directly proportional to the concentration of the absorbing species and the path length of light through the solution.(Flores-Gómez et al., 2023)

A linear relationship between absorbance and concentration can be established by drawing a standard curve and measuring the absorbance of known concentrations of a chemical. It enables you to determine the concentration of an unknown material by measuring its absorbance and comparing it to the standard curve. Standard curves are required in analytical chemistry and spectrophotometry for the quantitative analysis of compounds in solution. They give a consistent and accurate method for estimating a substance's concentration based on absorbance, allowing researchers to quantify the amount of a specific analyte in a sample.

3.7.1 UV-VIS sample

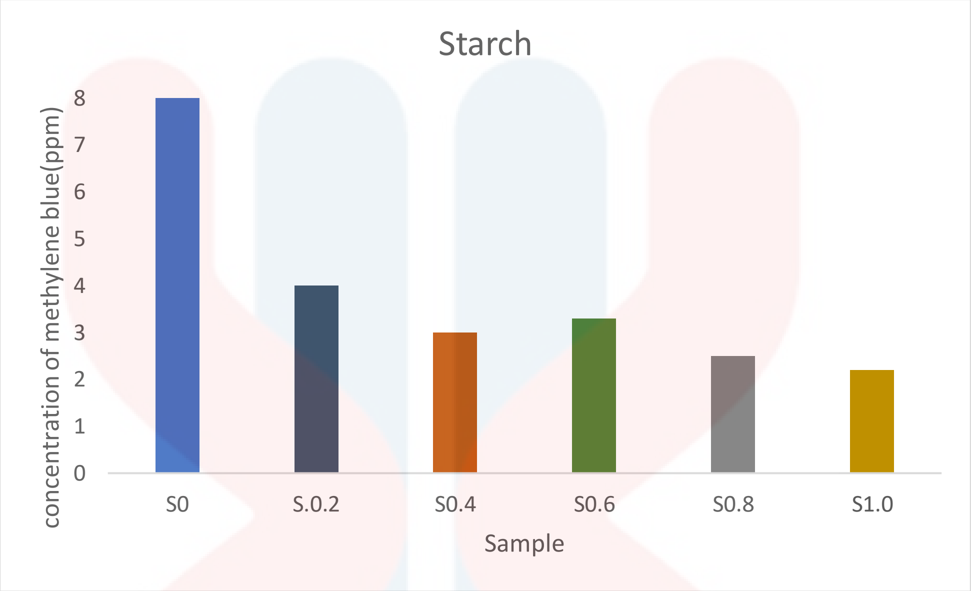

Figure 4.4: Absorbance of starch in methylene blue with different GO concentrations S0, S0.2, S0.4, S0.6, S0.8 and S1.0 after 24 hours.

The graph shows the methylene blue absorbance of tapioca starch with different concentrations which is 0,0.2,0.6,0.8 and 1.0 mg/ml graphene oxide loaded. The graph shows that absorbance of methylene blue by starch increased with concentration of graphene oxide loaded.

Sample pure with no graphene oxide loaded which is S0 is less absorbance of methylene blue which is the concentration shows at 8ppm than S1.0 with 1.0 mg/ml graphene oxide loaded shows at 2.2ppm. This is because starch can create hydrogen bonds with graphene oxide, which promotes film assembly and resists retrogradation, resulting in increased characteristics of the composite material.

GO with its vast surface area and functional groups, can boost the adsorption capacity of starch due to its great affinity for dyes such as methylene blue(Pinelli et al., 2021). The combination of graphene oxide with starch can lead to improved adsorption efficiency and capacity for dye removal applications. This technique takes advantage of the distinct features of graphene oxide and starch to produce a composite material with improved adsorption capabilities for contaminants such as methylene blue.

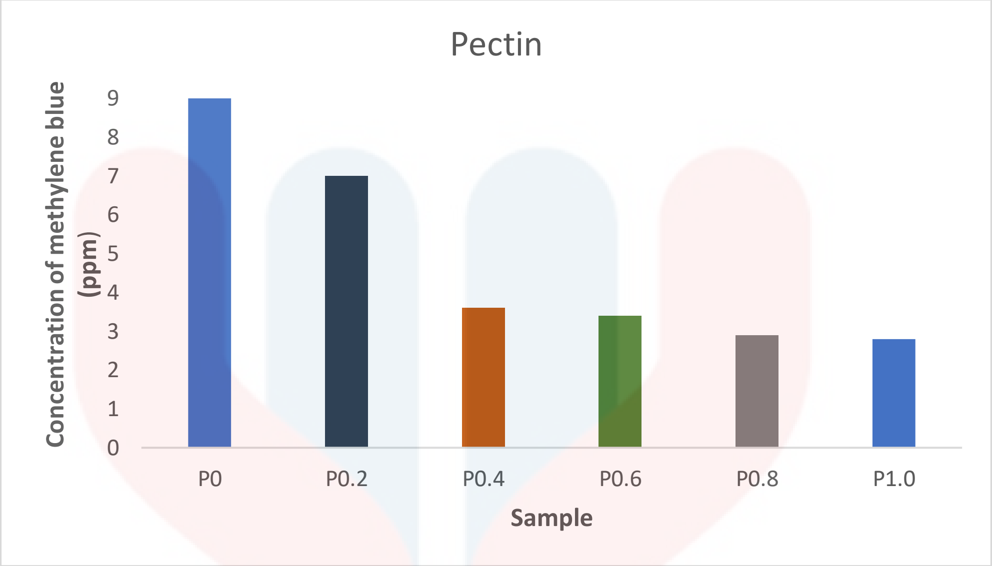

Figure 4.5: Absorbance of pectin in methylene blue with different GO concentrations (P0, P0.2, P0.4, P0.6, P0.8 and P1.0) after 24 hours.

Graph 4.5 shows the methylene blue absorbance of pectin starch with different concentrations which is 0,0.2,0.6,0.8 and 1.0 mg/ml graphene oxide loaded. The absorbance of methylene blue by pectin increased as the concentration increased.

This is because combining graphene oxide with pectin can improve absorption properties of methylene blue. The addition of graphene oxide can increase the composite's overall surface area, resulting in more active sites for dye adsorption. Furthermore, the functional groups on graphene oxide, such as the hydroxyl and carboxyl groups, can enhance interactions with methylene blue. For example, S0 shows concentration at 9 ppm and pectin with highest GO loaded which is S1.0 shows concentration of methylene blue at 2.88ppm.

Ultimately, combining pectin-starch with graphene oxide results in a composite material with improved adsorption properties, making it a suitable choice for wastewater treatment applications that need methylene blue removal.

3.7.2 Adsorption Capacity

Table 4.1: Degradation rate of starch in 24 hours

Samples	Weight Before (g)	Weight After(g)
S0.0	2	1.84
S0.2	2	1.82
S0.4	2	1.80
S0.6	2	1.83
S0.8	2	1.78
S1.0	2	1.75

From the table 4.1, it shows that S1.0 degrade the most which is 1.75g than S0.0 which is 1. 84g.This is because the combination of starch and graphene oxide (GO) can produce a polymer with a greater surface area than starch alone. This increased surface area creates more active sites for the adsorption of methylene blue molecules. Starch and GO have distinct features that can work together to improve the composite material's adsorption capacity. Starch, recognized for its biocompatibility and abundance of hydroxyl groups, can interact with methylene blue molecules via hydrogen bonds..(Huang et al., 2023)

The addition of GO to starch aerogels can result in the creation of a porous structure with linked networks. This porous shape offers improved access of methylene blue molecules to the active spots on the material, boosting the adsorption capacity. The incorporation of starch and GO may enhance the mechanical stability and structural integrity of the composite material, ensuring effective adsorption of methylene blue throughout numerous cycles without significant loss of adsorption capacity.(Huang et al., 2023)

3.7.3 Thermogravimetric analysis (TGA)

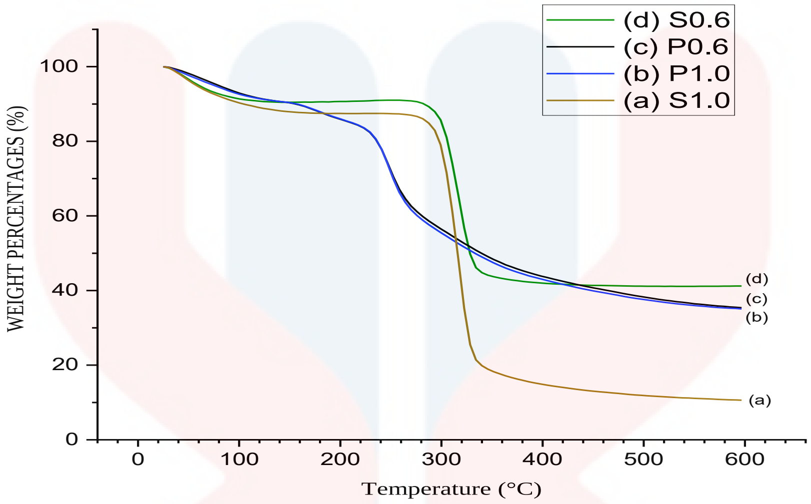

Figure 4.6: TGA graph of (a) S 1.0, (b) P1.0, (c) P0.6, (d) S0.6

Figure 4.6 shows TGA graph of (a) S1.0 which is starch with 1.0 mg/ml GO loaded, (b) P1.0 mg/ml, pectin with 1.0 mg/ml GO loaded, (c) P0.6, pectin with 0.6 mg/ml GO loaded and (d) S0.6, starch with 0.6 mg/ml GO loaded.

Thermogravimetric analysis (TGA) can provide useful insights into the thermal stability and breakdown behavior of materials such as pectin, starch, and graphene oxide when mixed in composite structures.

Based on the graph, all four samples show a similar pattern of weight loss, with a rapid decline beginning about 300°C. The addition of graphene oxide may affect the thermal stability of pectin and starch composites, resulting in changes in the onset temperature of decomposition or the extent of weight loss. S1.0 lose weight most slowly. This is because graphene oxide, with its strong thermal stability and barrier qualities, can operate as a protective layer or barrier surrounding starch particles.(Al-Mhyawi et al., 2023) The addition of graphene oxide can reduce the thermal degradation of starch by providing a physical barrier that slows the diffusion of volatile breakdown products, therefore lowering the rate of weight loss.

S0.6 loses weight the fastest of the four samples. This is because at lower graphene oxide loadings, the barrier of protection effect may not be adequate to effectively block starch heat deterioration. The restricted presence of graphene oxide may not form a continuous barrier surrounding the starch particles, allowing greater transport of volatile breakdown products, and resulting in rapid weight loss.(Al-Mhyawi et al., 2023)

P 1.0 loses weight slower than starch but faster than P 0.6 graphene oxide. The slower weight loss of P1.0 graphene oxide compared with starch samples and faster weight loss compared to samples with P0.6 graphene oxide can be attributed to a combination of thermal stability enhancement, char formation, thermal conductivity enhancement, strong interfacial interactions, and optimal graphene oxide loading in the pectin composite structure. These factors influence the rate of weight loss during thermal breakdown, resulting in the observed variations in weight loss behavior among the samples.

Finally, P0.6 loses weight faster than samples containing 1.0 mg/ml graphene oxide, but slower than starch samples. This is because of similar reason with P1.0 which is combination of thermal stability enhancement, char formation, thermal conductivity enhancement, strong interfacial interactions, and optimal graphene oxide loading in the pectin composite structure.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

To conclude, pectin and starch were good for absorption capacity of methylene blue. This is because of their good mechanical properties which is presence of functional group such as hydroxyl (-OH) and carboxyl (-COOH). We can see this from FTIR in figure 4.1 testing of both bio aerogels. Since its bio aerogels, it is biodegradable and environmentally friendly ingredients, making them viable candidates for dye adsorption applications. The structure of starch and pectin enables excellent interactions with methylene blue molecules, resulting in rapid adsorption and removal from aqueous solutions.(Pinelli et al., 2021)

Incorporating graphene oxide with pectin and starch will enhance more properties and methylene blue absorption because of the components' synergistic actions of both of it. This is because GO has a large surface area and functional groups for dye adsorption, but starch and pectin can improve the total adsorption capacity and efficiency of the composite material.(Pinelli et al., 2021) The incorporation of graphene oxide in the starch-pectin matrix can improve the adsorption effectiveness of methylene blue by providing more active sites for dye molecules to interact with. We can see this in UV-VIS methylene blue absorption of both pectin and starch in figure 4.3 and figure 4.4.

The incorporation of graphene oxide (GO) into starch and pectin can result in a more porous structure. This is because GO has a huge surface area because of its two-dimensional structure, which can produce greater surface area for interactions with starch and pectin molecules. The existence of GO can facilitate interactions with starch and pectin molecules, resulting in the production of a network structure with empty spaces that contribute to the porosity aspect of the composite material(Pinelli et al., 2021). Figure 4.1 shows the SEM testing of the sample and its shows by adding more concentration it will enhance more porous structure.

5.2 Recommendation

I strongly recommend continued research in this area by future researcher. This is because the topic is still in its early stages, and there aren't many researchers working on it. This work attracted my interest because of the material used, which is bio aerogel combined with nanomaterials (Graphene Oxide) as a dye water treatment.

Furthermore, I recommend that future researcher conduct X-ray diffraction (XRD) testing to gain deeper insights into the material ‘s crystalline structure and phase composition. When materials such as pectin, starch, and graphene oxide are combined, XRD examination can reveal important information about their crystalline structure and phase composition such as crystallinity changes.

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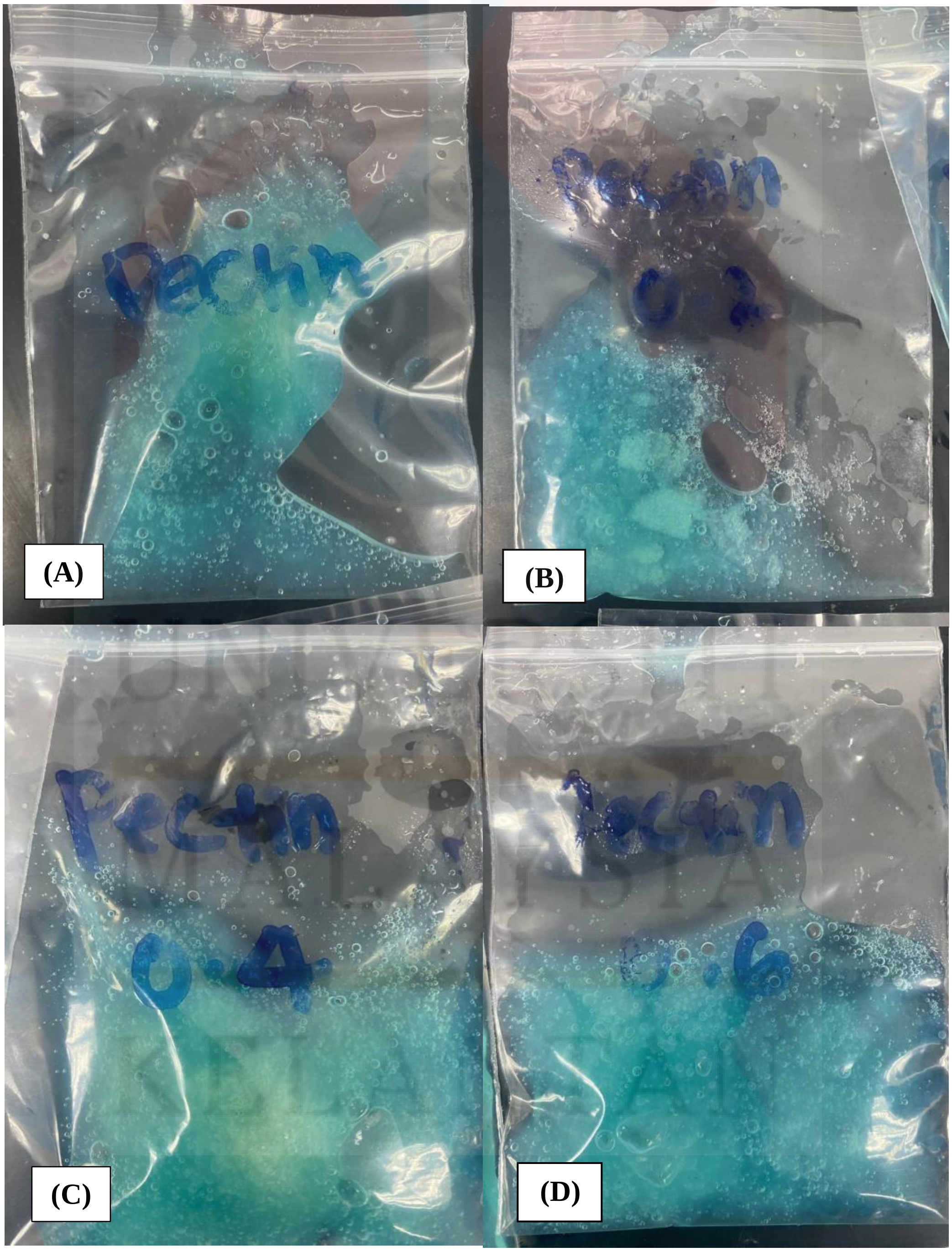
APPENDIX A

(A) Pectin, (B) Pectin 0.2%, (C) Pectin 0.4%, (D) Pectin 0.6%



(E) Pectin 0.8%,(F) Pectin 1.0%,(G) Starch,(H) Starch 0.2%

(I) Starch 0.4%,(J) Starch 0.6%,(K) Starch 0.8%,(L) Starch 1.0%