



**Faculty of Resource Science and Technology**

**Synthesis and Evaluation of the Cellulose-Based Hydrogels for Plant Growth**

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# Synthesis and Evaluation of the Cellulose-Based Hydrogels for Plant Growth

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## DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Malaysia Sarawak. Except where due acknowledgements have been made, the work is that of the author alone. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.



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## ABSTRACT

Hydrogels are hydrophilic polymeric materials characterised by three-dimensional crosslinked networks capable of absorbing and retaining substantial amounts of water. In recent years, research has predominantly focused on synthesising cellulose-based hydrogels using specific gelling agents, such as carboxymethylcellulose (CMC) in conjunction with epichlorohydrin (ECH), particularly for agricultural applications. However, comparative studies evaluating the influence of various gelling agents on the physicochemical and functional properties of cellulose-based hydrogels remain limited. In this study, cellulose extracted from paper waste was utilised to synthesise hydrogels incorporating different gelling agents—alginic acid (AA), carboxymethylcellulose (CMC), glycerol, and polyethylene glycol (PEG)—crosslinked with ECH. The morphological characteristics and functional groups of the hydrogels were investigated using Field Emission Scanning Electron Microscopy (FESEM) and Fourier-Transform Infrared Spectroscopy (FTIR), respectively. Among the formulations tested, the hydrogel containing 1.0 g of glycerol exhibited the highest swelling ratio, reaching 3562.97%, while the hydrogel formulated with 0.5 g of PEG demonstrated superior water retention capacity at 33.47%. Furthermore, hydrogels prepared with 1.75 g of CMC, 1.0 g of glycerol, and 0.5 g of PEG significantly enhanced soil moisture content, pH, and electrical conductivity across various soil types, including sandy, topsoil, gley, and clayey soils. The cellulose- 1.0g glycerol-based hydrogel also resulted in the highest seed germination with 65% of Choy Sum seeds (*Brassica chinensis* var. *parachinensis*). All hydrogel samples exhibited notable biodegradability, with approximately 87% degradation observed by day 60. These findings underscore the potential of tailoring gelling agents in cellulose-based hydrogel synthesis to optimize their performance for sustainable agricultural applications.

**Keywords:** Cellulose-based hydrogel, epichlorohydrin, gelling agents, soil moisture content, seed germination

## ***Sintesis dan Penilaian Hidrogel Berasaskan Selulosa untuk Pertumbuhan Tumbuhan***

### ***ABSTRAK***

*Hidrogel ialah bahan polimer hidrofilik yang dicirikan oleh rangkaian silang tiga dimensi yang mampu menyerap dan mengekalkan air jumlah besar. Dalam beberapa tahun kebelakangan ini, penyelidikan banyak tertumpu kepada sintesis hidrogel berasaskan selulosa menggunakan agen penggeli tertentu, seperti karboksimetilselulosa (CMC) bersama epiklorohidrin (ECH), khususnya bagi aplikasi pertanian. Walau bagaimanapun, kajian perbandingan yang menilai pengaruh pelbagai agen penggeli terhadap sifat fisikokimia dan kumpulan berfungsi hidrogel berasaskan selulosa masih terhad. Dalam kajian ini, selulosa yang diekstrak daripada sisa kertas telah digunakan untuk mensintesis hidrogel dengan pelbagai jenis agen pembentuk gel—asid alginik (AA), karboksimetilselulosa (CMC), gliserol, dan polietilena glikol (PEG)—yang dijalin silang menggunakan ECH. Ciri-ciri morfologi dan interaksi antara molekul dalam hidrogel telah dianalisis menggunakan Mikroskop Elektron Imbasan Pancaran Medan (FESEM) dan Spektroskopi Inframerah Transformasi Fourier (FTIR). Antara formulasi yang diuji, hidrogel yang mengandungi 1.0 g gliserol menunjukkan nisbah pembengkakan tertinggi, iaitu 3562.97%, manakala hidrogel yang diformulasi dengan 0.5 g PEG menunjukkan kapasiti pengekalan air tertinggi sebanyak 33.47%. Selain itu, hidrogel yang disediakan dengan 1.75 g CMC, 1.0 g gliserol, dan 0.5 g PEG telah meningkatkan kandungan kelembapan tanah, pH tanah, dan kekonduksian elektrik tanah dengan ketara dalam pelbagai jenis tanah termasuk tanah berpasir, tanah atas, tanah gley, dan tanah liat. Hidrogel berasaskan selulosa dengan 1.0 g gliserol juga menghasilkan kadar percambahan biji benih tertinggi sebanyak 65% bagi biji benih sawi Choy Sum (*Brassica chinensis* var. *parachinensis*). Semua sampel hidrogel mempamerkan sifat boleh terbiodegradasi yang*

*ketara, dengan anggaran 87% tahap degradasi direkodkan pada hari ke-60. Penemuan ini menekankan potensi pengubahsuaian agen pembentuk gel dalam sintesis hidrogel berasaskan selulosa bagi mengoptimumkan prestasi untuk aplikasi pertanian yang lestari.*

**Kata kunci:** *Hidrogel berasaskan selulosa, epiklorohidrin, agen pembentuk gel, kandungan kelembapan tanah, percambahan benih*

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## LIST OF ABBREVIATIONS

|       |                                             |
|-------|---------------------------------------------|
| AA    | Alginic acid                                |
| ECH   | Epichlorohydrin                             |
| FESEM | Field emission scanning electron microscopy |
| FTIR  | Fourier transform infrared                  |
| CMC   | Sodium carboxymethylcellulose               |
| NaOH  | Sodium hydroxide                            |
| NU    | NaOH/Urea                                   |
| PEG   | Polyethylene glycol                         |

# **CHAPTER 1**

## **INTRODUCTION**

### **1.1 Study Background**

Nowadays, food security worldwide is heavily affected by the lack of food supply as the world population grows and by the effects of climate change. Lots of crops cannot adapt to the changes in temperature, humidity, and pressure on the Earth caused by irregular weather patterns (Abbass et al., 2022). This affected the production of crops such as rice, flour, corn, and so on in developing countries. Besides, certain types of soils lose nutrients and water quickly due to drought, which directly affects the growth of vegetables and delays seed germination (Dietz et al., 2021). The moisture and the fertility of the soil were also affected by the change in temperature. Because structure develops considerably, aided by recurring cycles of soaking and drying, soils are particularly affected by climate change. Water can either drain away by side streams or directly penetrate the porous matrix from the surface soil, decreasing the soil's filtration function and increasing soil nutrient loss and water body pollution (Mekala & Polepongu, 2019). Thus, hydrogel is gaining much attention from researchers who are trying to improve the development of agriculture and solve the problem of crops planted in the country facing this issue (Wang et al., 2020). Hydrogel may offer advantages for reducing soil moisture and delaying seed germination because it is a hydrophilic polymer with a cross-linking network structure that can swell and retain many water molecules. It can be used as a water reservoir to provide a sustainable water supply to plants and soils and reduce the water shortage caused by drought stress. Hydrogel can be used as a soil conditioner to improve soil properties, such as moisture, pH, and salinity. Besides, it can be applied as a germination medium for the plant to provide a soilless

condition for horticulture due to its uptake and retention abilities. Some gelling agents, such as carboxymethylcellulose and glycerol, have recently been used to form hydrogels as soil conditioners and germination media (Omer et al., 2022; Qin et al., 2022; Zainal et al., 2019). Limited literature was studied on the effect of gelling agents on the characteristics of the cellulose hydrogel and applied to supply plant growth in the early stage.

Cellulose is the most popular starting material that is used in forming cellulose hydrogel because cellulose is the most abundant organic material that can be found in plants and biowaste with low-cost, stable, non-toxic, biodegradable, and hydrophilic properties (Zhang et al., 2017). Some studies synthesised the cellulose-based hydrogel by using sago pith waste, cotton, wastepaper, and wood (Chin et al., 2018, 2022; Pang et al., 2018; Jampi et al., 2021; Wong et al., 2021). All the hydrogels formed using those biowastes resulted in an excellent swelling ability, just like the synthetic hydrogel. In agriculture, the hydrogel's swelling and water retention ability are essential in germinating the seeds and maintaining soil moisture to help the crops overcome drought stress. Besides, cellulose extracted from the cellulosic waste can reduce yearly waste, especially for paper waste, because over 57,000 tons of paper waste are thrown into landfills yearly (Rahman et al., 2014; Zhang et al., 2017). Thus, the cellulose hydrogel in this study was utilised from the cellulose extracted from wastepaper.

In this study, cellulose extracted from waste paper are used to form cellulose-based hydrogels with different gelling agents to determine the effect of each gelling agent on increasing the effectiveness of the water swelling and retention ability of the hydrogel for plant growth.

## 1.2 Problem Statement

Lots of studies used the gelling agents of alginic acid (AA) and polyethylene glycol (PEG) in forming the hydrogel with other materials besides cellulose, and applied for pharmaceutical and environmental applications (Lee et al., 2020; Prusty & Swain, 2020; Sharma et al., 2022; Suhail et al., 2022). The AA had been used as a gelling agent for cellulose-based hydrogel, but it was also used in testing the performance in biomedicine (Sathasivam et al., 2018; Kahya et al., 2019; Morozkina et al., 2022). Thus, there is limited literature on cellulose hydrogels with these two gelling agents to improve soil properties and support seed germination.

Aparts, many studies only used individual gelling agents such as carboxymethylcellulose (CMC), glycerol, AA, and PEG to form cellulose hydrogel and investigated the effect of that gelling agent on the characteristics of the hydrogels (Kozłowska et al., 2018; Zhu et al., 2018; Lee et al., 2020; Miljković et al., 2021; Suhail et al., 2021; Qin et al., 2022). While these gelling agents offer physicochemical properties that influence the hydrogel behaviours, comprehensive studies comparing and optimising these materials under similar conditions remain scarce. This gap limits the understanding of how different gelling agents affect the hydrogel properties, such as swelling behaviour, biocompatibility and potential applications in agriculture. Besides, the hydrogel's use for improving the health of these soils, such as sandy soil, topsoil, gley soil, and clayey soil, is limited. Therefore, further investigation is needed to evaluate and compare the characteristics of hydrogels with these gelling agents to guide the development of more efficient and application-specific hydrogel systems for plant growth.

This study aimed to synthesise cellulose hydrogels with various gelling agents and compare their performance in swelling and retention ability, improve soil properties for various soil types, and provide soilless farming for seeds.

### **1.3 Objectives**

The objectives of the study are:

- i. To synthesise the cellulose hydrogels using various gelling agents.
- ii. To analyse the physical and chemical properties of the cellulose-based hydrogel.
- iii. To evaluate the potential application of cellulose hydrogels as soil conditioners under various soil types and seed germination.

### **1.4 Significance of the study**

This study is important for further studies on cellulose hydrogels. It employed a systematic research method to cover the major research gaps among the four gelling agents used to study the swelling and retention behaviours. Besides, this study also covered the findings on the factors that affected the surface properties and functional groups in the cellulose hydrogels caused by each gelling agent.

Furthermore, this study also introduced green technology to address the issue of soil caused by the rise in temperature. Cellulose hydrogels, formulated with various gelling agents, can act as water reservoirs to improve soil properties and help different soil types adapt to changing environmental conditions.

Soilless farming represents a significant innovation in sustainable and controlled agricultural systems. However, its adoption remains limited among farmers and households due to high implementation costs. This study explores the potential of cellulose hydrogels, formulated with various gelling agents, as a cost-effective germination medium to support the cultivation of plants and vegetables in soilless farming systems.

## **1.5 Layout of this thesis**

This thesis is organised into five chapters. Chapter 1 presents the study's background, problem statement, objectives, and significance. Chapter 2 reviews the relevant literature on hydrogels, their synthesis, properties, and applications in agriculture. Chapter 3 details the materials and methods used for hydrogel synthesis and characterisation, and the experiments include the soil conditioner, seed germination and biodegradability of the hydrogel. Chapter 4 presents the results and discussion of the experimental findings. Finally, Chapter 5 provides conclusions, implications, and recommendations for future work.

To determine the result of this study, cellulose hydrogels were synthesised using various concentrations of gelling agents. Each sample was subsequently characterised using Field Emission Scanning Electron Microscopy (FESEM) to examine surface morphology, and Fourier-transform Infrared Spectroscopy (FTIR) to identify the functional groups present. The swelling behaviour and water retention capacity of each hydrogel were evaluated. Additionally, Brunauer–Emmett–Teller (BET) analysis was performed to obtain detailed information on the pore characteristics of the hydrogels. For application purposes, the hydrogel formulated with each gelling agent that exhibited the highest swelling ratio and water retention was selected as a soil conditioner and seed germination medium.

## **1.6 Chapter Summary**

This chapter outlines the background and limitations that prompted the need for this research. It presents three specific objectives aimed at addressing the identified research gaps. Additionally, the significance of the study was discussed, and the structure of the thesis is outlined, highlighting how each set of results was obtained and presented.

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 Overview**

The ongoing devastation of various human activities has caused the temperature of the environment to rise sharply over the past 65 years, since 1880 (Currie & Deschênes, 2016; Abbass et al., 2022). Due to changes in the earth's temperature, humidity, and pressure, climate change has also resulted in erratic weather patterns (Abbas et al., 2022). The environmental conditions above the ideal range resulted in many crops losing water and being unable to survive because of decreasing soil moisture in many areas. This environmental problem has severely impacted human beings, significantly decreasing global food security, which has a direct impact on crop production in developing nations like Malaysia, which in turn has destroyed food security (Akbari et al., 2022). Numerous technologies have been devised to meet food needs, adapt to climate change, and increase agricultural productivity (Kumar et al., 2020; Piccoli et al., 2024). Due to its water-uptake and retention capabilities, the hydrogel has garnered significant interest for application in agriculture and is one of the technologies utilised to support sustainable agricultural output.

A cross-linked network of hydrophilic polymers forms the innovative three-dimensional structure known as a hydrogel. The hydrogel's unique design draws water molecules to its hydrophilic functional groups, enabling exceptionally high water absorption (Kurniati et al., 2021). This remarkable property makes hydrogels ideal for various applications, from agriculture to medicine, enhancing their value and effectiveness. Although it is a hydrophilic polymer, it did not dissolve in the water for a short period because of the intermolecular crosslinking networks (Yahia, 2015). Hydrogel is classified

into two groups: natural hydrogel and synthetic hydrogel. The formation of synthetic hydrogel involves the use of various polymers, such as poly (ethylene glycol) diacrylate, poly (acrylamide), and poly (vinyl alcohol), among others. However, this type of hydrogel often involves harmful chemicals, making it difficult to decompose in the soil (Yahia, 2015; Nguyen & Tran, 2022). Natural hydrogel, also known as biopolymer-based hydrogel, uses natural materials. Because of its biodegradability, non-toxicity, and compatibility characteristics, this hydrogel is more popular than synthetic hydrogels, leading to its widespread use in various applications (Nguyen & Tran, 2022).

The most prevalent organic material on earth is cellulose, a naturally occurring substance frequently used to form bio-hydrogels, starch, keratin, chitin, and gelatin. A linear chain of (1–4) connected D-glucose units composes the polysaccharide cellulose, as shown in Figure 2.1. It is a renewable resource readily available in plants, natural fibres, and biowaste, characterised by low cost, stability, non-toxicity, biodegradability, and hydrophilicity (Zhang et al., 2017; Kundu et al., 2022). Chemical reactions can modify cellulose's wealthy hydroxyl groups to form cellulose-based hydrogels, which are broadly cross-linked and hydrophilic polymers that function in three dimensions due to hydrogen bonding, ionic interactions, van der Waals interactions, and covalent bonds held within the hydrogel (Kundu et al., 2022).

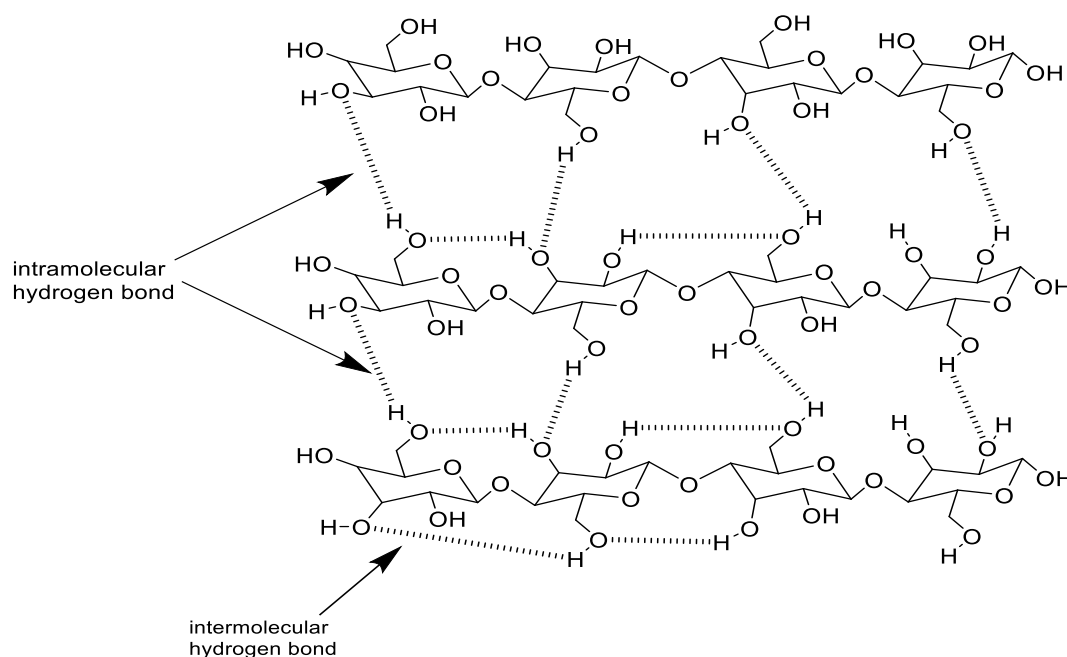


**Figure 2.1:** The chemical structure of cellulose chains.

Cellulose has been widely utilised in the production of hydrogels derived from various biowaste sources, such as Voon et al. (2017) was using cotton; Meng et al. (2019) was using lignin; Hu et al. (2019) was using paper; and Jampi et al. (2021) was using sago pith waste. These materials offer an abundant and renewable source of cellulose, making them attractive for developing sustainable hydrogel systems. An alkaline pre-treatment process is commonly employed to isolate cellulose from these complex waste matrices. This method effectively removes non-cellulosic components such as hemicellulose, lignin, and ink residues, especially from waste paper, thereby facilitating the extraction of high-purity cellulose (Beg et al., 2020).

The alkaline pre-treatment typically involves using sodium hydroxide (NaOH) to break the chemical bonds between lignin and hemicellulose, enhancing cellulose accessibility and yield. This process also aids in the recovery of fermentable saccharides, making it valuable for hydrogel synthesis and broader bioconversion applications. Numerous studies have applied this method in the synthesis of cellulose-based hydrogels, including

those by Voon et al. (2017), Ghodake et al. (2018), Kabir et al. (2018), Mohamed et al. (2023), and Ahmad et al. (2023), demonstrating its effectiveness and reliability in cellulose extraction and material preparation.

## **2.2 Dissolution process**

The dissolving process is a crucial step that must precede the production of the cellulose-based hydrogel. The dissolution process involves the use of an alkaline solution to break the intermolecular and intramolecular hydrogen bonds between cellulose chains. These bonds are formed by the abundant hydroxyl functional groups present on the surface of each cellulose molecule. Under low-temperature conditions, this process facilitates the formation of cellulose with a lower degree of polymerisation (Wong et al., 2021; Zainal et al., 2021). The dissolution process presents challenges due to the necessity of disrupting the complex interactions within the biopolymeric network, particularly among cellulose molecules (Baharm et al., 2016). Typically, sodium hydroxide (NaOH) and lithium hydroxide (LiOH) are the alkaline that will be used to dissolve the cellulose extracted from biomass like wastepaper, starch, cotton, and sago pith waste (Pang et al., 2011; Chin et al., 2011, 2018; Hu et al., 2019; Yang et al., 2020; Jampi et al., 2021; Jirawitchalert et al., 2022; Ahmad et al., 2023). According to the alkali dissolution theory, the hydroxyl ions replace the intramolecular and intermolecular hydrogen bonds within the cellulose polymer structures and promote the cellulose's interactions with water. In the meantime, the cellulose dissolves when the hydrated cation ions bond with it (Wong et al., 2021).

In addition to incorporating the alkaline into the delinking process, it was essential to introduce the hydrogen bonding acceptor into the delinking solvent to enhance the stability of the cellulose chains. Without this, the hydroxyl groups in the cellulose molecules will cause them to cluster quickly. Thus, adding the hydrogen bonding acceptor can form the

hydrogen bond with the hydroxyl groups of the cellulose molecules, reducing the hydroxyl groups that cause the aggregation of cellulose (Kang et al., 2016). Urea, in particular, has been identified as a practical component due to its ability to interact with the hydroxyl groups on the cellulose chain, thereby stabilising them through hydrogen bonding. This interaction forms a protective “jacket” around the cellulose chains, improving solubility and thermal stability, as illustrated in Figure 2.2. Among the various solvent systems explored, the sodium hydroxide/urea mixture has gained prominence for its high efficiency, low cost, and relatively low environmental impact (Salleh et al., 2018). Besides urea, some alternative reagents have been used to replace urea, like glycerol and polyethylene oxide, in recent years (Yan & Gao, 2008; Li et al., 2020). However, all the combinations with alkaline to dissolve the cellulose had a similar function to sodium hydroxide/Urea, and they did not affect the properties of the hydrogel (Swensson et al., 2020).

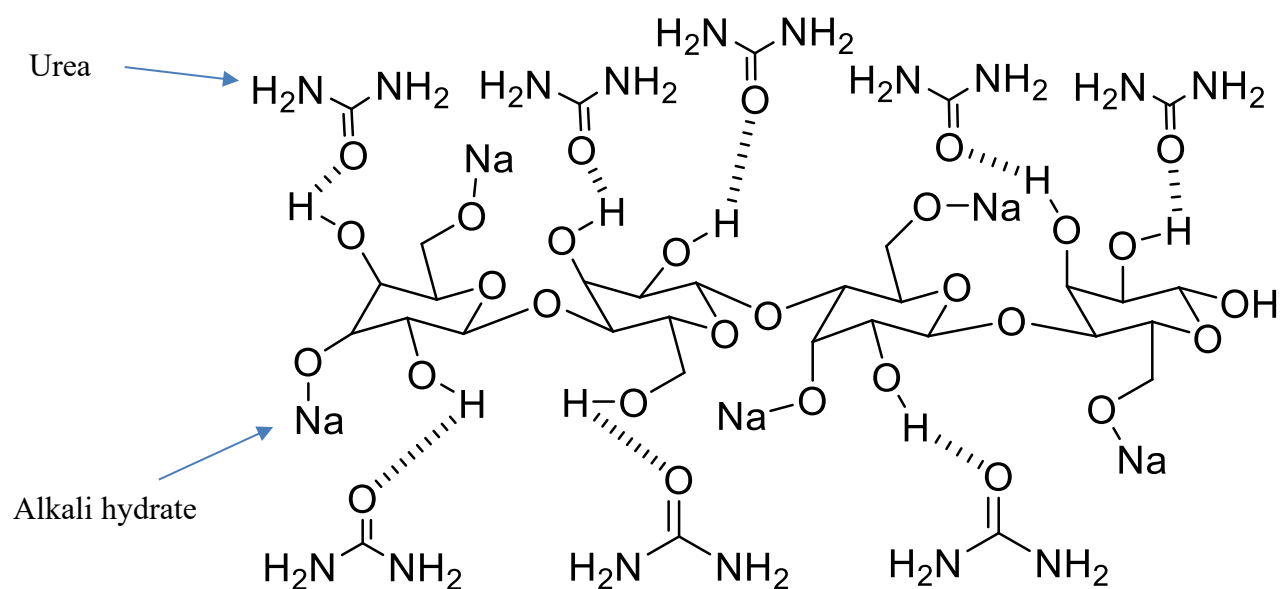


**Figure 2.2:** The cellulose-alkali-urea inclusion complexes are formed in an alkali/urea solution of cellulose.

### **2.3 The Process of Crosslinking**

Crosslinking is the process of chemically joining two or more polymer chains through covalent bonds, resulting in a three-dimensional network structure. In hydrogel preparation, the freeze-thaw technique is commonly employed during crosslinking, particularly when cellulose is combined with a gelling agent and a crosslinker. This method involves the crystallisation of bulk solvents or low molecular weight solutes at low temperatures, which reduces the free volume between polymer chains. As a result, the polymer concentration increases, encouraging the alignment and interaction of polymer chains. These interactions promote the formation of a stable hydrogel network through both hydrogen bonding and covalent linkages (Zainal et al., 2021). Crosslinking can be broadly classified into two types: physical crosslinking, which involves non-covalent interactions such as hydrogen bonds or ionic forces, and chemical crosslinking, which involves the formation of permanent covalent bonds between polymer chains.

Physical crosslinking involves the formation of network structures through non-covalent interactions, without chemical crosslinkers. Hydrogels formed by this method are typically reversible in nature, meaning they can undergo sol–gel transitions under certain environmental conditions (Varaprasad et al., 2017). Common mechanisms of physical crosslinking, such as the study done by Maitra & Shukla (2014), Parhi (2017), Varaprasad et al. (2017), Xue et al. (2022) include ionic interactions, crystallisation, stereo complex formation, hydrophobic interactions, polysaccharide interactions, protein–protein interactions, and hydrogen bonding.

In contrast, chemical crosslinking involves the formation of covalent bonds between polymer chains, resulting in the creation of permanent and typically irreversible hydrogels.

Various techniques fall under this category, including direct chemical crosslinking, grafting, radical polymerisation, condensation reactions, enzymatic crosslinking, and high-energy radiation-induced crosslinking (Varaprasad et al., 2017). These methods enable precise control over the resulting hydrogels' network structure and mechanical properties, making them suitable for a wide range of biomedical and industrial applications.

Among the various techniques for fabricating cellulose-based hydrogels, chemical crosslinking is the most widely employed. This preference is largely attributed to the favourable characteristics of chemical crosslinkers, which facilitate the formation of covalent bonds between polymer chains while often being non-toxic, chemically stable, and biodegradable (Demitri et al., 2013; Kundu et al., 2022). Chemically crosslinked hydrogels are particularly advantageous for drug and nutrient delivery or water absorption applications. Unlike physically crosslinked hydrogels, which tend to have limitations in curing time, pore size control, chemical functionalisation, and dissolution rate, chemically crosslinked hydrogels offer more consistent and tunable performance (Parhi, 2017). Crosslinkers generally used to form hydrogel are epoxy compounds with different chemical structures and amounts of epoxide groups (Klein & Poverenov, 2020). These epoxide-containing compounds enhance inter-chain interactions by forming covalent bonds with hydroxyl groups on the polymer backbone, thereby improving hydrogel properties such as elasticity, insolubility, viscosity, molecular weight, and overall stability (Salleh et al., 2019; Klein & Poverenov, 2020; Das et al., 2021; Zainal et al., 2021). Chemical crosslinkers frequently include epichlorohydrin (ECH), glyoxal, citric acid, and glutaraldehyde. These agents are generally favoured for their low cost, toxicity, and efficacy in improving hydrogel characteristics (Mali et al., 2018; Klein & Poverenov, 2020). Among these, epichlorohydrin (ECH) has gained prominence in recent years as a crosslinking agent in cellulose-based

hydrogel systems due to its ability to produce hydrogels with superior swelling capacity and water retention properties (Jampi et al., 2021; Abg Ahmad et al., 2023; Ahmad et al., 2023; Mohamed et al., 2023).

Among the various crosslinkers, epichlorohydrin (ECH), a short-chain epoxide organochlorine compound, is widely used in cellulose-based hydrogel synthesis due to its ability to readily react with polymeric hydroxyl groups. Hydrogels crosslinked with ECH are typically colourless, odourless, slightly soluble in water, and insoluble in polar solvents (Klein & Poverenov, 2020; Zainal et al., 2021). ECH has been shown to enhance several hydrogel properties, including pore size distribution, water absorption capacity, and mechanical resistance, while maintaining high chemical stability (Muharam et al., 2017; Zainal et al., 2021). Due to these advantages, ECH has become a focus of agricultural hydrogel research. For example, studies by Jampi et al. (2021) and Ahmad et al. (2023) demonstrated that cellulose-based hydrogels crosslinked with epichlorohydrin (ECH) exhibit enhanced mechanical strength and high elasticity, enabling them to absorb larger amounts of water without rupturing. Additionally, ECH improves swelling properties, including swelling kinetics and transparency, which are crucial for water retention and release applications (Budianto et al., 2015; Hussein et al., 2017; Muharam et al., 2017; Ciolacu, 2019). However, the concentration of ECH significantly influences the properties of the resulting hydrogel. Salleh et al. (2018) found that an optimal ECH concentration of 5 mL resulted in the best balance of flexibility and swelling capacity. Exceeding this amount led to an overly rigid hydrogel structure due to excessive crosslink density, thereby reducing its water swelling ability (Salleh et al., 2018; Rizwan et al., 2021). Furthermore, the concentration of the crosslinker directly affects gelation time: higher concentrations result

in faster gelation due to an increased number of intermolecular bonds formed between cellulose chains and the crosslinker (Nguyen & Tran, 2022).

## **2.4 Gelling agents**

The gelling agent, also known as a plasticiser, is a water-soluble polymer that can increase the viscosity of the solution and have the ability to form a gel (Jayakody et al., 2023). In synthesising cellulose-based hydrogels, typically achieved via chemical crosslinking methods, cellulose is commonly derived from natural sources such as plant biomass. However, a major limitation of native cellulose is its poor solubility in conventional solvents, including sodium hydroxide/urea (NaOH/urea, NU) systems. Gelling agents play a crucial role in enhancing the processability of cellulose by introducing functional groups that can partially substitute for hydroxyl groups along the cellulose backbone, thereby improving solubility and reactivity. Moreover, incorporating gelling agents has been shown to impart thixotropic behaviour to cellulose solutions, resulting in increased viscosity and improved gelation tendencies. These agents also promote the formation of a porous hydrogel network, which is essential for applications involving fluid absorption and release (Luo et al., 2020). Furthermore, gelling agents enhance the mechanical properties of hydrogels by increasing flexibility and elasticity, thereby reducing brittleness and improving elongation capacity (Rudyardjo & Wijayanto, 2017; Sun et al., 2018). This effect is attributed to the lubricating action of gelling agents, which minimises inter-chain friction within the polymer matrix (Javed et al., 2016). Gelling agents include carboxymethylcellulose (CMC), polyethylene glycol (PEG), glycerol, and alginic acid (AA). Commonly utilised gelling agents in hydrogel formulations. Despite their widespread application, there remains a lack of systematic studies evaluating the specific effects of these agents on the structural, chemical, and functional properties of cellulose-based hydrogels. There is a lack of studies

investigating and comparing the performance of various gelling agents in chemically crosslinked cellulose hydrogels, focusing on parameters such as water uptake capacity, water retention rate, and suitability for agricultural applications.

#### **2.4.1 Carboxymethylcellulose (CMC)**

Carboxymethylcellulose (CMC), a water-soluble cellulose ether characterised by the carboxyl groups, facilitates the formation of a crosslinking network with cellulose (Raafat et al., 2012; Salleh et al., 2019). Consequently, hydrogels synthesised with CMC exhibit an increased water retention capacity. In addition, CMC can benefit hydrogel development in conjunction with the crosslinker. This is because the modified cellulose exhibits a thixotropic behaviour, which contributes to an increase in the viscosity of the solution (Salleh et al., 2018). According to Salleh et al. (2019), the incorporation of sodium carboxymethylcellulose (CMC) is essential for producing hydrogels with enhanced water absorption capacity. The concentration of CMC should be at least 1 wt% or higher when added to the organic solvent system. At this concentration, CMC dissolves effectively due to its excellent solubility, resulting in a homogeneous solution without residue. This complete dissolution contributes to an increase in the viscosity of the hydrogel, which is beneficial for its structural integrity and swelling performance. This statement is also proved by another study done by Miljković et al. (2021). Additional cellulose derivatives with crosslinkers in the system enhance the degree of polymerisation. This occurs as more polymer chains bind adjacent molecules, leading to more significant and extensive network structures in the initial hydrogel (Salleh et al., 2019). So, the hydrogel is more accessible to form when the concentration of the CMC is high.

In addition to viscosity, the swelling capacity of cellulose-based hydrogels is strongly influenced by the concentration of CMC. Specifically, the water absorption of hydrogels increased from 1168% to 2181% as the concentration of sodium carboxymethylcellulose (CMC) was raised from 1.25 wt% to 1.75 wt%. This improvement is attributed to the increased concentration of  $\text{Na}^+$  ions at higher CMC levels, which generates Donnan osmotic pressure, thereby facilitating greater water uptake into the hydrogel matrix (Salleh et al., 2018, 2019; Miljković et al., 2021). Similarly, Ahmad et al. (2023) reported a swelling ratio of approximately 2000% for cellulose hydrogels containing comparable CMC concentrations. Conversely, the moisture content exhibits an inverse relationship with water absorption. When the CMC concentration exceeds 1.75 wt%, the hydrogel tends to retain higher moisture content, which may affect its overall swelling performance (Salleh et al., 2019). Furthermore, Tuan Mohamood et al. (2021) demonstrated that cellulose-based hydrogels formulated with 20 wt% sodium carboxymethylcellulose (CMC) and crosslinked using calcium chloride exhibited a swelling ratio of up to 4347%, underscoring the critical role of both CMC concentration and crosslinking agent in enhancing the hydrogel's water absorption capacity. In conclusion, optimising the type and concentration of gelling agents and crosslinkers is essential for tailoring the physicochemical properties of cellulose-based hydrogels to meet specific application requirements.

#### **2.4.2 Polyethylene Glycol (PEG)**

Polyethylene glycol (PEG) is an amphiphilic polymer characterised by its high solubility in both aqueous and organic solvents (Capanema et al., 2018). Structurally, PEG and polyethylene oxide (PEO) are analogous, as both are composed of repeating ethylene oxide units (Gibas & Janik, 2010; Yamaguchi et al., 2015). The primary distinction between the two lies in their molecular weight: PEG typically refers to polymers with molecular

weights below 20,000 g/mol, whereas PEO designates those above this threshold (Yamaguchi et al., 2015). In the present study, PEG is utilised to synthesise cellulose-based hydrogels. In biomedical applications, PEG has been widely employed as a gelling agent to enhance hydrogel porosity and modulate physicochemical properties, particularly when combined with carboxymethyl cellulose (CMC), as illustrated in Figure 2.3. Capanema et al. (2018) and Ghorpade et al. (2018) reported that the incorporation of PEG into cellulose-based hydrogels significantly increases their swelling capacity—from approximately 100% to as high as 5000%. This enhancement is attributed to interactions among CMC, PEG, and the crosslinking agent, which collectively influence the hydrogel's network structure. The inclusion of PEG contributes to improved thermal stability and facilitates water retention through increased polymer chain flexibility (Huntrakul & Harnkarnsujarit, 2019; Raihana et al., 2021). Additionally, PEG reduces the density of the crosslinked network, thereby increasing the hydrogel's capacity for swelling and water absorption (Mahmud et al., 2018). Although the functionalisation of cellulose-based hydrogels with PEG has been extensively investigated in biomedical contexts, such as drug delivery, tissue engineering, and surface modification (Liu et al., 2019; Gan et al., 2021; Sun et al., 2023). Limited research has explored their application in agriculture especially for potential use in agricultural systems.

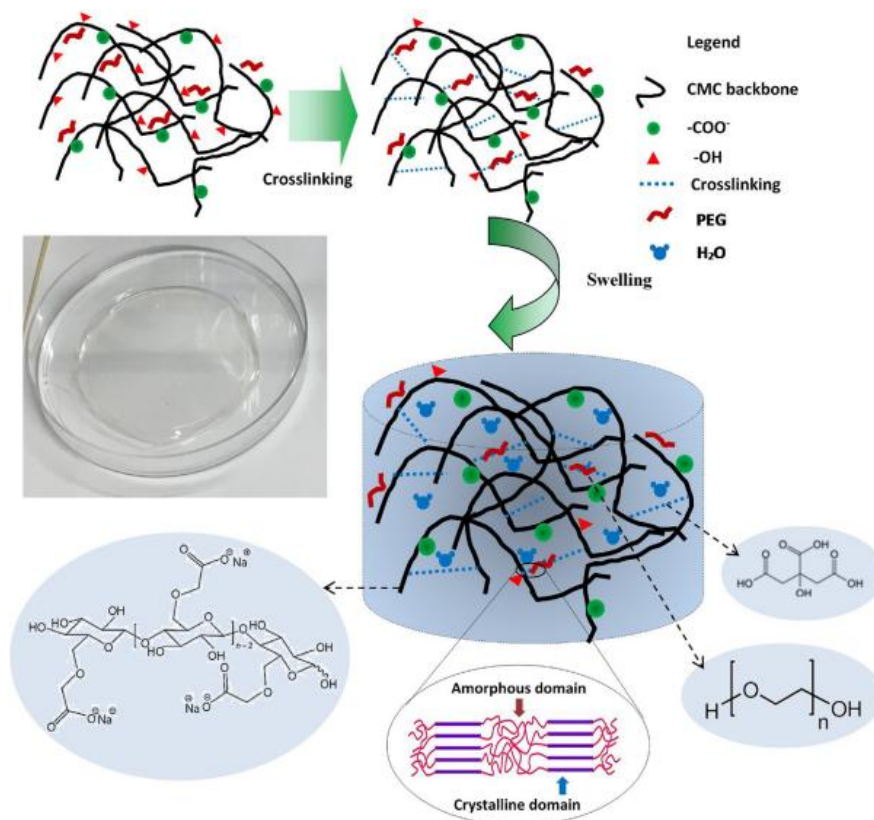


**Figure 2.3:** The hydrogel structure of the CMC, PEG, and crosslinker. Adapted [reprinted] from Capanema et al. (2018)

### 2.4.3 Glycerol

In recent years, glycerol has been widely employed as a gelling and plasticising agent in hydrogel synthesis, demonstrating significant improvements in the structural and functional performance of hydrogels. The incorporation of glycerol enhances both the mechanical strength and flexibility of hydrogels (Haske-cornelius et al., 2019). For instance, Kozłowska et al. (2018) reported that a hydrogel formulated with 1.5% glycerol exhibited the highest swelling ratio of 299%, which was attributed to increased flexibility of the polymer network. Similarly, Qin et al. (2022) demonstrated that glycerol improves the viscosity and stabilises the hydrogel structure. Additionally, the water retention capacity was enhanced, with glycerol-based hydrogels retaining water for up to eight hours. As a gelling agent, glycerol functions similarly to polyethylene glycol (PEG), promoting hydrogel

formation and improving flexibility. However, its influence on the polymer network is concentration-dependent. An increase in glycerol content can result in a denser cross-linked structure, which may reduce the swelling capacity due to a more compact network (Mohamed et al., 2023). Conversely, glycerol also contributes to increased porosity; higher glycerol concentrations facilitate the formation of larger and more numerous pores within the hydrogel matrix (Zhu et al., 2018). The integration of hydroxypropyl methylcellulose (HPMC), polyvinyl alcohol (PVA), glycerol, and blended paper has yielded hydrogels with enhanced water retention capabilities and a slow urea release profile, indicating their potential application in agriculture (Kareem et al., 2021). Despite these promising attributes, research on cellulose–glycerol-based hydrogels for agricultural use remains limited, particularly as soil amendments in arid or degraded soils. To date, only one study by Qin et al. (2022) has specifically evaluated the performance of cellulose–glycerol-based hydrogels in harsh soil conditions. In that study, the hydrogel was applied to soil planted with lettuce seeds, resulting in improved soil moisture retention and the highest observed germination rate of 38%.

#### **2.4.4 Alginic acid**

Alginic acid (AA) is a linear polysaccharide derived from the cell walls of brown microalgae or brown seaweed (Phaeophyceae) and is composed of D-mannuronic acid and L-guluronic acid residues arranged in block configurations along the polymer chain (Sahoo & Biswal, 2021; Suhail et al., 2021). Owing to its capacity for intermolecular binding, alginic acid is commonly used as a gelling agent in hydrogel formation. These bindings physically entrap water molecules following gelation, although water can still diffuse freely through the network. Hydrogels synthesised from alginic acid exhibit high swelling capacities, primarily due to increased viscosity and enhanced porosity of the hydrogel matrix (Suhail et

al., 2021). In an agricultural context, alginate has been used in combination with polyvinyl alcohol (PVA) and the crosslinker glutaraldehyde, resulting in hydrogels with high swelling ratios and improved soil water retention (Tariq et al., 2023). Despite its potential, the use of alginic acid as a gelling agent in combination with biopolymers such as cellulose for agricultural applications remains underexplored. To date, most studies have focused on its biomedical applications, particularly in drug delivery systems. Therefore, studying the performance of cellulose-based hydrogels incorporated with alginate in agricultural applications is crucial to evaluate their potential benefits.

## **2.5 Swelling property**

The ability to swell is a crucial physical characteristic for creating a hydrogel with superior water absorption and retention capabilities in agricultural applications such as preserving soil moisture and seed germination. The hydrogel's swelling properties allow it to trap liquid molecules through its cross-linked polymer network, which keeps the soil moist and promotes plant growth (Rizwan et al., 2021). The hydrogel's polymer matrix can hold the water molecules together. The minimal amount of water absorbed is between 10 and 20% of the water molecule. Nevertheless, the polymer matrix in hydrogels can retain water molecules through physical and chemical interactions. While the minimum water uptake typically ranges from 10% to 20% of the hydrogel's dry weight, under optimal conditions, hydrogels can absorb and retain up to several hundred to nearly a thousand times their dry weight in water. This substantial swelling is achieved as water penetrates the hydrogel network, causing the polymer chains to expand to their maximum extent (Yahia, 2015). The hydrogel will achieve its maximum water uptake rate once it has expended to the maximum amount of water due to the osmotic pressure created by the hydrophilic hydrogel groups of

the cellulose and the cross-linking agent (Raafat et al., 2012; Salleh et al., 2019; Wang et al., 2020; Lei et al., 2021).

Numerous studies have demonstrated that the concentration of the crosslinking agent directly influences the swelling capacity of hydrogels. As the crosslinker concentration increases, the hydrogel's ability to swell decreases. This reduction is attributed to the formation of a more rigid polymer network, where increased crosslink density restricts the mobility of polymer chains, thereby limiting their ability to absorb and retain water (Hussein et al., 2017).

The incorporation of gelling agents such as sodium carboxymethyl cellulose (CMC), polyethylene glycol (PEG), glycerol, and alginic acid during hydrogel formation has been shown to enhance the water uptake and retention capacities of hydrogels. Cellulose-based hydrogels plasticised with CMC have demonstrated excellent water absorption properties, as reported by Jampi et al. (2021) and Ahmad et al. (2023). Additionally, blending glycerol with CMC further improves the hydrogel's swelling capacity. A study by Qin et al. (2022) showed that the inclusion of glycerol significantly increased the hydrogel's absorbency, enabling it to retain more water compared to conventional hydrogels, which in turn supported better germination and growth of lettuce.

Moreover, alginic acid plasticised with polyvinyl alcohol (PVA) and crosslinked with glutaraldehyde has been reported to improve soil water retention, making it potentially beneficial for agricultural use (Tariq et al., 2023). While PEG and AA have been extensively explored in biomedical applications such as wound dressings and drug delivery systems (Capanema et al., 2018; Ghorpade et al., 2018; Suhail et al., 2021). The application of PEG and AA in agriculture remains limited.

## **2.6 Water retention ability**

Improved water retention enables a sustained moisture supply to plants over extended periods, particularly beneficial in arid and drought-prone regions. In this context, effective water conservation is critical in agriculture, as it enhances the soil's ability to retain water by applying hydrogels. This approach not only optimises water usage but also enhances the efficiency of water resources for agricultural production (Wang et al., 2020; Ashraf et al., 2021; Rizwan et al., 2021). Superabsorbent polymers (SAPs) have demonstrated an exceptional water reservoir in water conservation hydrogel, which can supply plants with adequate water in most arid regions. Additionally, research has been conducted on whey-based hydrogel regarding its water-holding capacity in enhancing agricultural products. The findings indicate that hydrogel produced from whey demonstrates effective water retention in sandy soil (Čechmánková et al., 2021). The water-holding capacity of hydrogels has been extensively researched, although cellulose-based hydrogels have not yet received as much attention. Thus, the water retention of the hydrogel plasticised with different gelling agents at various concentrations was determined and compared. Furthermore, the pH of the water used to soak the hydrogel impacted the hydrogel's ability to conserve water. Once the soaking media's pH has reached equilibrium with the alkaline hydrogel, the exchange of  $\text{OH}^-$  ions between the water and the hydrogel will be reduced because it has reached its limit (Salleh et al., 2019). However, the hydrogel's osmosis pressure is high. As a result, the soaking media must be replaced regularly to prevent the pH level from influencing the hydrogel's capacity to retain water.

## **2.7 Application of hydrogel in agriculture**

The hydrogel's ability to absorb and retain large quantities of water makes it particularly suitable for agricultural applications in arid and drought-prone regions.

Cellulose-based hydrogels are especially advantageous due to their biopolymeric origin, which ensures biodegradability in soil and minimises environmental impact (Yang et al., 2015; Miljković et al., 2021). The biopolymer-based hydrogel can quickly decompose in the soil without adversely affecting the environment. Besides, the reason that hydrogels are suitable and valuable in drought places is that hydrogels can lower the drought stress and the oxygen radical formation in the plant, and plants can also survive and grow in this type of environment with the help of hydrogels (Neethu et al., 2018). Several applications were studied for the agricultural sector. For example, the cellulose-based hydrogel was used as a controlled-release fertiliser (CRF) and acted as a water reservoir to keep the soil moist and provide water to the crops. It also acted as a growth medium for plants by boosting their growth.

As the controlled-release fertiliser, cellulose hydrogel with superior absorbent ability is constantly being used to deliver fertiliser at a slow rate to provide the nutrients the plants need. The hydrogel is used to control the amount of fertiliser, nutrients, and water in the scope because the hydrogel can give the right amount of fertiliser to the plant growth, which stores the fertiliser in the hydrogel, and this will not cause pollution to the environment (Zainal et al., 2021). The hydrogel absorbs several fertilisers, urea and Nitrogen, Phosphorus, and Potassium (NKP), with a ratio of 20:20:20. The cellulose hydrogel that is cross-linked with ECH has an excellent slow release rate of the nitrogen and able to continue to support the growth of the paddy plants along 60 days without additional adding any fertiliser (Abg Ahmad et al., 2023; Ahmad et al., 2023).

Hydrogel is also harmless and chemically stable for all chemical liquids; therefore, it is safe for agriculture and the environment and would not cause any problems on earth

(Neethu et al., 2018). In addition, the hydrogel can increase the water retention of the lands from 50% to 70% of the water stored in the soil, and there is much research that studies seed germination using hydrogel such as Akhter et al. (2004) and Cao & Li (2021). This is because hydrogels are capable of trapping water molecules within their porous network, with water retention largely influenced by the hydrogel's pore size distribution. The retained moisture supports plant roots, helping to prevent premature leaf shedding, preserve chlorophyll content, and maintain seed, fruit, and flower yields under water-deficient conditions (Radwan et al., 2018; Jampi et al., 2021). The incorporation of hydrogels into soil has been shown to positively influence plant growth by improving soil moisture content. Kumar et al. (2020) demonstrated that hydrogels effectively maintain water levels in the soil, thereby supporting sustained plant development. Since soil moisture content is a critical factor influencing plant growth, enhancing water retention through hydrogel application can improve crop performance. Moreover, hydrogels with high water-holding capacity can reduce water evaporation from the soil surface, further increasing soil moisture availability (Ai et al., 2021). In addition to their role in water conservation, hydrogels are also employed as fertiliser carriers due to their ability to slow nutrient release, ensuring a more controlled and sustained supply of nutrients to plants (Mohammadi-Khoo et al., 2016).

Jampi et al. (2021) demonstrated that the crosslinker used in hydrogel network formation significantly affects the hydrogel's seed germination performance. Hydrogels synthesised with an optimal concentration of epichlorohydrin (ECH), specifically 5 mL, exhibited superior water absorbency compared to those prepared without a crosslinker or with lower amounts of ECH. Increasing the crosslinker content enhances the hydrogel's flexibility and surface morphology, thereby increasing its capacity to retain water molecules. These results indicate that hydrogels incorporating an appropriate level of crosslinker can

serve as an effective water reservoir, mitigating soil moisture loss during dry seasons and supporting plant growth.

## **2.8 Chapter Summary**

This chapter has presented a comprehensive review of previous research related to cellulose-based hydrogels. It addressed key challenges faced by plants and soils, particularly in the context of water scarcity, and provided background information on hydrogels and cellulose as a biopolymer. The delignification process for extracting cellulose from cellulose waste was also discussed. Additionally, the chapter examined the role of chemical crosslinking in hydrogel formation, with specific attention to epichlorohydrin (ECH) as the chosen crosslinking agent. Various gelling agents used in hydrogel synthesis were reviewed, highlighting their influence on the swelling and water retention properties of hydrogels. These functional properties were emphasised through findings from prior studies. Finally, the application of cellulose-based hydrogels in agriculture, particularly for improving soil moisture and supporting plant growth, was explored in detail.

## **CHAPTER 3**

### **METHODOLOGY**

#### **3.1 Materials**

The wastepaper (the shredded A4 printed paper from the shredding machine) was collected from the Faculty of Resources Science and Technology, Universiti Malaysia Sarawak's office and used as the starting material to extract the cellulose in this study. Sodium hydroxide (NaOH) and urea purchased from HmbG Chemicals were used in the alkaline pre-treatment process of wastepaper and NaOH/Urea solvent preparation. Besides, ethanol with a purity of 95% from HmbG Chemicals (Germany) was used in removing the alkaline from the cellulose. The gelling agents used in the formation of the hydrogel are alginic acid (AA), carboxymethylcellulose (CMC), glycerol, and polyethylene glycol (PEG) with molecular weight of 400, 600, and 1000, which were purchased from Acros Organics (New Jersey, United States), Sigma-Aldrich (Darmstadt, Germany), and R&M Chemicals (Essex, United Kingdom). Epichlorohydrin (ECH) was purchased from Sigma-Aldrich (Darmstadt, Germany) and used in this study. The soil types (sand, topsoil, gley soil and clayey soil) used in this study were collected from the Real Living Lab (RLL), UNIMAS. The Choy Sum seeds with the brand Framoc were used in the germination assay on various cellulose hydrogels.

#### **3.2 Methods**

##### **3.2.1 Cellulose Extraction Process from Wastepaper**

The extraction process used in extracting the cellulose from the wastepaper has been reported by Ahmad et al. (2023). The wastepaper was soaked in the deionised water until it

became a pulp. The pulp was soaked in the NaOH solution (12%w/w) for 24 hours to remove the ink, lignin and hemicellulose contained in the wastepaper. After 24 hours, the extracted cellulose was washed and stirred with deionised water for 30 minutes. After washing, the cellulose was rewashed with 95% ethanol to remove the remaining NaOH from the cellulose. Then, the extracted cellulose was dried in the oven at 60 °C until all the water was removed by placing it in the desiccator with silica gel for 42 hours. After all the cellulose had dried, it was ground and sieved into white powder using a 60 (250 µm) mesh size sieve. The extracted cellulose in powder form was sent to undergo FTIR analysis for further confirmation of the purity of the cellulose.

### **3.2.2 Synthesis of cellulose-based hydrogel with different concentrations of gelling agents (alginic acid, carboxymethylcellulose, glycerol, and polyethylene glycol)**

The method for synthesising the cellulose hydrogel had been reported by Chin et al. (2022). 7g of NaOH and 12g of urea were added to 81g of deionised water to form sodium hydroxide (NaOH)/urea solvent. The mixture was precooled in the freezer until the temperature of the solvent reached -12 °C. 3g of the cellulose was dissolved in the pre-cooled NU solvent at -12°C and frozen for 12 hours. After 12 hours, the mixture was thawed and stirred until it reached a homogeneous solution. Then, the gelling agents, which are alginic acid (AA), carboxymethylcellulose (CMC), glycerol, and polyethylene glycol (PEG) (0.25g -2.5g), were added to the mixture, respectively. Then, 5 mL of epichlorohydrin (ECH) was added dropwise to the mixture and sonicated with a sonicator (Brand: UniLab) for 30 minutes. The mixtures were placed in the freezer (Brand: Sharp) at -12°C for 24 hours. The composition of the cellulose-based hydrogel with various gelling agents is displayed in Table 3.1. After the hydrogels had formed successfully, each underwent FTIR analysis to further

confirm the success of the cellulose extraction by referring to the presence of their functional groups.

**Table 3.1:** Composition of cellulose-based hydrogels with various gelling agents

| Types of gelling agents | Amount of gelling agent (g) | Amount of cellulose (g) | Volume of ECH (mL) |
|-------------------------|-----------------------------|-------------------------|--------------------|
| CMC                     | 0.25                        | 3                       | 5                  |
|                         | 0.50                        |                         |                    |
|                         | 0.75                        |                         |                    |
|                         | 1.00                        |                         |                    |
|                         | 1.25                        |                         |                    |
|                         | 1.50                        |                         |                    |
|                         | 1.75                        |                         |                    |
|                         | 2.00                        |                         |                    |
|                         | 2.25                        |                         |                    |
|                         | 2.50                        |                         |                    |
| AA                      | 0.25                        | 3                       | 5                  |
|                         | 0.50                        |                         |                    |

**Table 3.1:** Continued

|          |      |   |   |
|----------|------|---|---|
|          | 0.75 |   |   |
|          | 1.00 |   |   |
|          | 1.25 |   |   |
|          | 1.50 |   |   |
|          | 1.75 |   |   |
|          | 2.00 |   |   |
|          | 2.25 |   |   |
|          | 2.50 |   |   |
|          |      |   |   |
| Glycerol | 0.25 | 3 | 5 |
|          | 0.50 |   |   |
|          | 0.75 |   |   |
|          | 1.00 |   |   |
|          | 1.25 |   |   |
|          | 1.50 |   |   |
|          | 1.75 |   |   |
|          | 2.00 |   |   |
|          |      |   |   |

**Table 3.1:** Continued

|                       |      |   |   |
|-----------------------|------|---|---|
|                       | 2.25 |   |   |
|                       | 2.50 |   |   |
| PEG400/PEG600/PEG1000 | 0.25 | 3 | 5 |
|                       | 0.50 |   |   |
|                       | 0.75 |   |   |
|                       | 1.00 |   |   |
|                       | 1.25 |   |   |
|                       | 1.50 |   |   |
|                       | 1.75 |   |   |
|                       | 2.00 |   |   |
|                       | 2.25 |   |   |
|                       | 2.50 |   |   |

### 3.3 Characterization

#### 3.3.1 Field emission scanning electron microscopy (FESEM) analysis

Before running FESEM analysis, the small pieces of hydrogels were dried in the oven at 30°C until all the moisture had been removed, and the samples were placed in the desiccator with silica gel for 42 hours. The dried small pieces of hydrogels were stuck on the

metal plates and underwent metal coating. The surface morphology of each dried cellulose-based hydrogel with different concentrations of AA, CMC, glycerol, and PEG (0.25 g — 2.5 g) was observed using a Field Emission Scanning Electron Microscope (FESEM) (JEOLJSM-IT500HR) (Ahmad et al., 2023). The surface morphology of the specimen was observed at a magnification of 200×.

### 3.3.2 Fourier-transform infrared spectroscopy (FTIR) analysis

The small pieces of hydrogels were dried in the oven at 60 °C. The dried specimens were analysed using Fourier Transform Infrared (FTIR) Spectrometer. , Thermo Scientific Nicolet iS 10 (United States), within the wavenumber range of 400 to 500  $cm^{-1}$  using the ATR technique in absorbance mode (Ahmad et al., 2023). Each of the analyses was carried out in triplicate to increase the accuracy.

### 3.4 Swelling ratio

To determine the swelling ratio of each cellulose hydrogel with different concentrations (0.25 g - 2.5 g) of various gelling agents (AA, CMC, glycerol, and PEG), the samples were dried in the oven at 60 °C for 12 hours. The weight of the dried samples was measured and recorded. The dried samples were immersed in distilled water at 27°C with a thermometer to monitor the temperature of the distilled water. In 5 hours, the weight of each swollen sample was measured and recorded at every hour. To improve this experiment's accuracy, this experiment was repeated three times, and the average swelling of each hydrogel was calculated and recorded. Equation 3.1 was used to calculate the swelling ratio of the hydrogel (Chang et al., 2010).

$$\text{Swelling ratio} = \frac{W_s - W_d}{W_d} \times 100\%, \quad \text{Equation 3.1}$$

where  $W_s$  is the weight of swollen hydrogel while  $W_d$  is the weight of the dried hydrogel.

### 3.5 Water retention

To determine and compare the water capacity of the cellulose hydrogel with different concentrations (0.25g to 2.5g) of various gelling agents (AA, CMC, glycerol, and PEG), the hydrogels were soaked in distilled water at 27 °C with a thermometer. The specimens were taken out after achieving the swelling equilibrium in the water and were weighed as  $W_s$ . Each of the samples was placed in the open air at 31°C for 96 hours. The samples were weighted as  $W_t$  every 12 hours. To improve the accuracy of this experiment, the samples were triplicated, and the average water retention rate for each cellulose hydrogel was calculated and recorded. The water retention ratio (%) was calculated using Equation 3.2 (Chen et al., 2020).

$$\text{Water retention rate} = \frac{(W_t - W_d)}{(W_s - W_d)} \times 100\% \quad \text{Equation 3.2}$$

where  $W_t$  is the weight of the hydrogel every 12 hours;  $W_d$  is the weight of the dry hydrogel;  $W_s$  is the weight of the swollen hydrogels at 0 hour.

The hydrogels plasticised with four different gelling agents, which have excellent water uptake and retention capacities in this study, were selected, and their specific surface area, average pore size, and pore volume were determined and analysed by using the Brunauer-Emmett-Teller (BET) Surface Area Analyser (Quantachrome Autosorb iQ-AG) based on nitrogen gas sorption at 77 K and Quantachrome AS1Win software (Quantachrome Instruments Version 2.01) (Voon et al., 2017). 1g of the specimen was dried in the oven at 60 °C for 12 hours before the BET analysis. The dried specimens were degassed at 80°C for 6 hours.

### **3.6 Application of Cellulose-Based Hydrogel as a Soil Conditioner**

#### **3.6.1 Soil moisture of various types of soils**

Four types of soil (sandy soil, topsoil, gley soil and clayey soil) were collected from the Real Living Lab (RLL), Universiti Malaysia Sarawak (UNIMAS) (longitude: 1°28'09.3"N; latitude: 110°25'41.3"E), and were used in this study. Before use, all the soils were dried under the sun for 24 hours to avoid any water content. The dried soils were sieved using a 20 (840  $\mu\text{m}$ ) mesh size sieve to remove other debris like branches, stones, and root systems and the soil moisture of each dried soil was determined using a digital soil moisture meter (Extech Instruments-MO750) to make sure the soil moisture had been removed. Then, 250g of each cellulose hydrogel with different gelling agents, which had excellent performances in their water uptake and retention capacity, was added and mixed with 250g of each soil type for a minute before putting it into the planting bag (size of 6 cm $\times$ 6 cm $\times$ 6 cm) to ensure all hydrogels are evenly mixed with the soil. The soil moisture of each sample was measured in triplicate using a soil moisture meter (Extech Instruments-MO750) for 15 days. The average of each soil moisture was used to plot the graph of the soil moisture (x-axis = the observation period (days); y-axis = soil moisture of each of the soil samples (%))

#### **3.6.2 Soil pH and soil electrical conductivity of various types of soils**

2 g of soil samples from each planting bag were collected and put in centrifuge tubes with a volume of 50 mL, an internal diameter of 27mm and an external diameter of 29mm every 3 days within 15 days. Then, 10 ml of water was added to each centrifuge tube. The samples were hand shaken for a minute to ensure the soil samples and the water were mixed homogeneously. The pH and the electrical conductivity of the samples were determined using a portable pH/conductivity meter (Cole-Parmer PC100) during the observation period.

All the pH and electrical conductivity of each sample were collected in triplicate in three different areas, and the average of the readings was used to plot the graph of soil pH (x-axis = the observation period (days); y-axis = soil moisture of each of the soil samples (%)).

### 3.7 Seed germination assay in each hydrogel

This study also determined the seed germination rates of each hydrogel with different gelling agents, and this experiment was carried out in the lab. The vegetable seeds Choy Sum (*Brassica chinensis* var. *parachinensis*) were used to plant onto the hydrogel. The seeds were wetted with water for 4 hours before being used in this experiment. The wet cotton pad was used as the base for the control set of this experiment. Then, the hydrogels with different gelling agents were placed in the petri dishes. 20 seeds were added to these six conditions (on cotton pad and with watering; on cotton pad and without watering; on cellulose-1.75g CMC-based hydrogel and without watering; on cellulose-2g AA-based hydrogel and without watering; on cellulose-1g glycerol-based hydrogel and without watering; on cellulose-0.5g PEG600-based hydrogel and without watering) and each of the condition had five replications respectively to increase the accuracy of the result. The Petri dishes were placed in the lab at 31°C for 30 days without recording the humidity. The seed germination of each condition was observed and recorded daily until the end of the observation period. The germination rate will be calculated based on the International Seed Testing Association (ISTA, 1985) using Equation 3.3, where “a” is the total number of germinated seeds and “b” is the total number of seeds used.

$$\text{Germination Rate (\%)} = \frac{a}{b} \times 100 \% \quad \text{Equation 3.3}$$

Besides the germination rate of the Choy Sum vegetable seeds, the germination value (GV) was calculated using Equation 3.4, where DGS represents daily germination speed, and N represents the number of daily counts.

$$\text{Germination Value} = \frac{(\text{sum of the DGS})(N)}{\left(\frac{\text{final cumulative germination rate}}{10}\right)} \quad \text{Equation 3.4}$$

### 3.8 Biodegradability of each hydrogel with each gelling agent

The method reported by Laftah & Hashim (2013) was used in this study to test the biodegradability of the cellulose-based hydrogel with various gelling agents. The hydrogels were weighed and recorded before being buried in 5kg of normal garden soil (brand of Miracle Gro), known as the loamy soil (with an equal ratio of sand, silt, and clay soil). Then, the weighted hydrogels were buried 5 to 10 cm deep into the loamy soil. The compost materials attached to the surface of the hydrogel were cleaned by a brush before weighing the mass of the hydrogels on day 14, day 21, day 28, day 35, and day 60. The experiment stopped when the weight of the samples did not change. Equation 3.5 was used to calculate the weight loss of the hydrogel through the biodegradability test, where  $W_o$  is the initial weight of the hydrogel and  $W_i$  is the weight of the hydrogel on days 14, 21, 28, 35, and 60 (Laftah & Hashim, 2013).

$$\text{Weight loss (\%)} = \frac{W_o - W_i}{W_o} \times 100\% \quad \text{Equation 3.5}$$

### 3.9 Chapter Summary

This chapter covers the materials and instruments utilised in this research. It also describes the process of creating cellulose-based hydrogels with various gelling agents. This section also states the procedures for obtaining the hydrogels' characteristics, including their surface morphologies, chemical bonding, swelling, and capacity to retain water. This part

also includes information about the experiment's soil moisture, pH, electrical conductivity, seed germination, and biodegradability assay.

## CHAPTER 4

### RESULTS AND DISCUSSION

#### 4.1 Extraction of the cellulose

The wastepaper underwent a pretreatment process with an alkaline solution to remove the ink, hemicellulose, and lignin from the paper (Ghodake et al., 2018). After the alkaline pre-treatment process, the extracted cellulose became white powder in Figure 4.1, proved the ink on the wastepaper had been removed after one time of the alkaline pretreatment process.

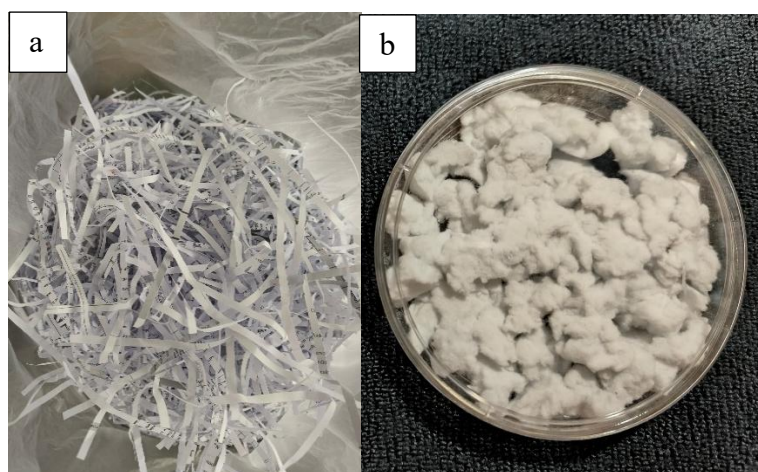


**Figure 4.1:** The comparison of the process before and after the alkaline pre-treatment process (a) the raw material before undergoing the alkaline pretreatment process;(b) extracted cellulose

The extracted cellulose functional groups were also determined using Fourier-transform infrared spectroscopy (FTIR) to verify that the cellulose had been extracted successfully from the wastepaper through the alkaline pre-treatment process. The FTIR result of the extracted cellulose was compared with the FTIR result of the commercial cellulose, which is shown in Figure 4.2. Based on the FTIR spectra in Figure 4.2, the extracted cellulose has similar functional groups to commercial cellulose. By comparing the

functional groups of both of the cellulose, both of the cellulose have a peak at  $3330.29\text{ cm}^{-1}$ , represented to the presence of -OH functional groups because the band at  $3330\text{ cm}^{-1}$  to  $3500\text{ cm}^{-1}$  is the spectra that are showing the intermolecular O-H stretching (Okwonna, 2013; Hanafiah et al., 2019). Besides, both types of cellulose show a peak at  $1025\text{ cm}^{-1}$  to  $1028\text{ cm}^{-1}$ , which indicates that the presence of C-O stretching in both of the chemical structures (Akalin & Pulat, 2018). In addition, the band at around  $894\text{ cm}^{-1}$  in the FTIR result of the wastepaper cellulose is also one of the peaks that indicates the cellulose extracted from the cellulosic material is a pure cellulose like commercial cellulose. Based on the study done by Okwonna (2013) and El-Sakhawy et al. (2018), the band at the range of  $849\text{ cm}^{-1}$  to  $894\text{ cm}^{-1}$  are the C-O-C pyranose ring vibration and the C-H out of plane ring stretching in cellulose due to beta-glycosidic linkages. Based on the studies done by Hanafiah et al. (2019) and Sisak et al. (2015), the raw material of cellulose always have the aryl alkyl ether in lignin and the C=O acetyl groups of hemicellulose ester present at the wavelength around  $1248\text{ cm}^{-1}$  and  $1735\text{ cm}^{-1}$ . By comparing the spectra of the extracted cellulose and raw wastepaper, the hemicellulose with the wavelength of  $1732\text{ cm}^{-1}$  (the C=O acetyl groups of hemicellulose) and  $1229\text{ cm}^{-1}$  (aryl alkyl ether in lignin) did not appear in the structure of the extracted fibres. On the FTIR spectrum, no more hemicellulose and lignin appear in the structure of the extracted cellulose.

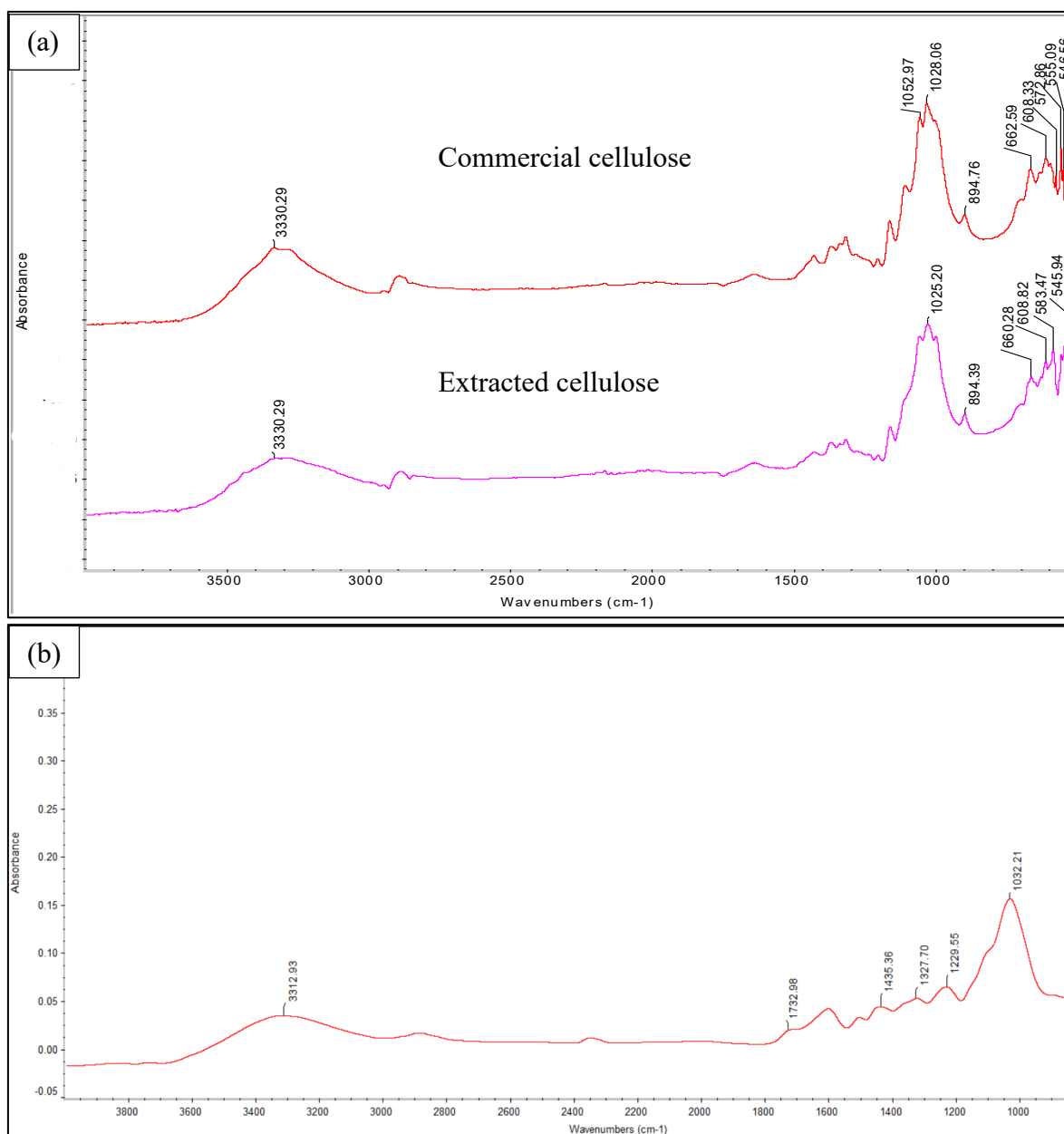


**Figure 4.2:** The FTIR spectra of (a) the cellulose extracted from wastepaper with the commercial cellulose and (b) the raw material of the wastepaper

In conclusion, the alkaline pre-treatment process successfully extracted cellulose from wastepaper. All the lignin and hemicellulose had been removed, and this was confirmed through the FTIR analysis by the absence of the functional groups of the lignin and hemicellulose in the spectrum of the extracted cellulose.

## **4.2 Preparation of cellulose-based hydrogel with various gelling agents**

The amount of cellulose used in the formation process of the cellulose hydrogel is 3g, as the hydrogel with this amount of cellulose fibre has high porosity in its surface morphology, which was proved in our previous study in Jong et al. (2024). The 3 g of cellulose must be dissolved in the precooled sodium hydroxide/urea (NU) solvent and frozen for 12 hours before adding the gelling agent and crosslinker into it (Jampi et al., 2021; Ahmad et al., 2023). This is because the intermolecular and intramolecular hydrogen bonds in the cellulose fibre can be broken in the NU solvent at a low temperature by producing a low degree of polymerisation, which helps the cellulose to dissolve completely (Zainal et al., 2021). So, the mixture of the NU with the cellulose will be homogenised after the freeze-thaw process, as in Figure 4.3. Furthermore, the amount of the crosslinker, which is epichlorohydrin (ECH), used in forming the cellulose-based hydrogel of this study, is 5 mL. Too little or too much crosslinking agent will cause the gelation process to be too low or too high. The crosslinked network between cellulose chains will prevent the hydrogel from forming or forming too tight, which means it will lose its elasticity (Mali et al., 2017; Salleh et al., 2018). Besides, a high amount of ECH will also cause the water uptake capacity to decrease due to the high crosslinking network among the cellulose chains, resulting in less elasticity (Hussein et al., 2017).

The cellulose-based hydrogels with ten concentrations of each gelling agents were synthesised successfully through chemical crosslinking. Besides, PEGs with different molecular weights of 400, 600, and 1000 were used in forming the cellulose-PEG-based hydrogels in this study. From this study's observation, the PEG with a molecular weight higher than 1000 cannot form a hydrogel because the gelling process took a long time in the NU solvent at 80°C. Thus, the cellulose chains coagulated in the NU solvent due to the high

temperature. By forming the cellulose-based hydrogel in this study, the gelling time of PEG to plasticise with the cellulose chains was increased from PEG of 400 molecular weight to 1000 molecular weight. This can lead to the conclusion that PEG with a molecular weight of 400 causes the fast polymerisation process with cellulose. Lower PEG's molecular weight made more functional chain ends available, raising the hydrogel's crosslink density. The increase in the hydrogel stiffness and reduction in the mesh size also cause an increase in the crosslinking density. The mesh size corresponds to the pore size in the hydrogel because it is the average distance between the crosslinks to determine the solutes permissible (Perera et al., 2018).

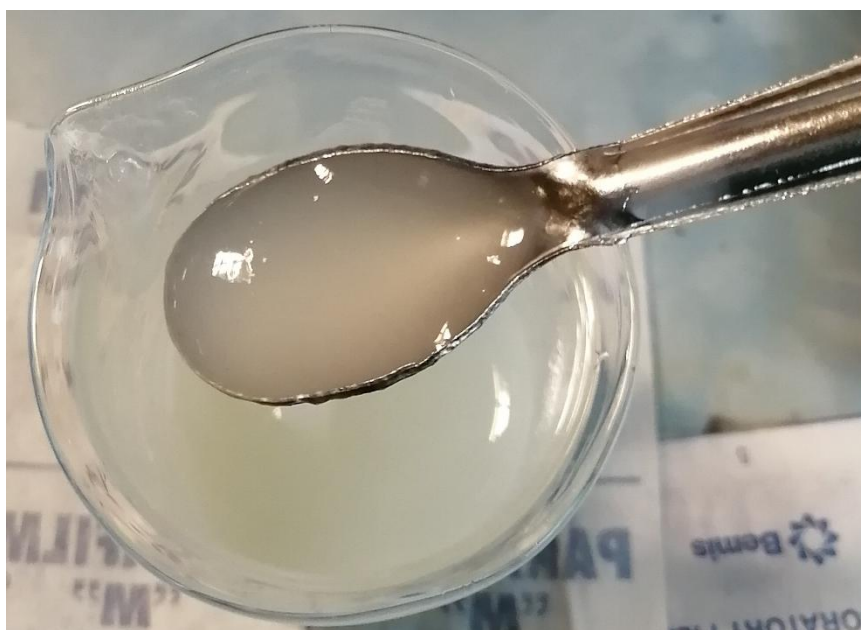


**Figure 4.3:** The cellulose fibre was mixed in the precooled NU solvent after a night (homogeneous mixture)

#### **4.3 Field emission scanning electron microscopy (FESEM) analysis**

In this section, the surface morphology of each cellulose-based hydrogel with different concentrations of each gelling agent, which are 0.25g to 2.50g, was determined by using a Field Emission Scanning Electron Microscope (FESEM) (JEOLJSM-IT500HR).

For the surface morphologies of the hydrogels with AA in Figure 4.4, the hydrogels with 1.25g, 1.50g, 1.75g, 2.25g, and 2.5g of AA have less porous surface morphologies compared with other hydrogels with other weights of AA. The size of the pores in the hydrogel decreased, and the amount of porosity increased when the amount of AA reached 2.0g, because the hydrogel with 0.25g to 1.75g of AA had larger pore sizes and the porosity decreased, but the hydrogel has more pores of a small size when with 2.0g of AA. The cellulose hydrogel with 2.0g of AA also has high porosity with a small size, but its pore walls are thin, based on the study done by Chang et al. (2009), which is different from this study. This may be because the amount of ECH used in this study is higher, causing the high crosslinking along the cellulose chains and AA chains by ECH, resulting in the thicker pore wall.

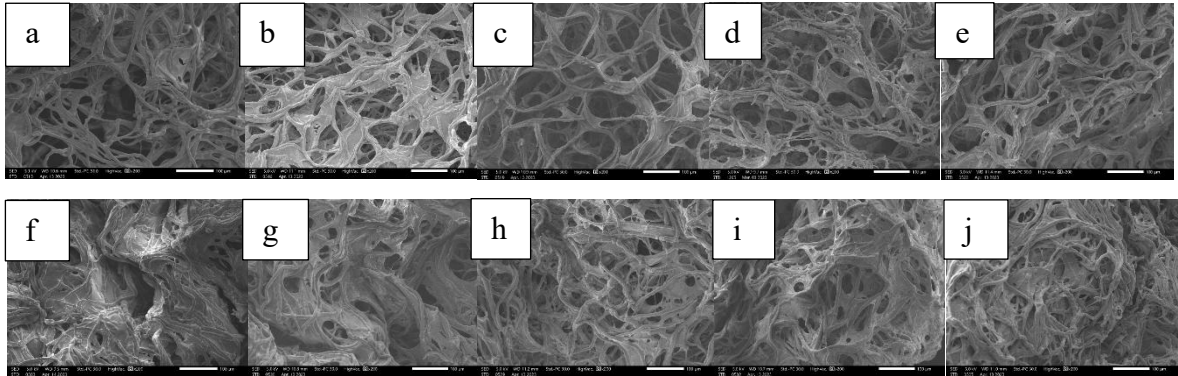


**Figure 4.4:** The surface morphology of the cellulose-based hydrogel with (a) 0.25g; (b) 0.5g; (c) 0.75g; (d) 1.0g; (e) 1.25g; (f) 1.5g; (g) 1.75g; (h) 2.0g; (i) 2.25g; (j) 2.5g of AA at magnifications of 200×

The porosity of the hydrogels with CMC in Figure 4.5 decreased from 0.25 g to 1.5 g, but the porosity increased when CMC reached 1.75g. It can prove that the porosity of the hydrogel improved when the amount of CMC reached the optimum amount, which is 1.75g. Another study found out the cellulose hydrogel with this amount of CMC had high porosity with high swelling ability is the study done by Ahmad et al. (2023). While the porosity

decreased when the amount of CMC more than 1.75g because the increase of the concentration of CMC causes higher crosslinking in the hydrogel polymeric structure and increase the gel fraction, thus decreasing in the porosity of the hydrogel (Salleh et al., 2019).

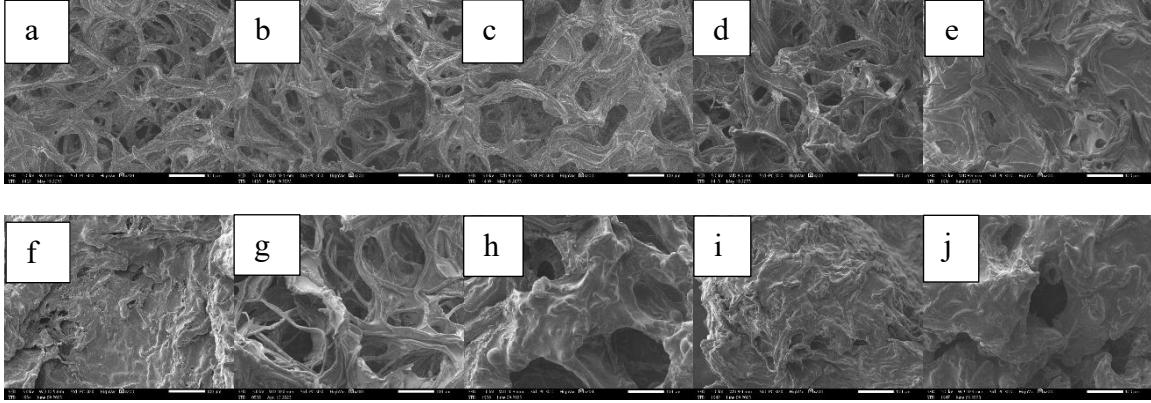


**Figure 4.5:** The surface morphology of the cellulose-based hydrogel with (a) 0.25g; (b) 0.5g; (c) 0.75g; (d) 1.0g; (e) 1.25g; (f) 1.5g; (g) 1.75g; (h) 2.0g; (i) 2.25g; (j) 2.5g of CMC at magnifications of 200×

For the surface morphologies of the hydrogels with 0.25 wt% to 2.50 wt% of glycerol, the interpenetrating network shows high porosity when the dosages of glycerol increase, except for the hydrogels with 0.50 wt% and 1.75 wt% of glycerol. The crosslinking process in the hydrogels may increase when the amount of glycerol increases, improving the effective crosslinking density of the cellulose hydrogels (Zhu et al., 2018). The hydrogels with 1.75 wt% of glycerol only have a few nano-sized pores in their structure. In contrast, the hydrogels with 0.50 wt% of glycerol have only fewer big pores in the intermolecular structure, as shown in Figure 4.6.

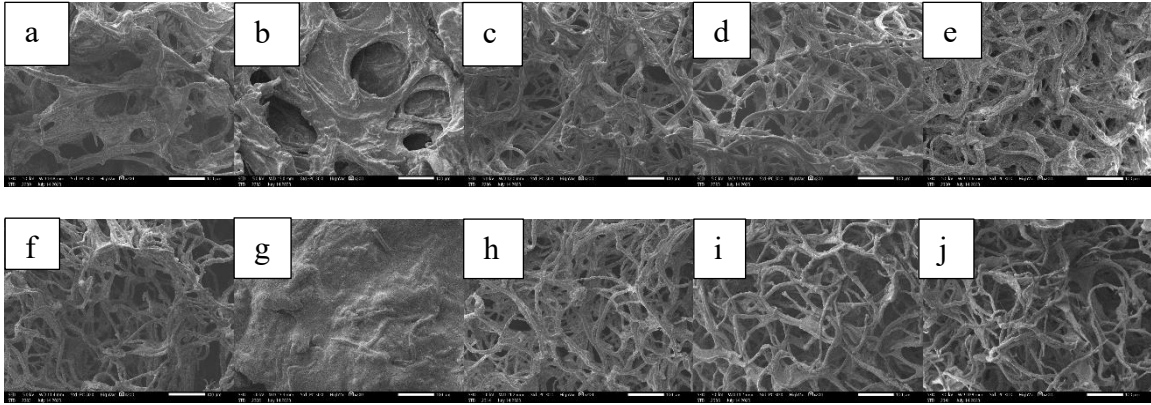


**Figure 4.6:** The surface morphology of the cellulose-based hydrogel with (a) 0.25g; (b) 0.5g; (c) 0.75g; (d) 1.0g; (e) 1.25g; (f) 1.5g; (g) 1.75g; (h) 2.0g; (i) 2.25g; (j) 2.5g of glycerol at magnifications of 200×

PEG is a polymer with water-soluble ability because of the repeating groups of  $-\text{CH}_2\text{CH}_2\text{O}-$  with two  $-\text{OH}$  groups (Yacob et al., 2021). The PEG with a molecular weight of 600 and below will remain in a liquid state at room temperature, while the molecular weight above 600 will stay in a solid state at room temperature. To determine the effect of the PEG with different molecular weights on the formation of the hydrogel, the PEG with molecular weights of 400, 600, and 1000 were used to prepare the cellulose-based hydrogel in this study. The FESEM surface morphology of the hydrogel containing PEG with molecular weights of 400, 600, and 1000 is displayed in Figures 4.7 to 4.9.

First of all, the hydrogel containing 0.50g to 0.75g of PEG with molecular weights of 400 and 600 formed a high porosity in Figures 4.7 and 4.8, and this is the confirmation that these three amounts of PEG400 and PEG600 are the optimum amount to produce a high porosity hydrogel. The hydrogel with others amount of PEG 400 have less and even no pores in the structures. This is because PEG, with this molecular weight, has many hydroxyl groups in its intermolecular structure, which makes the cellulose chains, PEG, and ECH stick together tightly, giving the hydrogels a high degree of crosslinking and decreasing the mobility of the polymer chains, which have a stiffer structure. (Yacob et al., 2021).

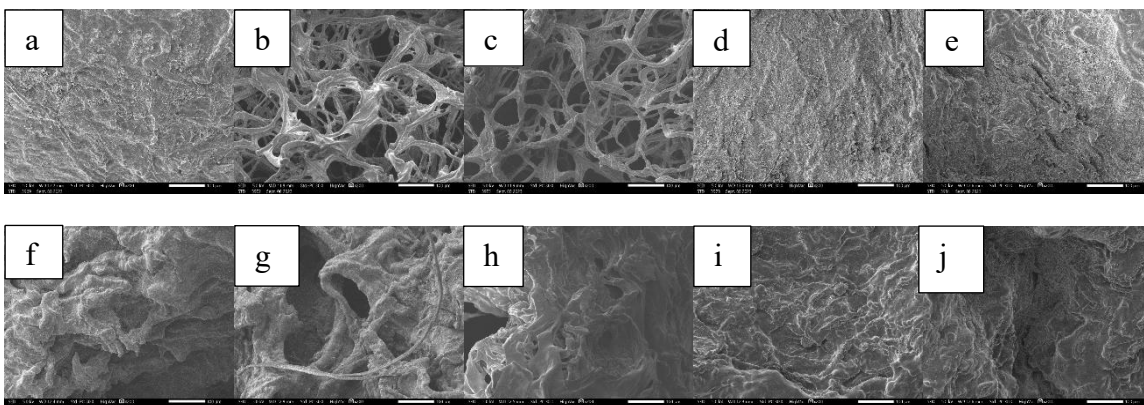


**Figure 4.7:** The surface morphology of the cellulose-based hydrogel with (a) 0.25g; (b) 0.5g; (c) 0.75g; (d) 1.0g; (e) 1.25g; (f) 1.5g; (g) 1.75g; (h) 2.0g; (i) 2.25g; (j) 2.5g of PEG400 at magnifications of 200×

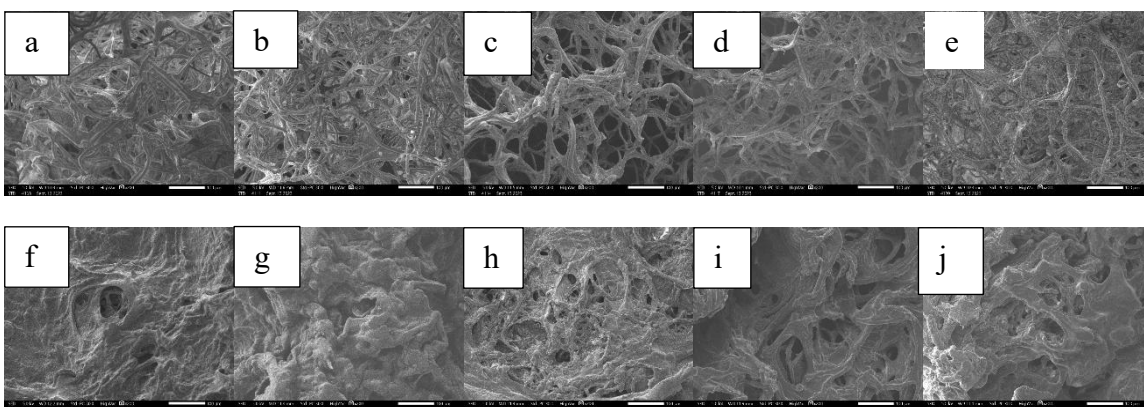


**Figure 4.8:** The surface morphology of the cellulose-based hydrogel with (a) 0.25g; (b) 0.5g; (c) 0.75g; (d) 1.0g; (e) 1.25g; (f) 1.5g; (g) 1.75g; (h) 2.0g; (i) 2.25g; (j) 2.5g of PEG600 at magnifications of 200×

In addition to the effect of PEG 400 on the formation of cellulose-based hydrogel, PEG 600 and PEG 1000 also caused some changes in the formation of the cellulose-based hydrogel. The increase of PEG600, a few of the pores were available in the hydrogel's structure, as shown in Figure 4.8. The hydrogel with PEG1000 works in another way, as shown in Figure 4.9. The hydrogel with a low concentration of PEG1000 has fewer pores, but when the amount of PEG1000 is increased, the porosity also increases.

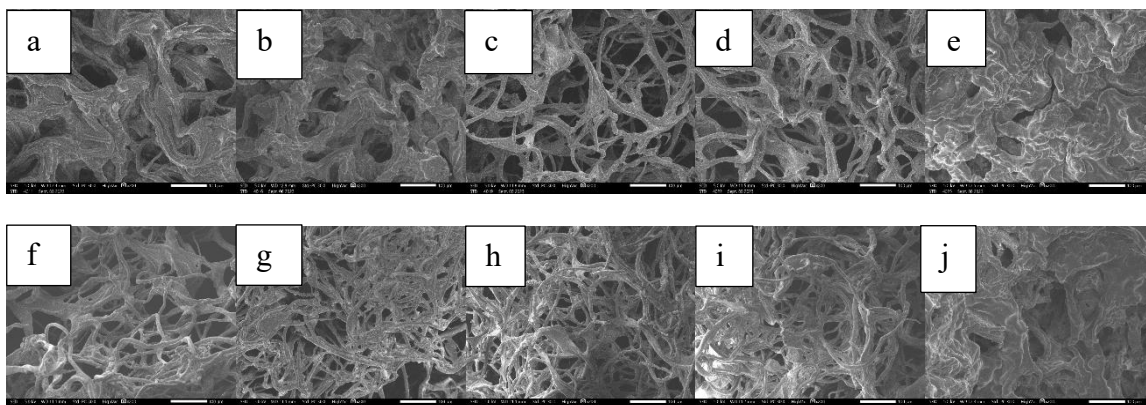


**Figure 4.9:** The surface morphology of the cellulose-based hydrogel with (a) 0.25g; (b) 0.5g; (c) 0.75g; (d) 1.0g; (e) 1.25g; (f) 1.5g; (g) 1.75g; (h) 2.0g; (i) 2.25g; (j) 2.5g of PEG600 at magnifications of 200×

In conclusion, the cellulose hydrogels containing 2.0g of AA and 1.75g of CMC have high porosity compared to the other amounts of AA and CMC. Thus, these two amounts of AA and CMC are ideal for forming a hydrogel with high porosity. All cellulose hydrogels with glycerol have more pores in the structure, except those with 0.5g and 1.75g glycerol. Moreover, the hydrogels with PEG400 and PEG600 have weak porosity at high concentrations of both PEGs, but the hydrogel with PEG1000 has the opposite side.

#### 4.4 Fourier-transform infrared spectroscopy (FTIR) analysis

This study also determined the functional groups of each cellulose hydrogel using the Fourier-transform infrared spectroscopy (FTIR) to ensure which functional groups are available in the crosslinking network structure of each hydrogel. All the hydrogels that underwent the FTIR analysis were kept in the same concentration of gelling agents, which is 1.75g.

Firstly, the peak at  $3293.62\text{ cm}^{-1}$  in the cellulose-1.75 g-AA based hydrogel (Figure 4.10 (b)) was referred to the functional group of OH. Through the chemical structure in Figure 4.10 (a), these hydroxyl groups were from cellulose, AA, and ECH chains, but mainly

from the cellulose chains because cellulose, as in the polysaccharide polymer chains, has numerous hydroxyl groups. This is also stated by Ahmad et al. (2023) that the broad peak at around  $3360\text{ cm}^{-1}$  resulted from the stretching vibration of the hydroxyl group in polysaccharides, encompassing both intermolecular and intramolecular hydrogen bond vibrations in cellulose. Besides, the wavelength at  $2881\text{ cm}^{-1}$  represented the C-H stretching in the hydrogel. This C-H bonding refers to the aliphatic CH bonding (Salleh et al., 2019). The peak around  $1609\text{ cm}^{-1}$  in the hydrogel's spectrum confirms the availability of carboxyl groups in its structure, which can be observed in the chemical structure shown in Figure 4.10(a) (Ahmad et al., 2023; Salleh et al., 2019). However, the peak intensity of the carboxyl group of the hydrogel done by Baysal et al. (2013) is at  $1627\text{ cm}^{-1}$  maybe because the raw material used is chitosan, and AA are more willing to attach to the chitosan chains compared to cellulose chains. Furthermore, the presence of the OH bending in the carboxylic group in AA was determined at  $1413.95\text{ cm}^{-1}$  in the hydrogel's spectrum. The wavelength indicated the epoxy group of ECH should be at around  $914\text{ cm}^{-1}$ , but there is no peak at this wavelength range because the epoxy group of ECH was broken, and a new bond was formed with the carboxylate group in AA and a hydrogen ion. This statement resulted in ECH successfully crosslinking the cellulose and AA. In addition, the C-O-C bonding between AA and cellulose was also determined at the wavelength  $1019.51\text{ cm}^{-1}$ . A small peak at  $897.10\text{ cm}^{-1}$  represented the C-O-C bonding ( $\beta$ -(1-4)-glycosidic bond in cellulose).

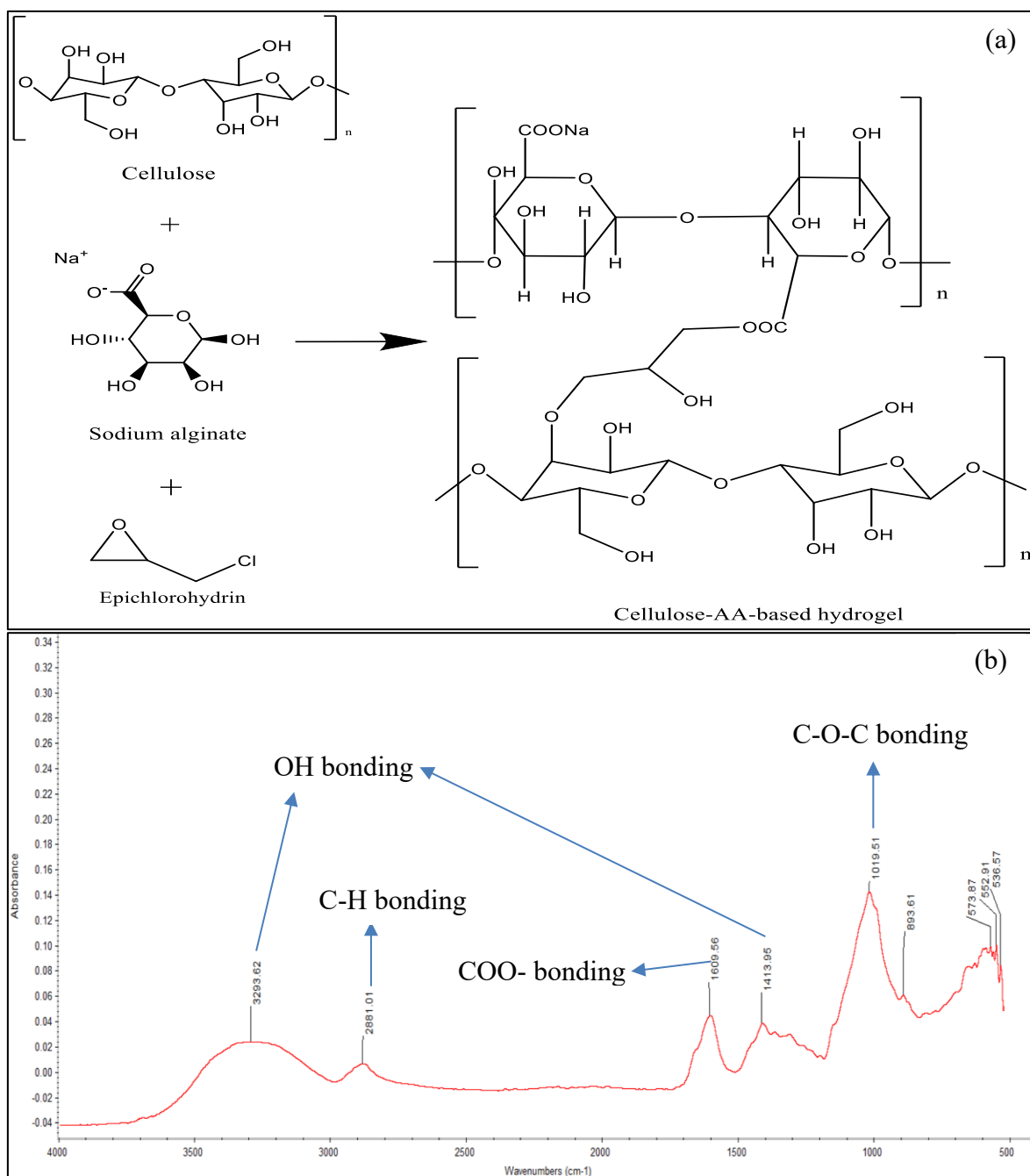


**Figure 4.10:** The comparison between (a) the chemical mechanism and (b) the FTIR spectrum of the cellulose-AA-based hydrogel

Based on Figure 4.11 (b), the peak of the cellulose-CMC-based hydrogel at 3297.10  $\text{cm}^{-1}$  corresponds to the hydroxyl functional groups that resulted in strong intermolecular hydrogen bonding in the structure of the hydrogel in the cellulose-CMC-based hydrogel. This also indicated the -OH bonding in the cellulose and CMC in the hydrogel polymeric

structure. Besides, the wavelength at  $1591.03\text{ cm}^{-1}$  in the cellulose-CMC-based hydrogel represented by the carboxyl groups in CMC are present in the hydrogel (Chang, Zhang, et al., 2010). The presence of the CH stretching and CH banding (aromatic ring of cellulose and CMC) was also observed at the wavelengths of  $2869.66\text{ cm}^{-1}$  and  $1663.32\text{ cm}^{-1}$ . Besides, the peak at around  $1413\text{ cm}^{-1}$  belonged to the stretching vibration of  $\text{COO}^-$  asymmetric (Salleh et al., 2019). The wavelength observed in the cellulose hydrogel at  $1365\text{ cm}^{-1}$  confirms that the crosslinking process successfully occurred with the C-O-C bonding between the polymers (Salleh et al., 2019). In addition, no peak appears at around  $914\text{ cm}^{-1}$  (the epoxy group in ECH), so it can be concluded that the epoxy bonding in ECH had broken and bonded to the hydroxyl group in the cellulose chain and the hydrogen ion from the water molecule, which shown in Figure 4.11 (a). The cellulose's C-O-C bonding  $\beta$ -(1-4)-glycosidic bond was observed at  $893.93\text{ cm}^{-1}$ .

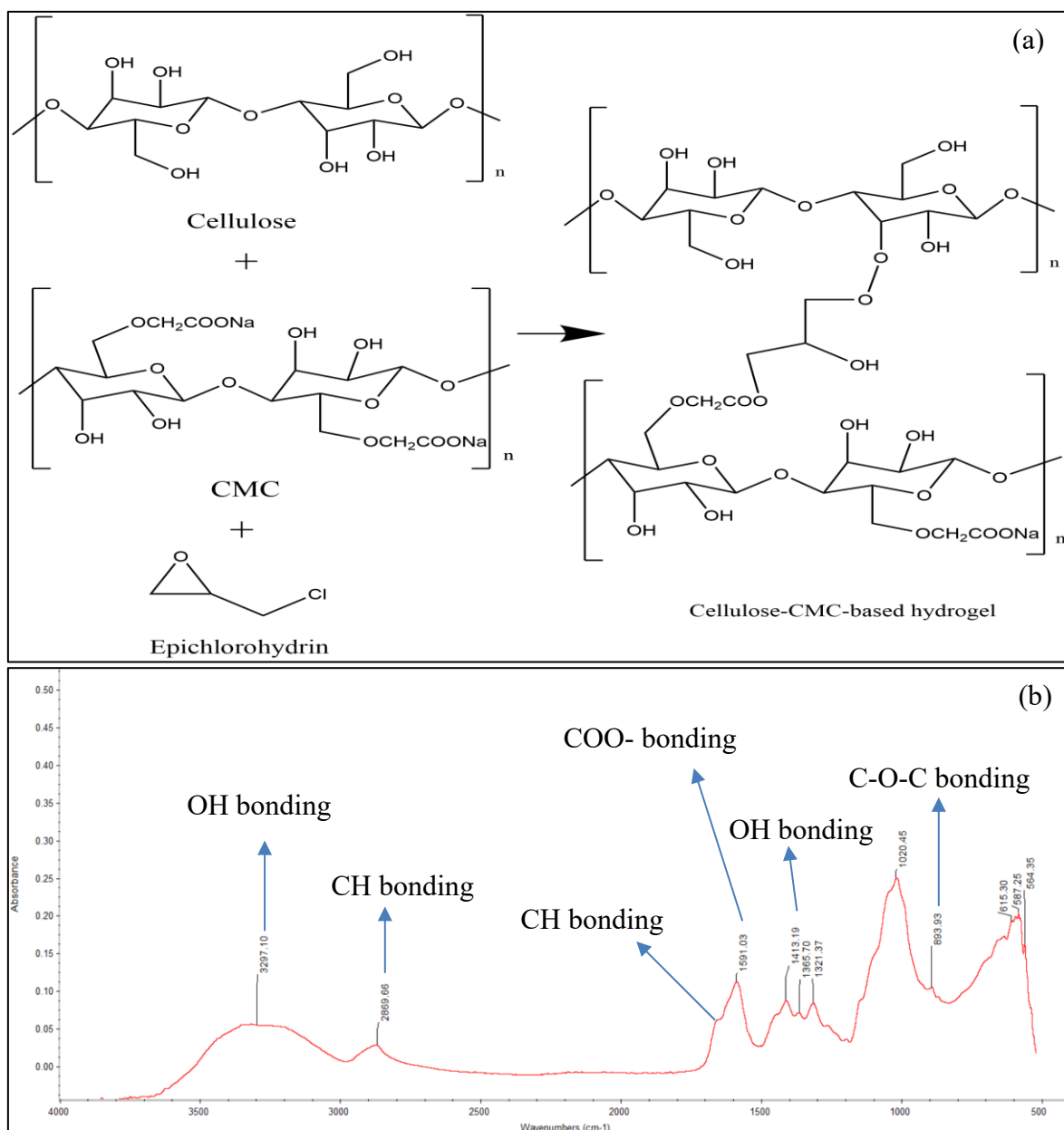


**Figure 4.11:** The comparison between (a) the chemical mechanism and (b) the FTIR spectrum of the cellulose-CMC-based hydrogel

In the spectrum of the cellulose hydrogel that was plasticised with glycerol in Figure 4.12 (b), there is a broad peak observed at 3363.59 cm<sup>-1</sup>, which represents the -OH stretching bonding and indicates that a high concentration of hydroxyl groups is available in its structure, which shown in Figure 4.12 (a). This certifies that the gelation process of glycerol with cellulose chains is available due to the hydrophilic nature of the glycerol and cellulose (Basiak et al., 2018). Besides, the wavelength at around 2923 cm<sup>-1</sup> and 2850 cm<sup>-1</sup> are the

C-H stretching of the aliphatic groups ( $-\text{CH}_2$  and  $-\text{CH}_3$ ) (Khotsaeng et al., 2023; Wang et al., 2016). The aliphatic ether ( $-\text{CO}$  stretching) was observed at around  $1022\text{ cm}^{-1}$ .

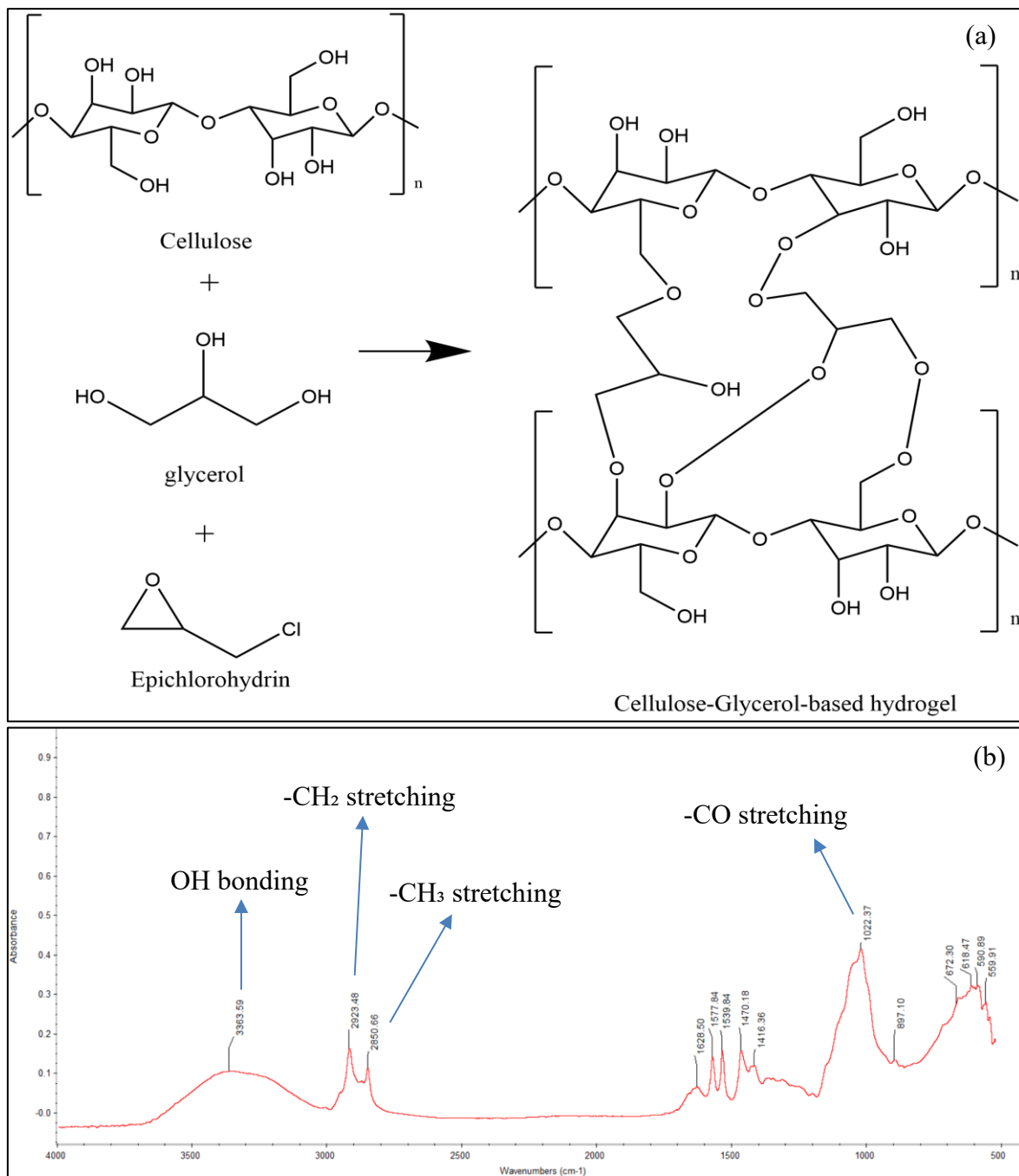


**Figure 4.12:** The comparison between (a) the chemical mechanism and (b) the FTIR spectrum of the cellulose-glycerol-based hydrogel

For the FTIR analysis of the cellulose-PEG600-based hydrogel in Figure 4.13 (b), the hydrogel has an absorption peak at  $3330\text{ cm}^{-1}$ , which represents the OH stretching

available in the hydrogel in Figure 4.13 (a) through either self-association or entanglement of cellulose chains through the hydroxyl bond between the reaction of cellulose and ECH (Jampi et al., 2021). Besides, the wavelength in the spectra of PEG at around  $2850\text{ cm}^{-1}$  represented the symmetric stretching vibration of  $\text{CH}_2$ . The wavelength at  $1417\text{ cm}^{-1}$  also observed in the hydrogel's spectrum in Figure 4.13 (b), which is the scissoring bending vibration of  $\text{CH}_2$  and have the same peak as the hydrogel with PEG in the study was done by Kono (2014). The anomeric carbon of  $\beta$ -D-glucopyranosyl of cellulose was determined at the wavelength of  $897.42\text{ cm}^{-1}$ .

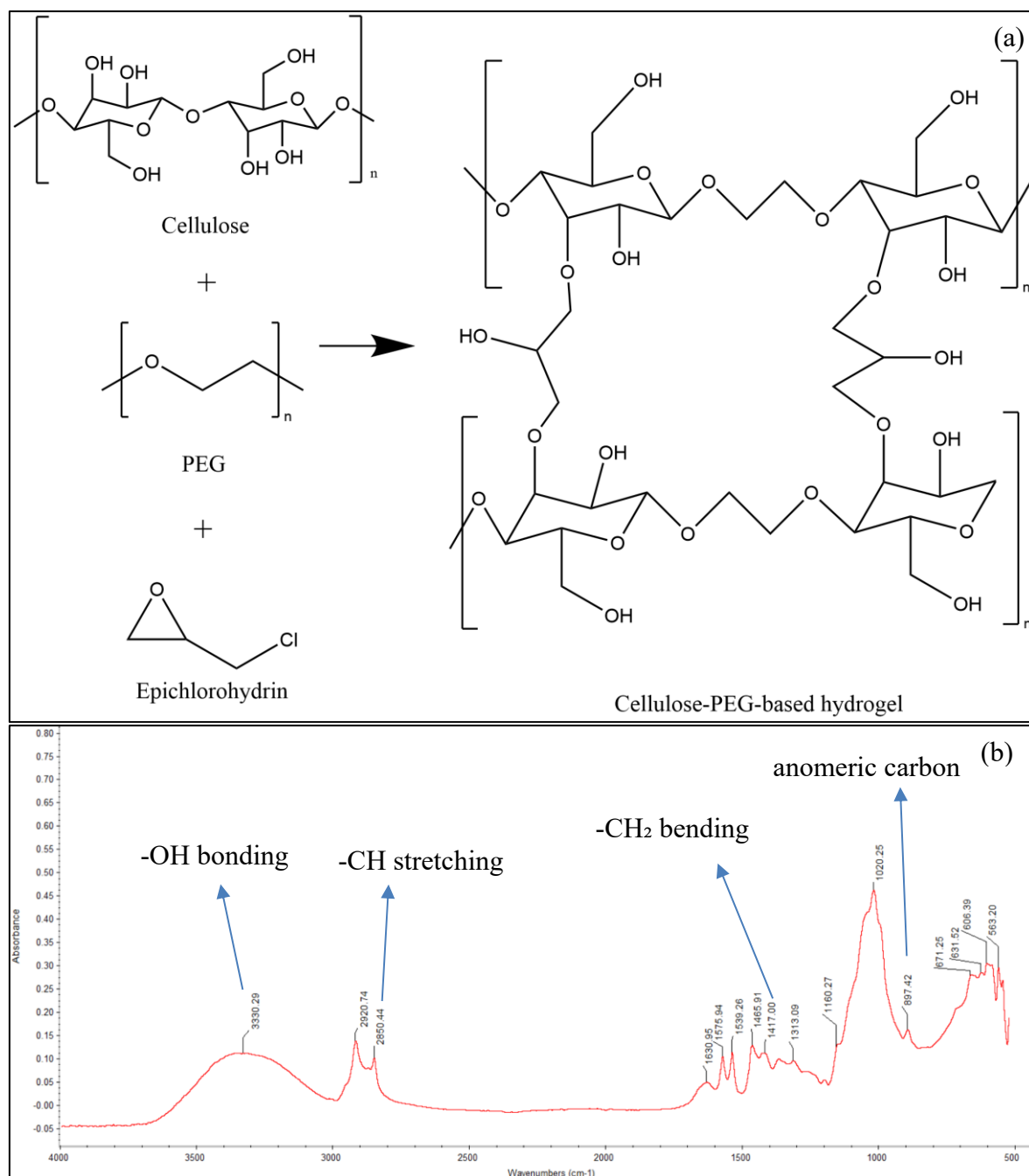


**Figure 4.13:** The comparison between (a) the chemical mechanism and (b) the FTIR spectrum of the cellulose-PEG600-based hydrogel

The FTIR spectra of the cellulose-based hydrogel, which was plasticised with PEG with different molecular weights, were also compared in Figure 4.14. From the spectra, the peak intensity in the hydrogel which plasticised with the PEG with a molecular weight of 400 are higher than another hydrogel plasticised with 600 and 1000 molecular weight of

PEG, especially of the absorption peak at  $2918\text{ cm}^{-1}$ ,  $2849\text{ cm}^{-1}$ ,  $1466\text{ cm}^{-1}$ , and  $1418\text{ cm}^{-1}$  which is the asymmetric stretching vibration of aliphatic  $\text{CH}_2$ , symmetric stretching vibration  $\text{CH}_2$ , the C-H bending vibration, scissoring bending vibration of  $\text{CH}_2$  (Kono, 2014; Yacob et al., 2021). The high peak intensity indicates a higher concentration of those functional groups present in the hydrogel structure.

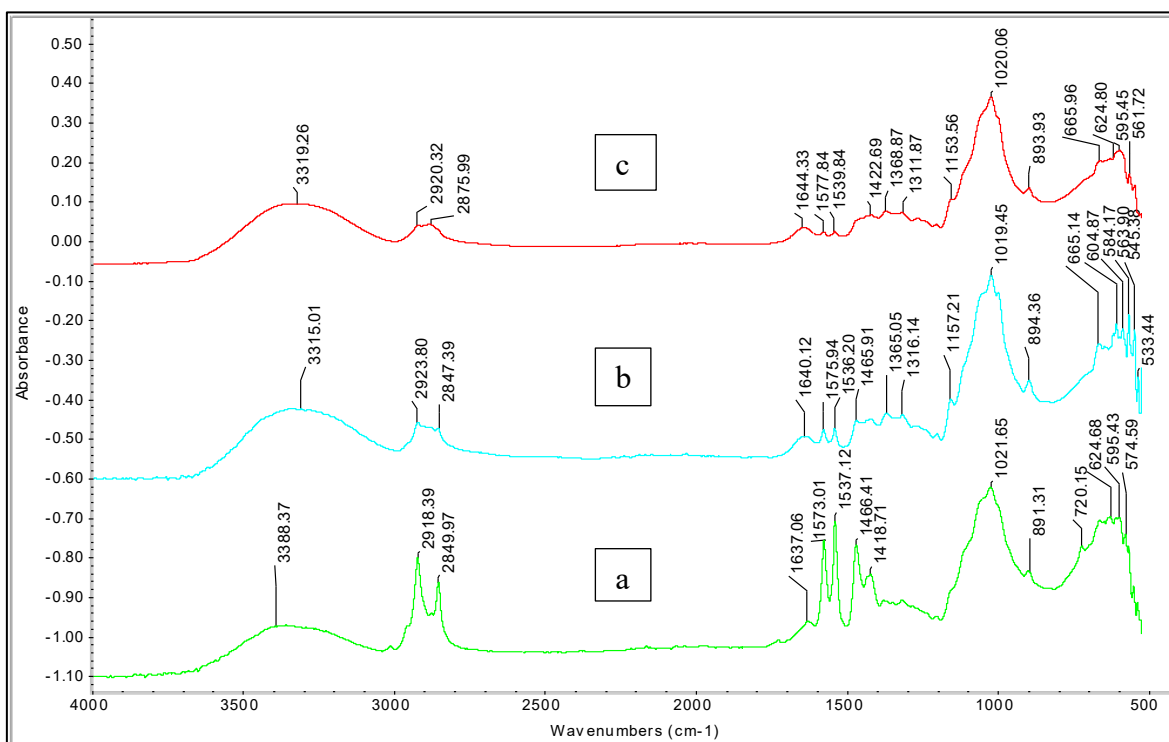


**Figure 4.14:** The FTIR spectra of the cellulose-PEG-based hydrogel plasticised with different molecular weights of PEG, which are (a) PEG 400, (b) PEG 600, and (c) PEG 1000

In conclusion, all the hydrogels in this study were successfully synthesised due to the presence of the functional groups, as stated in each of the hydrogels' chemical structures. The hydrogels with glycerol and PEG did not have the hydrophilic functional groups of carboxylate groups because the chemical structure of glycerol and PEG lacked carboxylic groups. Besides, the epoxy group of ECH in all hydrogels do not appear because the epoxy compound has broken and formed new bonds with the cellulose and gelling agents.

#### **4.5 Swelling ratio of cellulose-based hydrogels**

Based on Figure 4.15, the cellulose-based hydrogels with 2.0 g of AA have the highest swelling ratio compared to other concentrations after 3 hours, and the graph trend shows a continuous increase every 30 minutes. The hydrogel at this concentration of AA has the highest swelling ratio after 5 hours, which is 808.30 %. By linking the swelling ratio data with the surface morphology of the cellulose-based hydrogels plasticised with AA (Figure 4.4) in this study, the hydrogels plasticised with 2.0 g of AA have lots of smaller pores and have a better swelling ratio compared to the hydrogels with other concentrations of AA, so numerous of small pores provided more empty spaces for the water molecular to fill in. Besides, the high porosity of the hydrogels caused an increase in the amount of water uptake due to the improved chain mobility, which helps the network expansion (Vishal Gupta & Shivakumar, 2012). The hydroxyl and carboxyl groups in the cellulose-2.0 g AA-based hydrogel's structure also affected the swelling ratio by forming hydrogen bonds with water molecules. Hence, this hydrogel can absorb water into its structure.

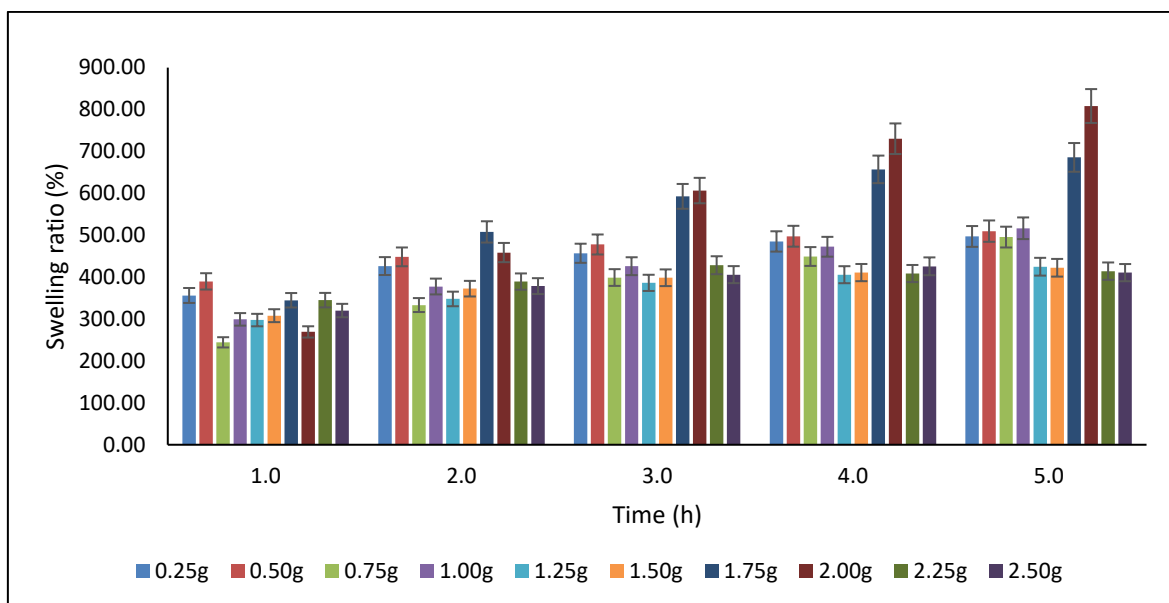


**Figure 4.15:** The swelling ratio of cellulose-based hydrogels with AA (0.25 g - 2.50 g). The error bar represents the standard deviations from the triplicate (n=3)

For the swelling ability of the hydrogels with different concentrations of CMC (Figure 4.16), the swelling ratios of these hydrogels with 0.25 g, 0.50 g, 0.75 g, 1.0 g, and 1.75 g of CMC are around 2100 % to 2600 %. From there, the cellulose-CMC-based hydrogels in these dosages of CMC have better water absorption due to their high porosity structures by observing their surface morphologies, as shown in Figure 4.5. So, these hydrogels have more space for the water to fill in. The cellulose-CMC-based hydrogels also have a higher swelling ability than the cellulose-AA-based hydrogels because CMC provides many carboxylic groups besides hydroxyl groups when it acts as the gelling agent. Both functional groups are hydrophilic functional groups in forming hydrogels, as shown in Figure 4.11, which have high peak intensity in the hydroxyl group and carboxyl group in the FTIR spectra. Hence, the more water is attached to the cellulose-CMC-based hydrogel via hydrogen bonds. The swelling ratio of the hydrogels with a concentration of CMC above 1.75 g has a weaker water uptake capacity because the increase in the viscosity of the hydrogels limits the movement of the polymer, causing the loss of the hydrogel (Salleh et

al., 2019). The Flory–Rehner theory can also be applied to describe the water uptake ability of the hydrogel, where the interplay between osmotic pressure and elastic restorative force influences the equilibrium volume of the cellulose hydrogel networks (Ahmad et al., 2023).

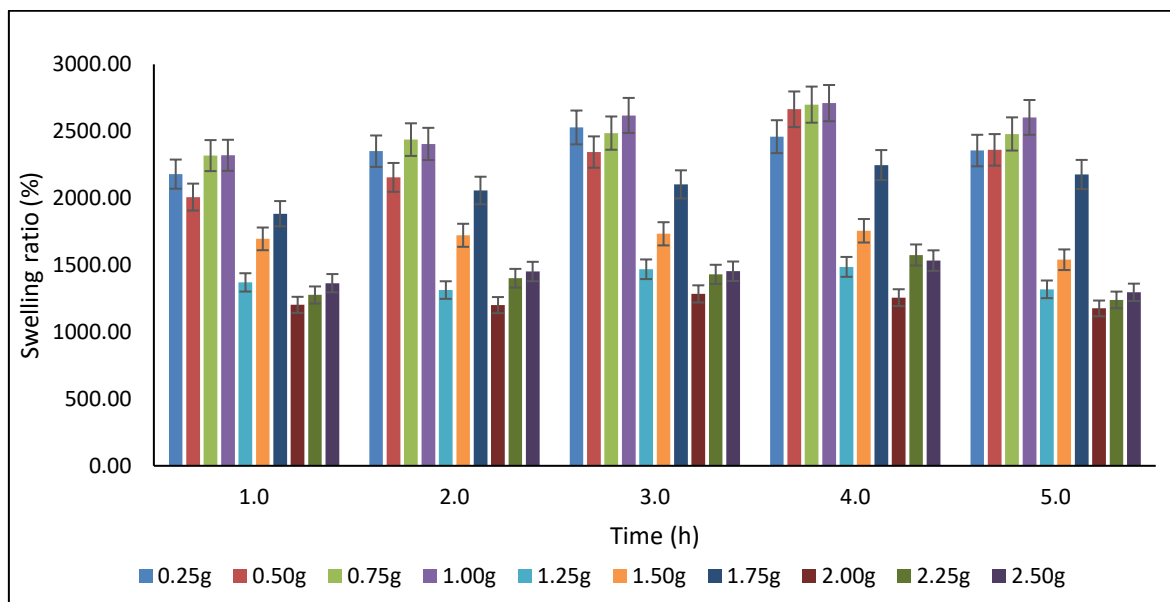


**Figure 4.16:** The swelling ratio of cellulose-based hydrogels with CMC (0.25 g - 2.50 g). The error bar represents the standard deviations from the triplicate (n=3)

Besides the swelling ratio of the hydrogels plasticised with AA and CMC, the cellulose-glycerol-based hydrogels' swelling ratios with various glycerol concentrations were also determined and shown in Figure 4.17. The hydrogels' water uptake capacity with these two glycerol concentrations, which are 0.50 g and 1.75 g, is only 1457.87 % and 969.68 %, respectively. These two hydrogels' intermolecular structure networks have fewer pores in their surface morphologies, as shown in Figure 4.6, indicating that less spacing is available for the water. Moreover, the hydrogels with glycerol concentrations other than 0.50 g and 1.75 g have great water uptake capacity, ranging from 2680 % to 4190 %. This shows cellulose-glycerol-based hydrogels have better water uptake ability than cellulose-AA-based and cellulose-CMC-based hydrogels, which can only reach their highest levels of 808.30 % and 2603.24 %, respectively. The maximum swelling ratio of the cellulose-glycerol-based

hydrogel can be reached at 4192.17 %. This is because glycerol can maintain the viscosity of the hydrogel and promote high flexibility for the cellulose chain networks, therefore causing the elasticity of the hydrogel to increase, expand and absorb more water (Zhu et al., 2018).

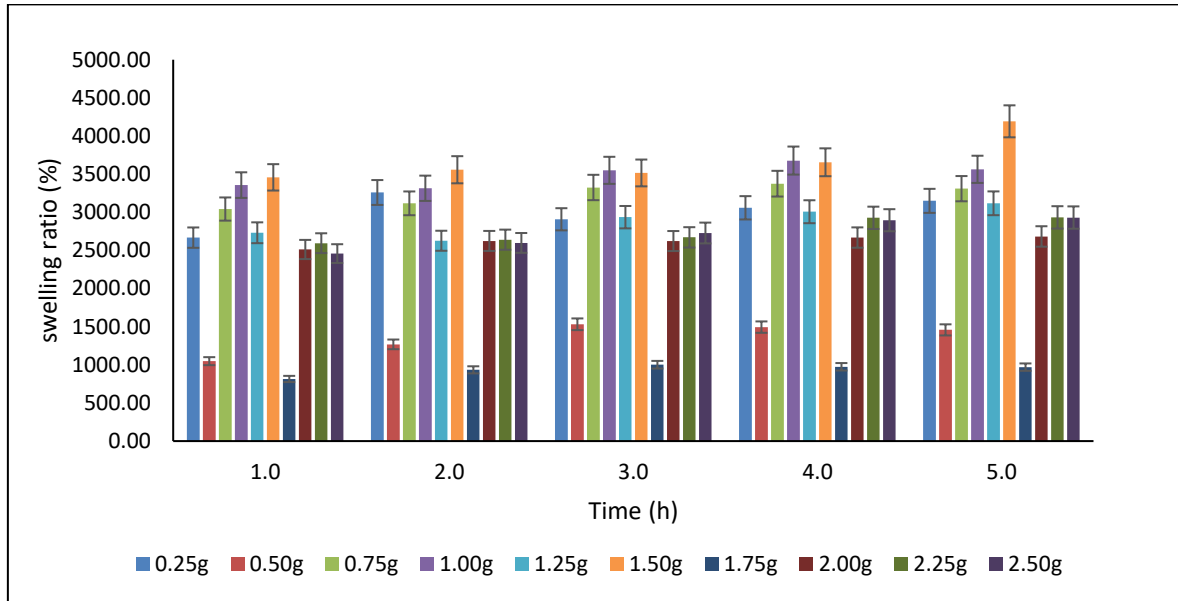


**Figure 4.17:** The swelling ratio of cellulose-based hydrogels with glycerol (0.25 g - 2.50 g). The error bar represents the standard deviations from the triplicate (n=3)

The hydrogels with lower PEG 400 and PEG 600 concentrations have higher swelling ratios after 5 hours, as shown in Figures 4.18 and 4.19. The swelling ratios of the hydrogel with 0.5g and 0.75g of PEG400 are at 2461.56% and 2460.33%. Meanwhile, the swelling ratio of PEG600, with 0.25g to 1.25g, is 2424% to 2500%. This may be because fewer unreacted or free PEG chains exist in the hydrogel's structure when PEG's molecular weight decreases. The larger PEG molecules will be caused by the enormous steric hindrance, which blocks the cellulose chains and crosslinker from binding to the ends of the molecules (Mikos et al., 2002). On the other hand, the hydrogels with PEG1000 with these six concentrations (0.75g, 1g, 1.5g, 1.75g, 2g, and 2.25g) have swelling ratios ranging from 2621% to 2823%, as shown in Figure 4.20. The increase in swelling ratio is because when

the molecular weight of PEG increases, the hydrogel with PEG1000 has the ether bonding from the PEG molecular chains, which increases the water affinity of the hydrogel, resulting in high water uptake capacity (Lee et al., 2020). However, the study done by Gao et al. (2021) had the opposite view on this, which stated that the increase in the molecular weight of PEG directly caused the decrease of the swelling due to the rigidity of the hydrogel and reduces the mobility of the molecular chains, causing numerous –OH groups to become integrated within the molecular structure, forming hydrogen bonds. This results in a reduction of their degrees of freedom and subsequently decreases the hydrophilicity of the hydrogel.

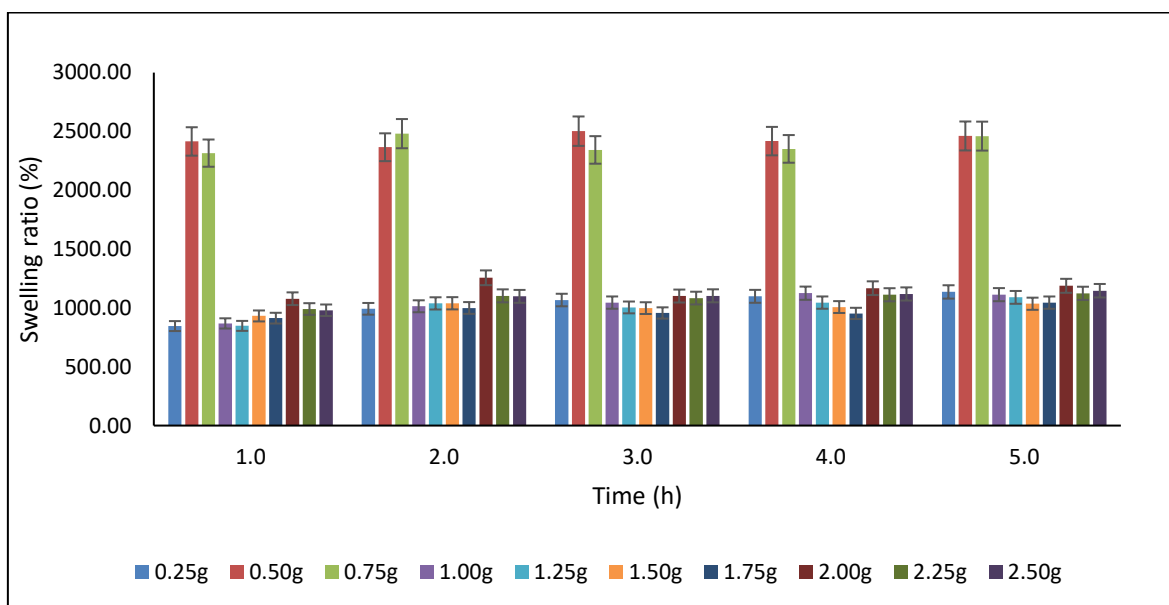


**Figure 4.18:** The swelling ratio of the cellulose-based hydrogel with PEG400 (0.25 g - 2.50 g). The error bar represents the standard deviations from the triplicate (n=3)

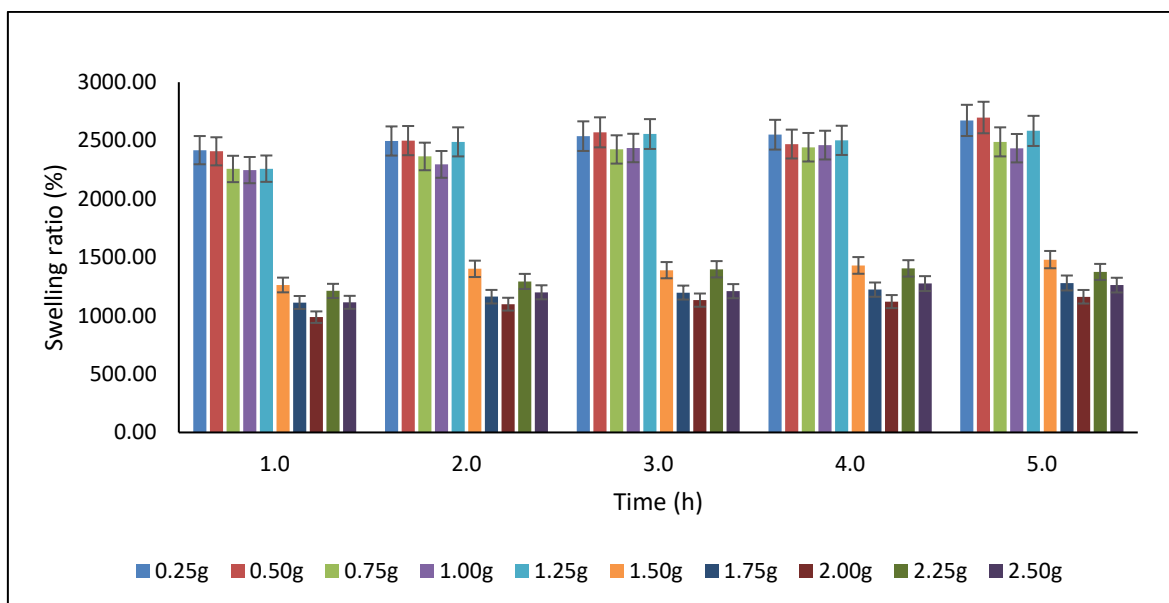


**Figure 4.19:** The swelling ratio of the cellulose-based hydrogel with PEG600 (0.25 g - 2.50 g). The error bar represents the standard deviations from the triplicate (n=3)

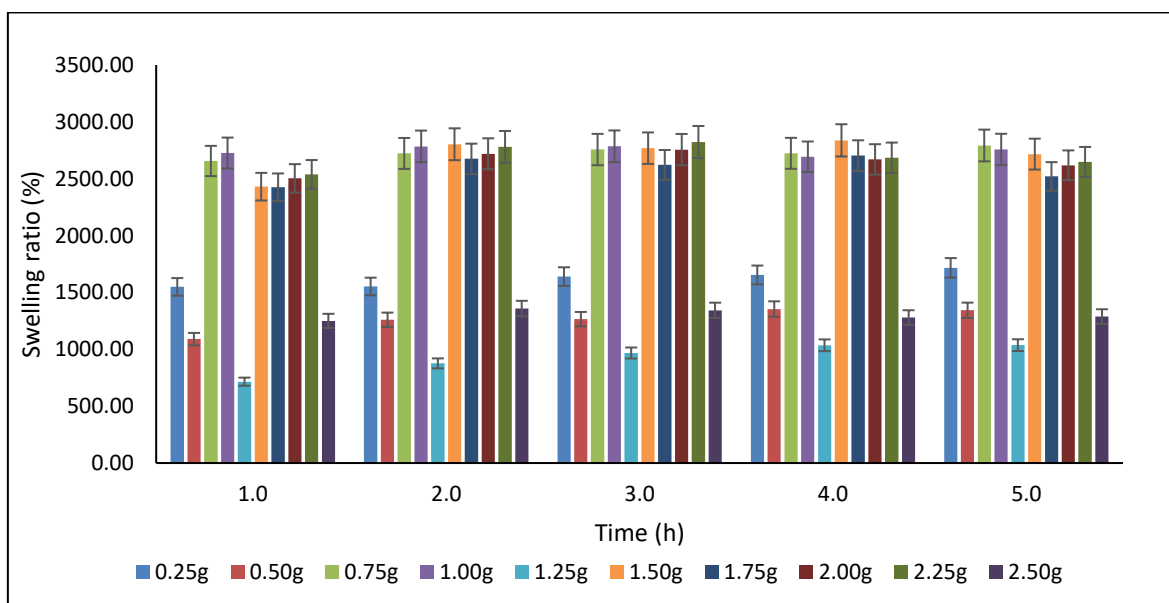


**Figure 4.20:** The swelling ratio of the cellulose-based hydrogel with PEG1000 (0.25 g - 2.50 g). The error bar represents the standard deviations from the triplicate (n=3)

In conclusion, the cellulose hydrogel with glycerol has the highest water uptake capacity, 4192.17%, among all the gelling agents because of the hydrogel's elasticity caused by the glycerol. The hydrogel with 2.0g of AA has the highest swelling ability compared to the hydrogel with other concentrations of AA. However, it was the hydrogel with the weakest

swelling ability among all the gelling agents. Herein, glycerol is the recommended gelling agent for forming the cellulose hydrogel with excellent water uptake capacity.

#### **4.6 Water retention of different types of cellulose-based hydrogels**

For the hydrogels that formed using AA as a gelling agent, the water retention of the hydrogels with 2.0 g of AA in Figure 4.21 has a better water retention than other AA concentrations. The initial water retention of all the hydrogels with AA is 100 %. The water retention of the hydrogel containing 2.0g of AA remained high, with 76.06%, after being placed in the open air for 24 hours. However, the water retention for other hydrogels dropped below 60 % after 24 hours. Then, the water retention of the cellulose-2g AA-based hydrogel remained at 9.24 %, while the water retention for other hydrogels was all less than 1%. This indicates that 2.0 g of AA is ideal to produce a hydrogel with high water retention in all concentrations. This is all affected by the porosity of the hydrogel (Jampi et al., 2021). Due to the high swelling ratio and water retention of cellulose-2.0g AA-based hydrogel, the Brunauer–Emmett–Teller (BET) analysis was carried out to determine further reasons behind the high water uptake and retention among all the concentrations of AA. The hydrogels containing 2.0 g of AA have many small pores (Figure 4.7) with a volume of  $4.900\text{ cm}^3\text{g}^{-1}$  and  $26.40\pm 0.3\text{ nm}$ . In contrast, cellulose-2.5g AA-based hydrogel's pores only have a volume of  $3.196\text{ cm}^3\text{g}^{-1}$  and  $17.9\pm 0.3\text{ nm}$ . By connecting the result of the BET analysis with the water uptake and retention, the deeper volume of the pores in the hydrogel with 2.0g AA allows it to retain more water. Among all the concentrations, the hydrogel containing 2.5 g of AA had the weakest water uptake and retention, and its pore size was smaller than that of the hydrogel containing 2.0 g of AA; however, its water retention was lower due to a shallower pore volume.

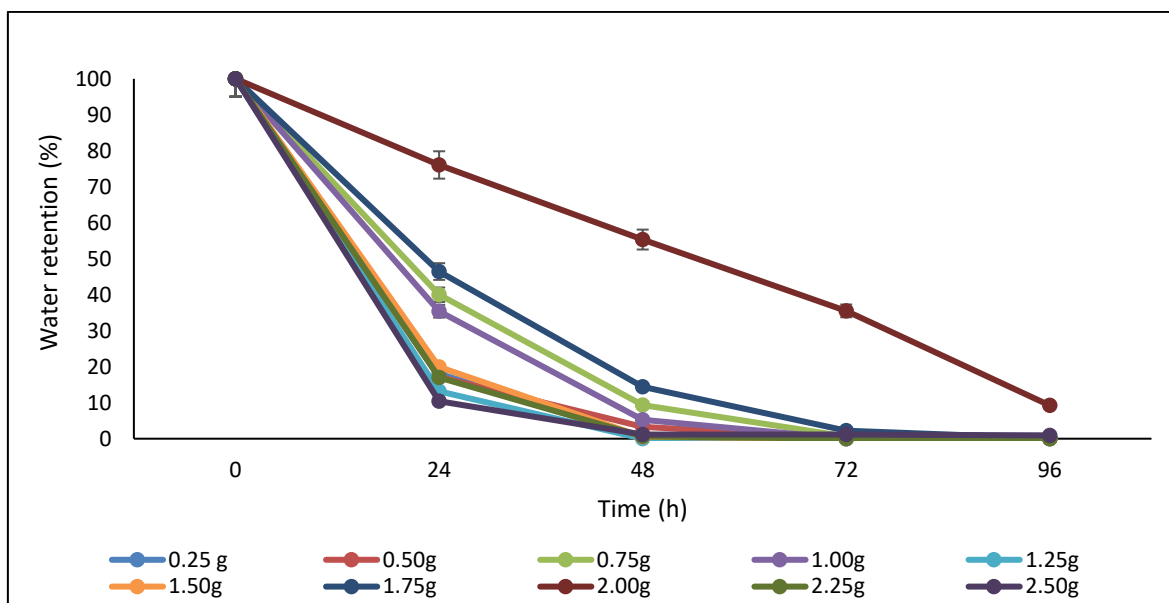


**Figure 4.21:** The water retention of the cellulose-based hydrogels plasticised with different amounts of AA. The error bar represents the standard deviations from the triplicate (n=3)

For the water retention of hydrogels with CMC, as shown in Figure 4.22, the hydrogels with 1.75 g of CMC had the highest water retention compared to other cellulose hydrogels with other concentrations of CMC. It still had 11.95 % of the water in its structure after 96 hours, but the rest of the hydrogels could not retain the water over 72 hours. The reason that the hydrogel at this concentration has high water retention is the presence of Donnan osmotic pressure causes a high amount of  $Na^+$  ions were present throughout the hydrogel's polymeric structure, and carboxylic groups increased (Ahmad et al., 2023; Salleh et al., 2019). This resulted in the cellulose-CMC-based hydrogel of this concentration having more hydroxyl and carboxyl groups, which can form hydrogen bonds with the water molecules in its structure. Therefore, the water retention of this hydrogel is better. The BET analysis was used to determine the pore information, such as pore volume and pore size of the hydrogels, to determine the relationship between the retention and the volume of the pores. Through the BET analysis result, the size of the pore in the hydrogel with 1.75g of CMC, which has the highest water retention capacity, is  $53.5 \pm 0.3$  nm, but the hydrogel with

2.0 g of CMC, which has the lowest water uptake and retention, is only  $14.6 \pm 0.3$  nm. This proved the observations from the surface morphology (Figure 4.5) of the cellulose hydrogel with 1.75 g of CMC, which had large pores available on its structure. The leading cause of it has the highest water uptake and retention among the other concentrations of CMC, and 2.0g AA was the deepest volume of the pores, with  $8.168 \text{ cm}^3\text{g}^{-1}$ . Thus, the hydrogel with 1.75 g of CMC has better swelling and water retention ability among all the concentrations of CMC and AA due to its large pore volume, which allows more water to be retained in the hydrogel.

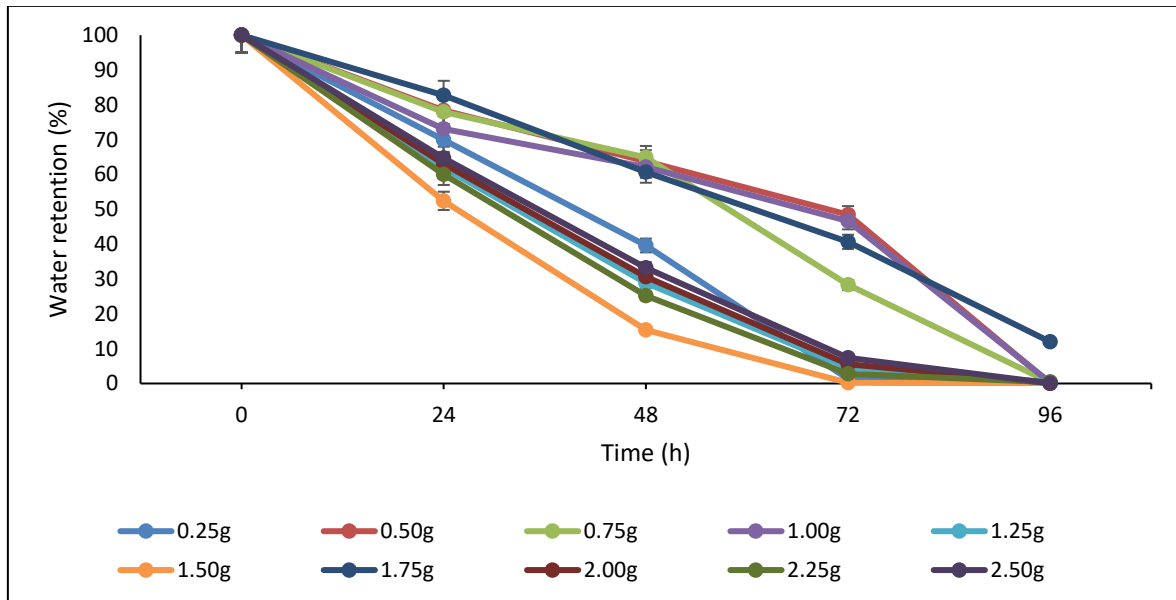


**Figure 4.22:** The water retention of cellulose-based hydrogels plasticised with different amounts of CMC. The error bar represents the standard deviations from the triplicate (n=3)

Moreover, the cellulose-glycerol-based hydrogels at these two concentrations (1.00 g and 2.00 g) retained water for longer because their water retention at 96 hours remains above 10 % and 8 %, respectively, as shown in Figure 4.23. Due to the surface morphology of both hydrogels being highly porous, the BET analysis was conducted to determine the factor that caused the hydrogel with 1.0 g of glycerol to have better water uptake and retention than the one with 2.0 g of glycerol. From the BET result, the volume of the pores

in the hydrogel with 1.0g of glycerol is  $6.535 \text{ cm}^3\text{g}^{-1}$ , but the volume of the pores of the hydrogel with 2.0 g of glycerol is only  $4.315 \text{ cm}^3\text{g}^{-1}$ , although its average pore size is  $31.3 \pm 0.3 \text{ nm}$ , which is smaller compared to  $37.70 \pm 0.3 \text{ nm}$  of the cellulose-1.0g glycerol-based hydrogel' pore size. The data found by BET analysis can further prove that a larger amount of water can be retained in the hydrogel with 1.0g of glycerol than in the other hydrogel, which has AA ( $4.900 \text{ cm}^3\text{g}^{-1}$ ) and CMC ( $8.168 \text{ cm}^3\text{g}^{-1}$ ), for a longer time, due to its larger volume of pores. Besides, the glycerol as the gelling agent in the formation of the hydrogel can cause the hydrogel matrix to become less dense, thus promoting the movement of the cellulose chains, resulting in the flexibility and malleability of the hydrogel (Basiak et al., 2018). The water retention of hydrogels with 0.75 g, 1.50 g, 2.25 g and 2.50 g of glycerol retained 4.30 % to 1.50 % of the water in their structure after 96 hours. The hydrogels with 0.5 g and 1.75 g of glycerol showed none of the water retained in their structure, due to their surface morphologies, which have fewer pores in their structure, could not retain the water after 72 hours and started to dry after a day.

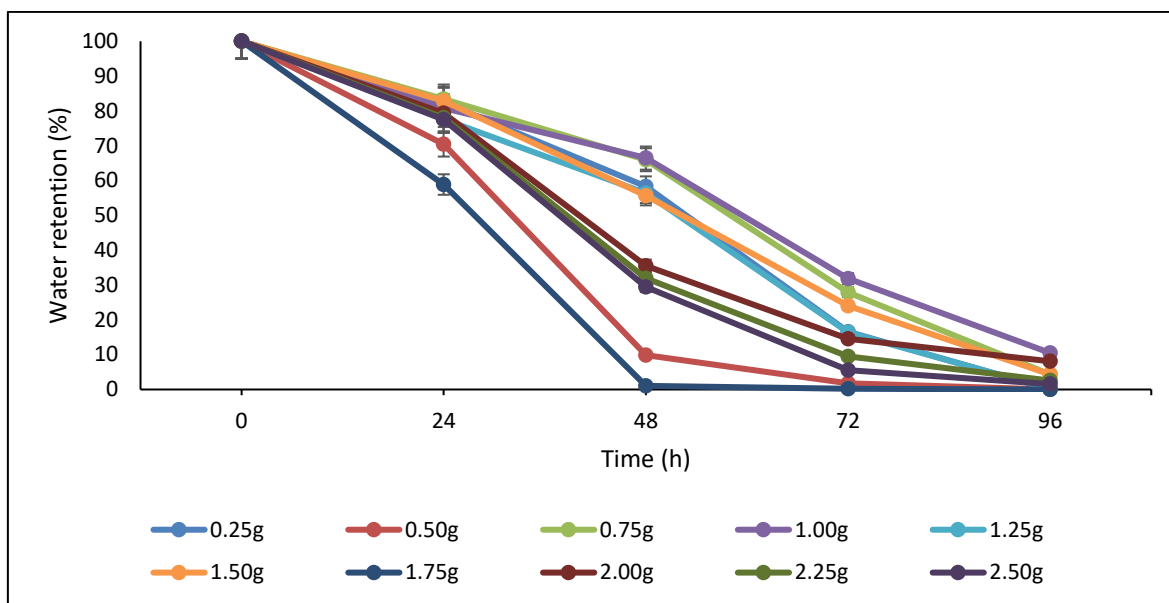


**Figure 4.23:** The water retention of cellulose-based hydrogel plasticised with different amounts of glycerol. The error bar represents the standard deviations from the triplicate (n=3)

From the results in Figures 4.24, 4.25, and 4.26, the hydrogel with a lower concentration of PEG400 and PEG600 has shown higher water retention after 96 hours. The hydrogels with 0.5 g and 0.75 g of PEG 400 have left 34.31% and 32.80% of water in their structure. In contrast, the hydrogel with 0.25 g to 1.25 g of PEG600 retained 22.61%, 33.47%, 33.34%, 31.18%, and 33.16% of the water, respectively. The hydrogel with 0.25 g, 0.5 g, 1.25 g and 2.50 g of PEG1000 cannot retain much water (only 0.24% to 0.04% of the water) in the structure for a long period. Meanwhile, the hydrogels with other concentrations of PEG1000 retained 26 % to 22 % of the water in those hydrogels after 96 hours. From this statement, the higher molecular weight of PEG is not compulsory for high water retention. In conclusion, the hydrogel with 0.5 wt% of PEG 600 has excellent water retention ability, with 33.47 % remaining after 96 hours, which is more suitable for agricultural applications. Besides, its surface morphology in Figure 4.7 formed the high porosity corresponding with the BET result on its total pore surface area is  $104.575 \text{ m}^2\text{g}^{-1}$  with a small pore size

( $11.92 \pm 0.3 \text{ nm}$ ) and volume of the pores is  $6.232 \text{ cm}^3 \text{ g}^{-1}$ . From here, this concentration is the optimum amount of PEG600 for producing a hydrogel with high porosity.

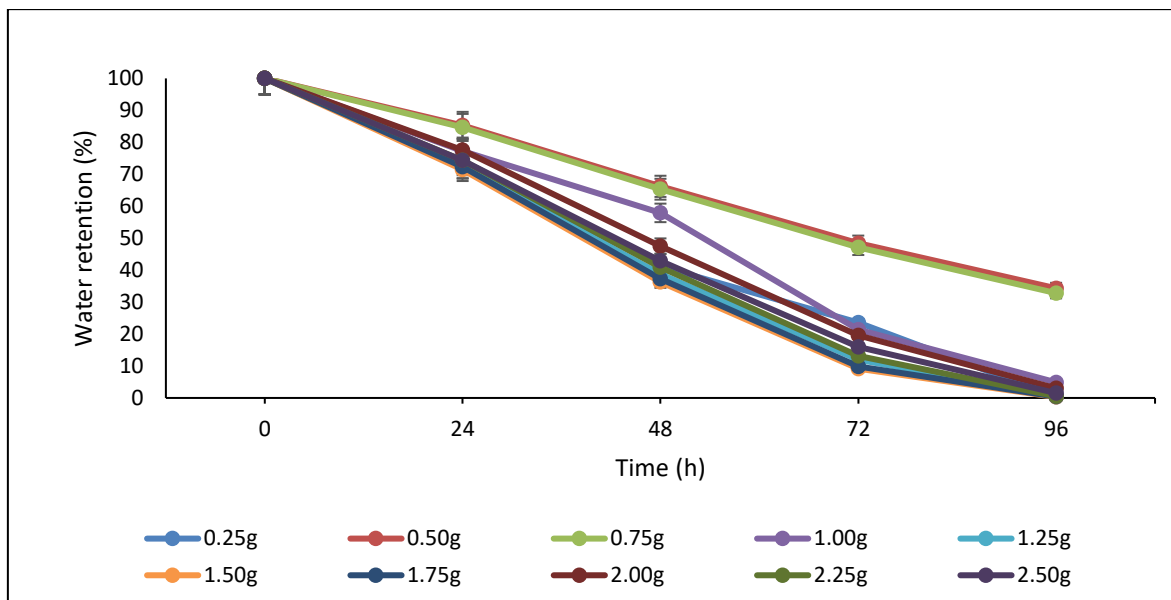


**Figure 4.24:** The water retention of the cellulose-based hydrogel plasticised with different amounts of PEG 400. The error bar represents the standard deviations from the triplicate (n=3)

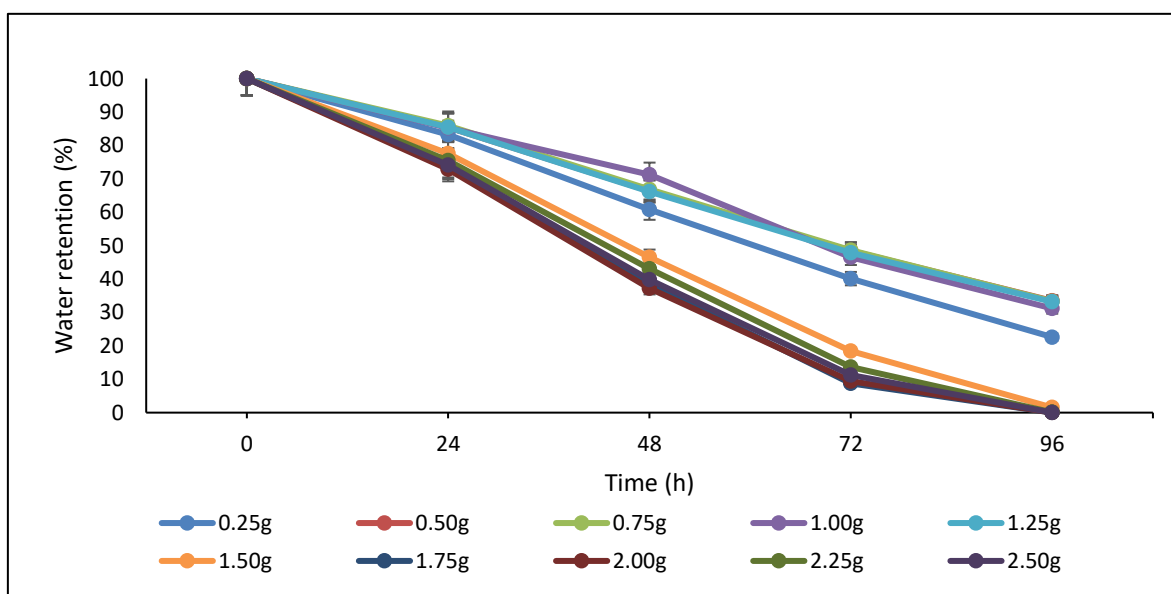


**Figure 4.25:** The water retention of cellulose-based hydrogel plasticised with different amounts of PEG 600. The error bar represents the standard deviations from the triplicate (n=3)

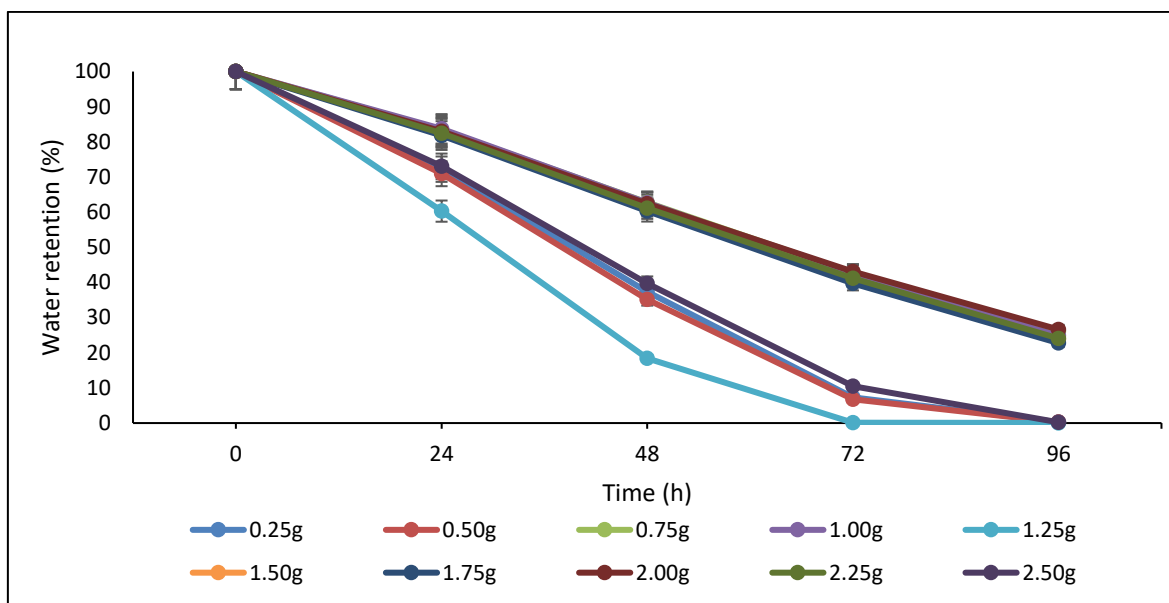


**Figure 4.26:** The water retention of the cellulose-based hydrogel plasticised with different amounts of PEG 1000. The error bar represents the standard deviations from the triplicate (n=3)

By comparing the water retention rate of the hydrogel with AA, CMC, glycerol, and PEG in Figure 4.27, the hydrogel with PEG600 has the highest and best water retention rate. However, the hydrogel with AA has the lowest and weakest water retention, with only 9.24%. From here, it can be known that PEG600 acts as a gelling agent and can decrease the intermolecular force and improve the mobility of the polymer chains, also because of the hydroxyl groups present in the PEG chains, causing high moisture content available (Yacob et al., 2021). Pore surface area of the cellulose-PEG-based hydrogel has the highest surface area with  $104.575 \text{ m}^2\text{g}^{-1}$ , indicating numerous pores are available in the hydrogel with PEG600. The size of the pore is only  $11.92 \pm 0.3 \text{ nm}$ , the smallest among all the gelling agents. This feature can prevent the water molecules in the hydrogel's structure from escaping. However, the volume of the pores is only  $6.232 \text{ cm}^3\text{g}^{-1}$ , which causes the swelling ratio to be only 2697.49%. In contrast, the hydrogels with 1.0g of glycerol have the highest water uptake capacity (3562.97%) among all the gelling agents due to the largest volume of the pores ( $6.535 \text{ cm}^3\text{g}^{-1}$ ). The hydrogel with AA has shown the lowest and weakest water

retention ability compared to the other three hydrogels because its pore volume is only  $4.900 \text{ cm}^3\text{g}^{-1}$ , resulting in its weak water uptake and retention capacity due to a lesser volume for retaining the water in this hydrogel.

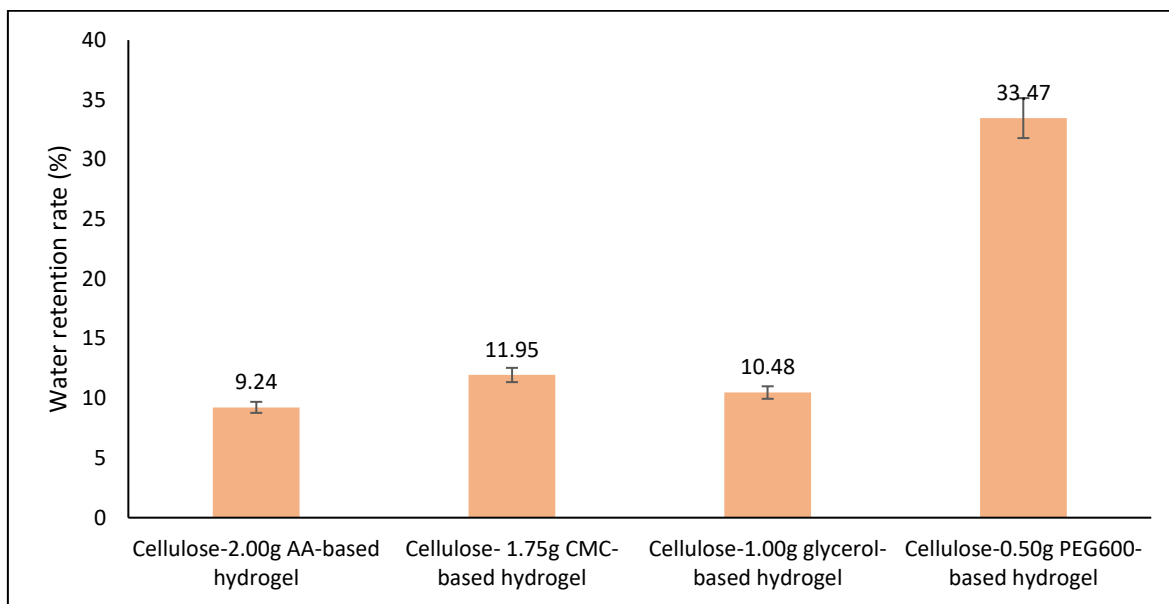


**Figure 4. 27:** The comparison of the maximum water retention of each cellulose-based hydrogel. The error bar represents the standard deviations from the triplicate (n=3)

In conclusion, the cellulose hydrogel with 0.5g of PEG600 had the best water retention among all the gelling agents due to the characteristics of the PEG600 and the size of the pores.

#### 4.7 Application of cellulose-based hydrogel as a soil conditioner

Based on the characterisation results (Table 4.2) of the cellulose-based hydrogels with various gelling agents, the cellulose hydrogels plasticised with 1.75 g of CMC, 2.0 g of AA, 1.0 g of glycerol, and 0.5 g of PEG600 showed excellent water uptake and retention. So, these hydrogels were used in the application part to determine the effect of different types of cellulose-based hydrogels on soil moisture, soil pH, soil electrical conductivity, seed germination of Choy Sum vegetable seeds, and the biodegradability of different types of cellulose-based hydrogels.

**Table 4.1:** The swelling ratio and water retention of the selected cellulose-based hydrogels

| <b>Types of cellulose-based hydrogel</b> | <b>Swelling ratio (%)</b> | <b>Water retention (%)</b> |
|------------------------------------------|---------------------------|----------------------------|
| Cellulose-1.75g CMC-based hydrogel       | 2603.24                   | 11.95                      |
| Cellulose-2.0g AA-based hydrogel         | 808.30                    | 9.24                       |
| Cellulose-1.0g glycerol-based hydrogel   | 3562.97                   | 10.48                      |
| Cellulose-0.5g PEG600-based hydrogel     | 2697.49                   | 33.47                      |

#### **4.7.1 Soil moisture of various types of soils**

The effect of each selected cellulose-based hydrogel on soil moisture in different soil types, such as sandy soil, topsoil, gley soil, and clayey soil, was determined in this study. All the moistures of the soils were measured using a soil moisture meter before the experiment started because the soil used in the experiment must not contain any moisture, which will affect the result of the experiment. This experiment was conducted for 15 days because the results started to be constant after 15 days, and the results were collected within these 15 days (the specimens were collected every 3 days). The soil moisture of all the soil types without hydrogel, as shown in Figures 4.28 to 4.31, also known as the control set, did not increase by remaining at 0 % for 15 days. This means no external factors, such as the humidity of the surroundings, affect the soil's moisture level.

Sand is a type of soil rarely used in agriculture and has brought enormous challenges due to its low water-holding capacity (Krishnan & Alsharaeh, 2023). However, this study showed a good sign of improving the moisture of the sand by using several types of cellulose-

based hydrogel in Figure 4.28. By comparing the performance of the hydrogels that contained PEG and CMC on the soil moisture of sandy soil, both gelling agents plasticised with the cellulose chains had improved the water bounding capacity of the hydrogel due to the hydrophilic functional groups which are carboxyl groups, hydroxyl groups and ether groups that available in the structure of those two gelling agents (Akalin & Pulat, 2018; Gao et al., 2021; Salleh et al., 2019). The cellulose-PEG-based hydrogel had better water retention (33.47%) because of the smallest size of pores (11.95nm), and improved the soil moisture of the sand by helping the sand retain the water. Besides, the hydroxyl groups available in PEG600 polymer chains can bind with more water via hydrogen bonding and slow down the evaporating rate of the water from the sand (Gao et al., 2021). Meanwhile, the moisture in the sand containing cellulose-glycerol-based hydrogel increased gradually until day 7 and slightly by 1.2 % on day 15. The moisture of the sand with AA-containing hydrogel dropped and increased dramatically from day 1 to day 7, dropped again by 2.6 % and remained at 15.6 % until the end of the observation period. From the statement here, it could be that AA, as the gelling agent in forming the hydrogel with the cellulose fibres through the chemical crosslinking process, cannot help the sand store much water like other hydrogels in this study. This may be because the water retained in the cellulose-2.0g AA-based hydrogel is not able to be retained for longer times because its pore volume is only  $4.900 \text{ cm}^3\text{g}^{-1}$ , and also, not many hydrophilic groups are formed between 2.0g of AA and cellulose chains, failing to maintain moisture in the sand. Therefore, this gelling agent may not be as effective in improving soil moisture as the other gelling agents used in this study. However, due to its viscoelasticity and mechanical toughness, it performs better in biomedical fields like drug delivery, antibacterial and wound healing (Rana et al., 2024).

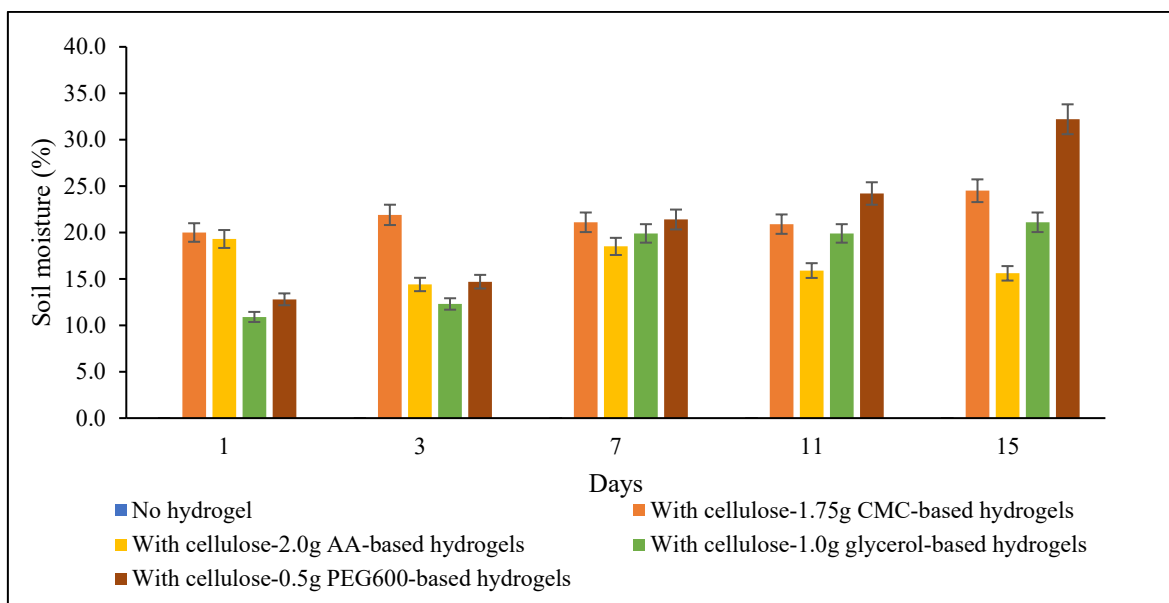


**Figure 4.28:** The soil moisture of the sand with types of cellulose-based hydrogels over a 15-day period. The error bar represents the standard deviations from the triplicate (n=3)

Apart from sandy soil, topsoil is grouped as red–yellow podzolic soil according to the Sarawak Soil Classification System (Ahmad et al., 2023). It is a common soil type found at the upper layer of the soil in the A horizon and the O horizon. When types of cellulose hydrogels were applied in the topsoil for 15 days in this study (Figure 4.29), all the cellulose-based hydrogels shown the improvement on the soil moisture of the topsoil but not as good as the sandy soil due to the topsoil is a shallow soil which had many empty spaces and the water is easier evaporate from the topsoil (López-Vicente & Álvarez, 2018). The highest soil moisture of the topsoil with cellulose-1.0g glycerol-based hydrogel on day 7 was 29.2 %, with the highest swelling ratio of 3562.97% among all the gelling agents. However, its soil moisture had dropped to 21.3 % on day 15, although it was still the highest soil moisture compared with the soil moisture of other hydrogels. The reason that caused its soil moisture to drop by 7.9 % from day 7 to day 15 is that the water retained by the hydrogel in the topsoil had evaporated due to there being many empty spaces in the topsoil, causing the water is not able to retain for a longer time (López-Vicente & Álvarez, 2018). As a result, the water

retained in the topsoil eventually evaporated. This situation also happened in the topsoil with cellulose-0.5g PEG600-based hydrogel, but it did not increase much in the soil moisture of the topsoil because its swelling ratio (2697.49%) is not as high as the hydrogel with 1.0g of glycerol, although its water retention (33.47%) is high in this study. The soil moisture of the topsoil with this type of hydrogel had increased from 10.5 % on day 3 to 19.9 % on day 7, but dropped to 16.6 % on day 15. This could be attributed to the evaporation of water released by the hydrogel. Nevertheless, the hydrogel still enabled the topsoil to retain 16.6% of moisture on day 15. While the cellulose-CMC-based hydrogel had maintained the soil moisture of the topsoil consistently for over 15 days in the range of 12 % to 16 %, it did not have a significant increase or decrease due to the properties of the topsoil.

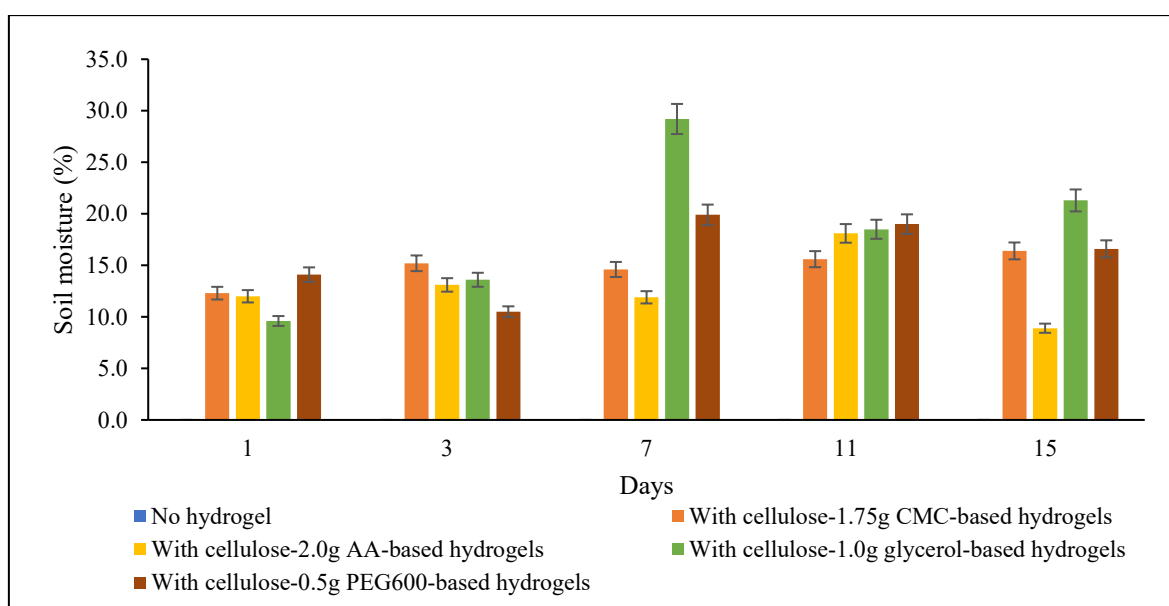


**Figure 4.29:** The soil moisture of the topsoil with types of cellulose-based hydrogels over a 15-day period. The error bar represents the standard deviations from the triplicate (n=3)

For the effect of the types of cellulose-based hydrogels on the soil moisture of the gley soil in Figure 4.30, the gley soil with the cellulose-AA-based hydrogel had the lowest soil moisture during day 15, with 10.7 %, but the soil moisture was able to reach 18.1 % on day 7. The water from this hydrogel had increased the moisture of the gley soil but could not

be sustained until day 15 because the water uptake capacity of cellulose-2.0g AA-based hydrogel is 808.30%, which was not enough to keep the gley soil moist for more than a week due to the gley soil is containing a high level of the clay particles and the organic matters that required much more water to keep it in moist (Aluoch et al., 2022). Furthermore, the soil moisture of the gley soil that contained the cellulose-0.5g PEG600-based hydrogel was the highest at 41.3 % compared to other hydrogels due to its excellent water retention (33.47%) that can slowly provide water to the soil instead of providing the water faster in a short period, so the clay particles can retain the water that provided by the hydrogel without leaching out from the soil. Besides, the soil particle size in the gley soil is more packed and minor, causing the water released by the hydrogel to be retained in the soil even in dry weather (Nassaj-Bokharaei et al., 2021). Additionally, the cellulose-1.0g glycerol-based hydrogel and cellulose-1.75g CMC-based hydrogel performed similarly on the soil moisture of the gley soil, with both hydrogels still having 22.8 % and 21.1 % of water remaining in the soil during day 15. This outcome is primarily attributed to the comparable water retention capacities of the two cellulose-based hydrogels, with no statistically significant difference observed (9.24% vs. 10.48%), leading to only a minor variation in soil moisture levels.

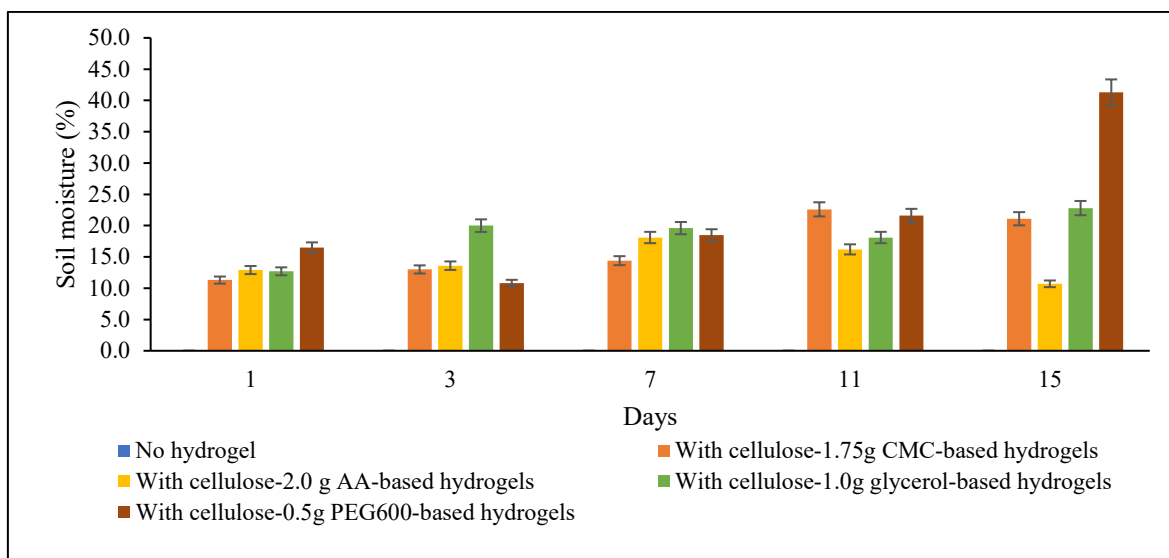


**Figure 4.30:** The soil moisture of the gley soil with types of cellulose-based hydrogels over a 15-day period. The error bar represents the standard deviations from the triplicate (n=3)

The effect of different types of cellulose-based hydrogel on clayey soil moisture was also determined. Through the result of the soil moisture of the clayey soil with types of cellulose-based hydrogel in Figure 4.31, the cellulose-0.5g PEG600-based hydrogel had the highest water available in the clayed soil with 20.5 % at day 15, and the soil moisture constantly increased from day 1 to 7 and stayed at 20% and above. The soil moisture of the hydrogel with 1.75g of CMC, 1.0g of glycerol, and 2.0g of alginic acid at 15 days is 14 % to 19 %. Based on the result above, the soil moisture of clayey soil with cellulose-based hydrogels is lower than the soil moisture of gley soil with cellulose-based hydrogels because the clayey soil is full of clay particles, which require more water molecules to fill in large surface area of the porosity in the soil system, so more water was used to fill in the space and caused the water available in the hydrogels are absorbed by the clay particles, resulting in the hydrogel could not retain more water (Abdelghafar et al., 2024).

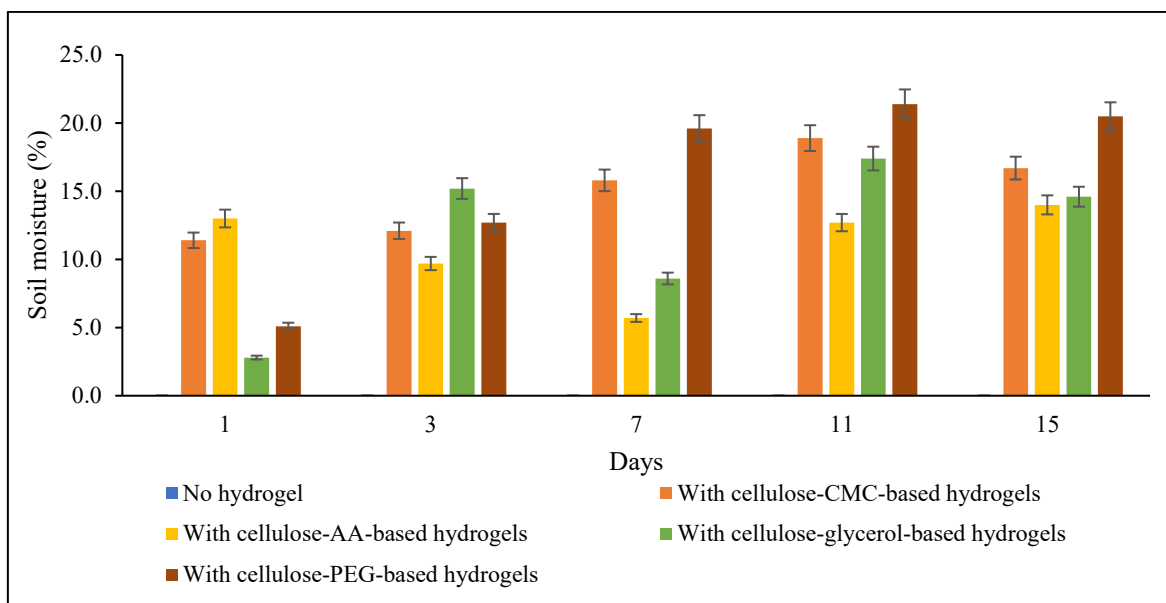


**Figure 4.31:** The soil moisture of the clayey soil with types of cellulose-based hydrogels over a 15-day period. The error bar represents the standard deviations from the triplicate (n=3)

In conclusion, the cellulose-based hydrogels with various gelling agents can increase soil moisture in sandy soil, topsoil, gley soil, and clayey soil. However, the cellulose-0.5 g PEG600-based hydrogel, the cellulose-1.0g glycerol-based hydrogel and the cellulose-1.75g CMC-based hydrogel had the potential to become soil conditioners to improve the soil moisture of the four soil types used in this study due to their water uptake capacity and water retention.

#### 4.7.2 The pH of various types of soil

The pH of the soil is also a factor that can influence the growth of the plants. When the soil pH is in the optimum range (pH 5 to pH 8), suitable for most plants, especially vegetables, to grow well in this range (Sang et al., 2011; Gentili et al., 2018). Besides, plants can efficiently uptake the nutrients needed when the soil pH is correct. So, this study aims to determine whether cellulose-based hydrogels plasticised with 1.75g CMC, 2.0g AA, 1.0g

glycerol, and 0.5g PEG600 can increase the soil pH and how the pH can be maintained within 15 days.

First, the sand without any hydrogel, known as the control set, had a constant soil pH of 7.2 over 15 days in Figure 4.32. After 250g of the types of cellulose-based hydrogels were mixed with 250g of sand in each planting bag, all the soil pH increased to pH 8 and above, and the soil pH was maintained constantly after day 3. It is believed that the increases in soil moisture directly influenced the pH value of the soil because soil moisture in this study improved after day 1, and the pH value started to be constant. This also had the same result as the study done by Tefera et al. (2022), which stated that the pH value of the sand is directly influenced by its moisture.

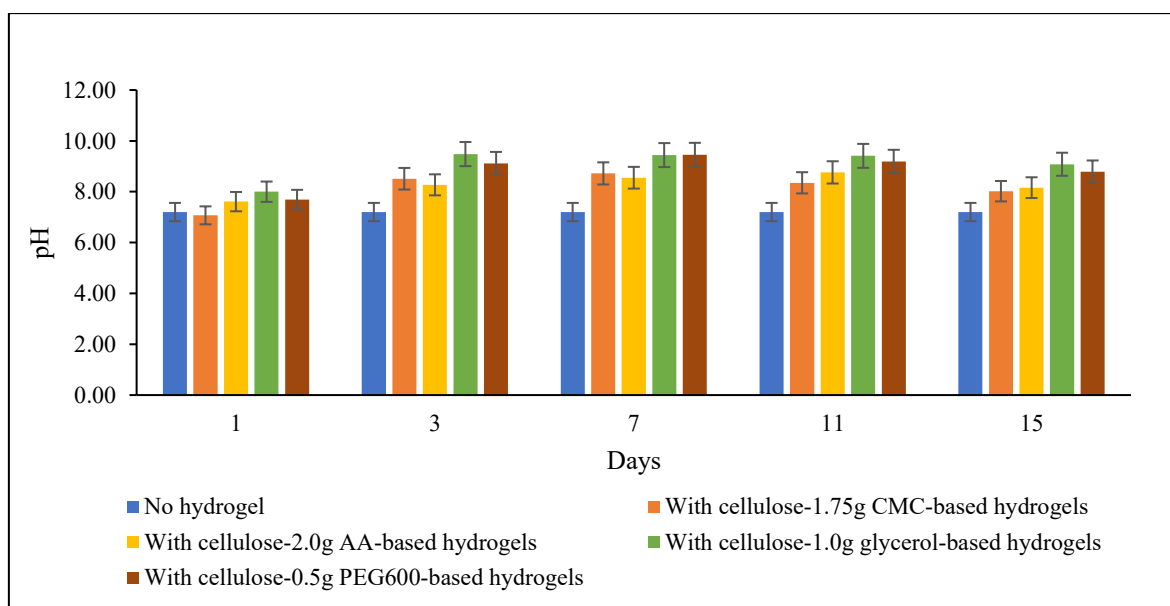


**Figure 4.32:** The pH of the sand with types of cellulose-based hydrogels. The error bar represents the standard deviations from the triplicate (n=3)

All the topsoil's pH values increased in all conditions, bringing the pH value from acidic pH to slightly alkaline or alkaline pH of the topsoil, except for the condition without any hydrogel, shown in Figure 4.33. The pH value of topsoil before the hydrogel was pH 5, and all the pH values in all conditions were increased to pH 6.53 (with cellulose-1.75g CMC-

based hydrogels), pH 5.63 (with cellulose-2.0g AA-based hydrogels), pH 7.77 (with cellulose-1.0g glycerol-based hydrogels), and pH 7.84 (with cellulose-0.5g PEG600-based hydrogels). The pH values of the topsoil in all conditions except for the condition without any hydrogel increased from acidic pH to alkaline pH in Figure 4.33.

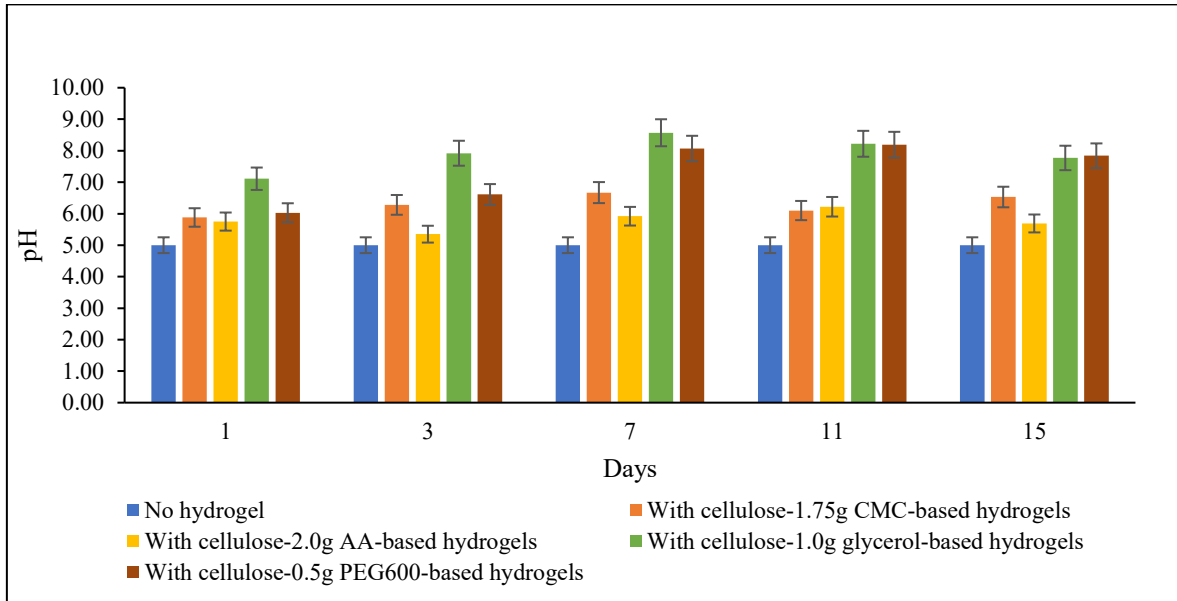


**Figure 4.33:** The pH of the topsoil with types of cellulose-based hydrogels. The error bar represents the standard deviations from the triplicate (n=3)

Moreover, the gley and clayed soil, initially with a pH of 4.58 and 5.12, respectively, had also increased to slightly alkaline in all the conditions with hydrogels, as shown in Figures 4.34 and 4.35. By referring to the soil pH of the gley soil in Figure 4.34, the soil pH with the cellulose-1.0g glycerol-based hydrogel in the gley soil kept increasing until day 15. This is due to the mechanism of diffusion-driven developed between the exterior and interior of the cellulose-1.0g glycerol-based hydrogel, which caused the water to be released at a slow rate, so the soil moisture increased slowly, and the pH value also increased slowly (Kabir et al., 2018; Albalasmeh et al., 2022). Besides, the gley soil contains a high amount of clay particles, but not as high as the clay particles in clayey soil and organic matter, causing it to retain more water inside, which results in the pH value growing over 15 days.

The study done by Tefera et al. (2022) also proved that hydrogel can increase the soil pH effectively.

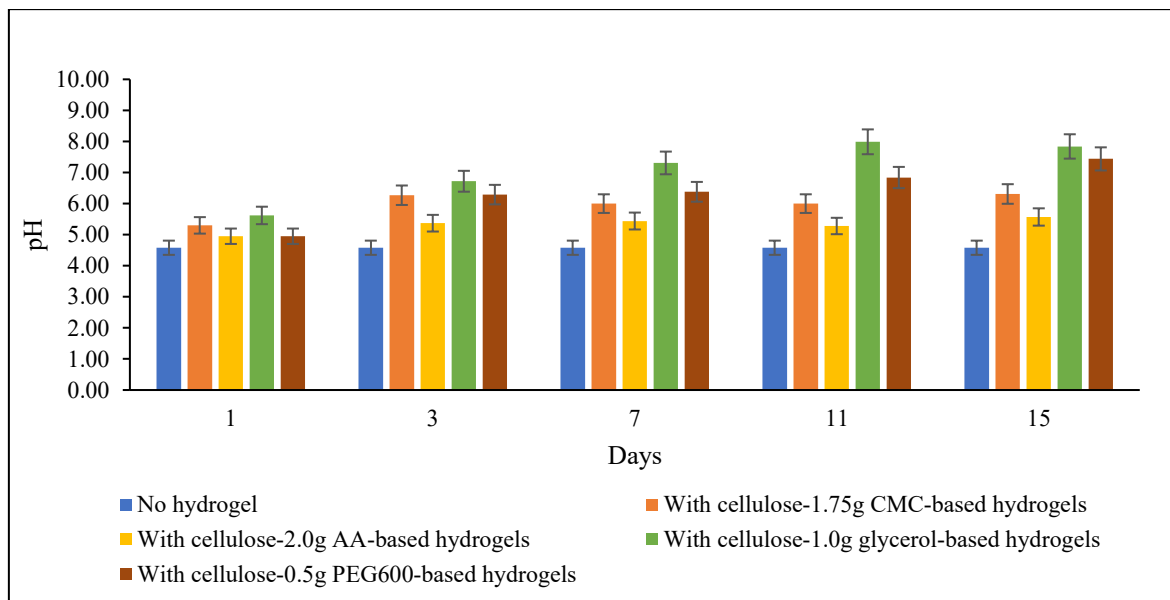


**Figure 4.34:** The pH of the gley soil with types of cellulose-based hydrogels. The error bar represents the standard deviations from the triplicate (n=3)

In addition, the clayey soil pH with the cellulose-1.0g glycerol-based hydrogel and the cellulose-0.5g PEG600-based hydrogel increased to pH 6.94 and pH 7.14 on day 15, respectively (Figure 4.35), compared to the clayey soil without hydrogel. This is because the high water retention of these two hydrogels, which were able to retain 10.48% and 33.47% of the water. Furthermore, the rising trend of pH value in clayey soil was increased due to the water retention effect of hydrogels, and it is believed that the water contained in the hydrogels is responsible for the increase in pH value in clayey soil.

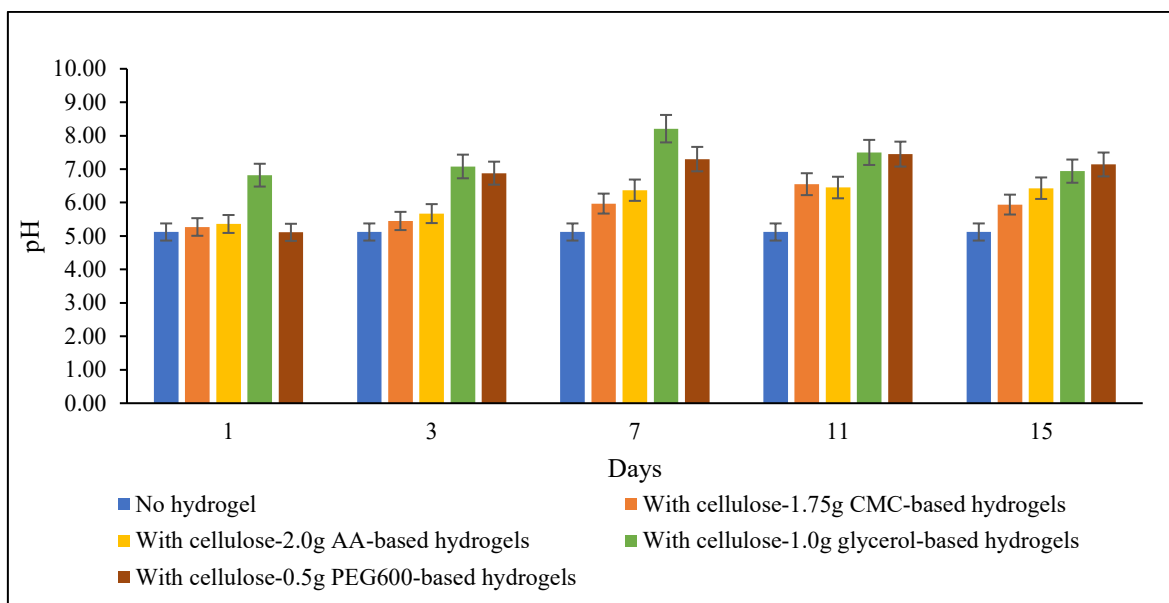


**Figure 4.35:** The pH of the clayey soil with types of cellulose-based hydrogels. The error bar represents the standard deviations from the triplicate (n=3)

In conclusion, the cellulose hydrogel with 1.75g CMC, 1.0g glycerol, and 0.5g PEG600 improved the soil pH of sand, topsoil, gley soil, and clayey soil at day 15. Thus, the hydrogels with these gelling agents can be applied to soil types with acidic pH, which is not suitable for plant growth, and increase the soil pH to neutral and slightly alkaline for planting most plants.

#### 4.7.3 The electrical conductivity (EC) of various types of soils

The soil electrical conductivity, also known as salinity, is an index to measure the number of salts (cations and anions) present in the soil that the plant requires, and it is related to soil properties that directly affect crop productivity (Piccoli et al., 2024). The high soil electrical conductivity at around 200 to 800  $\mu\text{S}/\text{cm}$  is available for the plants to thrive. The effect of the various cellulose-based hydrogels on the salinity of each soil type is important because this can determine whether those selected hydrogels can improve the salinity of sand, topsoil, gley soil, and clayey soil. Figures 4.36, 4.47, 4.38, and 4.39 show the electrical

conductivity of sand, topsoil, gley soil, and clayed soil with each type of cellulose-based hydrogel.

The soil electrical conductivities of the sand, topsoil, gley soil, and clayey soil in the control set are 63.9  $\mu\text{s/cm}$ , 44.2  $\mu\text{s/cm}$ , 55.0  $\mu\text{s/cm}$ , and 113.9  $\mu\text{s/cm}$ . All the salinity in the control sets did not change over 15 days. It can be confirmed that no external factors, such as temperature or humidity, affect the results of each soil type during the experimental period. For the effect of the types of hydrogels on the electrical conductivity of the sand, the cellulose-1.75g CMC-based hydrogels caused the electrical conductivity to increase constantly over 15 days. They had the highest salinity in the sand among all the cellulose-based hydrogels on day 15, which reached 803.3  $\mu\text{s/cm}$ . This may be due to the hydrogel with 1.75g CMC having the presence of the  $\text{Na}^+$  ions that roam freely in the hydrogel's structure. The cation's presence will increase the sand's electrical conductivity (Salleh et al., 2019; Jeevan et al., 2023). Besides sand, the cellulose-0.5g PEG600-based hydrogels had the highest electrical conductivity in the topsoil (689.7  $\mu\text{s/cm}$ ), gley soil (446.7  $\mu\text{s/cm}$ ), and clayed soil (386.7  $\mu\text{s/cm}$ ), followed by the cellulose-1.0g glycerol-based hydrogels, with a second higher salinity in topsoil (572.0  $\mu\text{s/cm}$ ), gley soil (338.0  $\mu\text{s/cm}$ ), and clayed soil (336.3  $\mu\text{s/cm}$ ). The hydrogel with 0.5g PEG600 and 1.0g glycerol can improve the electrical conductivity of the gley soil and clayey soil because the presence of these hydrogels can reduce the leaching loss of the basic cations that are available in the soil and the amount of the hydrogel let the water is not allowed to drain adequately due to these hydrogels providing the water to the soil slowly. Besides, based on the result of the soil salinity of the sand with cellulose-1.0g glycerol-hydrogel on day 1 is unexpectedly high and can be explained the sand in day 0 is in the dry condition but after applying the hydrogel with 1.0 glycerol the sand suddenly in the moisture condition due to the water containing in the hydrogel with

1.0g glycerol is high. The studies done by Bai et al. (2010) found that the first drying and wetting cycle occurring in the soil is 1.7 times greater on the soil's electrical conductivity.

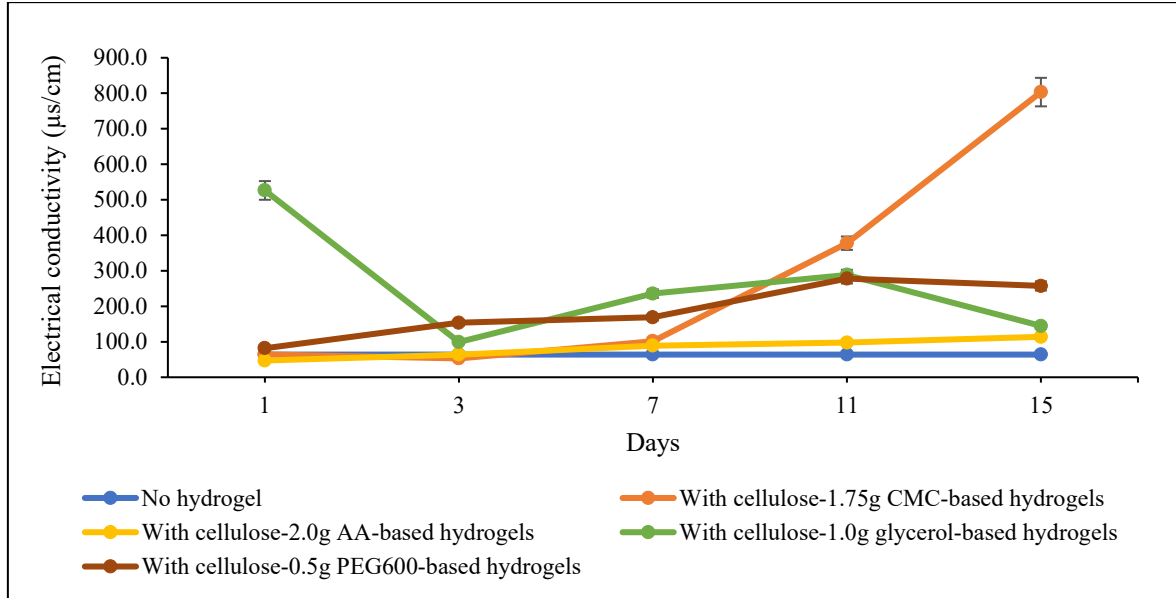


**Figure 4.36:** The electrical conductivity of sand with types of cellulose-based hydrogel. The error bar represents the standard deviations from the triplicate (n=3)

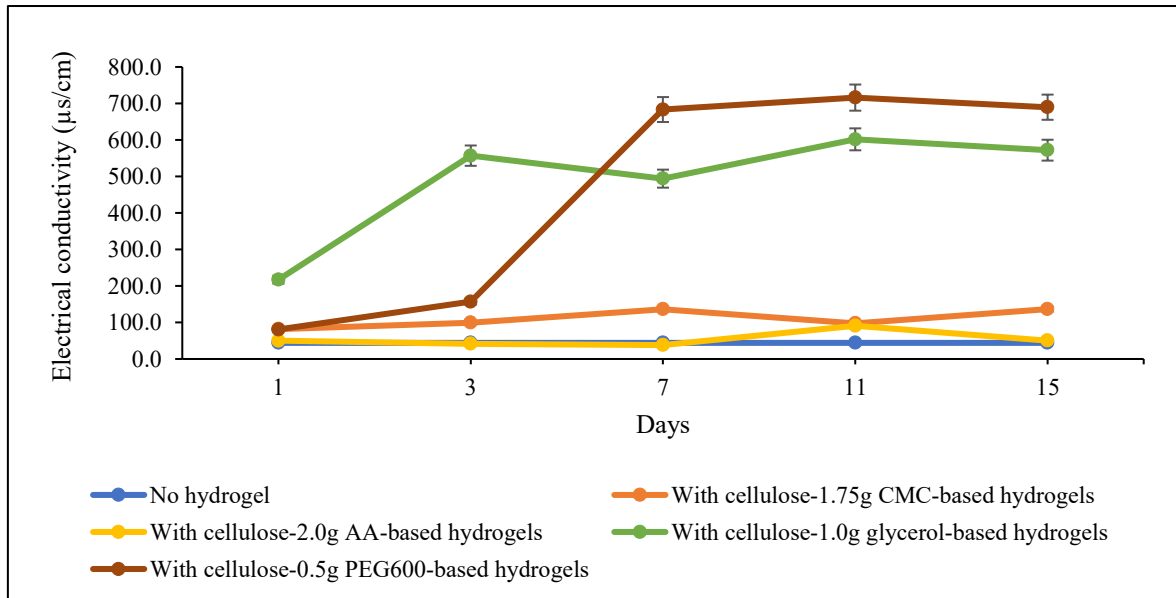


**Figure 4.37:** The electrical conductivity of topsoil with types of cellulose-based hydrogel. The error bar represents the standard deviations from the triplicate (n=3)

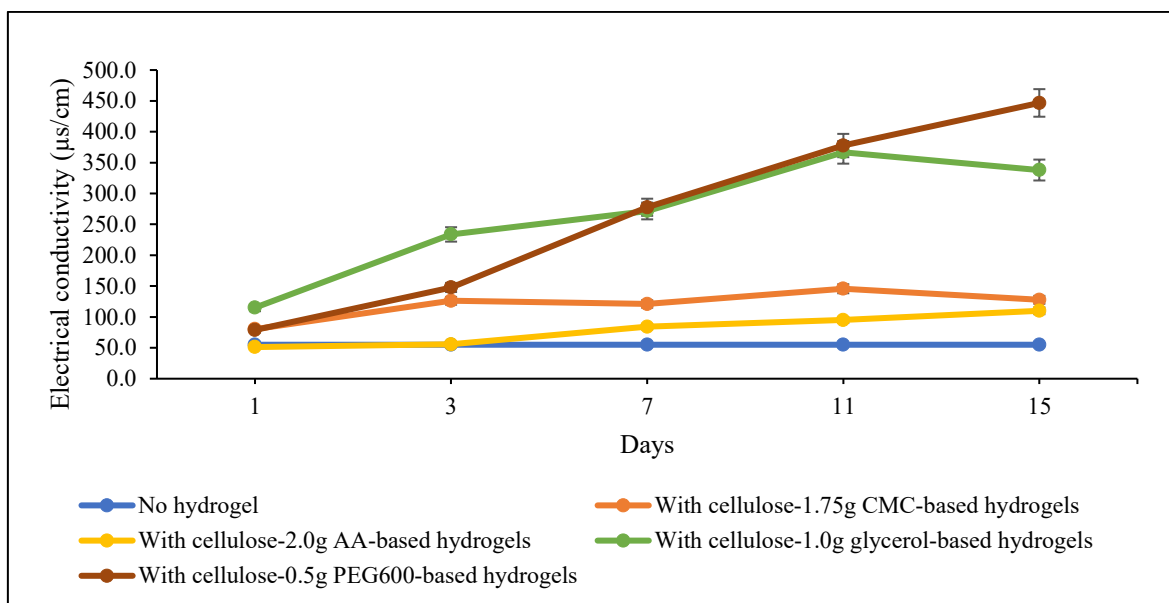


**Figure 4.38:** The electrical conductivity of gley soil with types of cellulose-based hydrogel. The error bar represents the standard deviations from the triplicate (n=3)

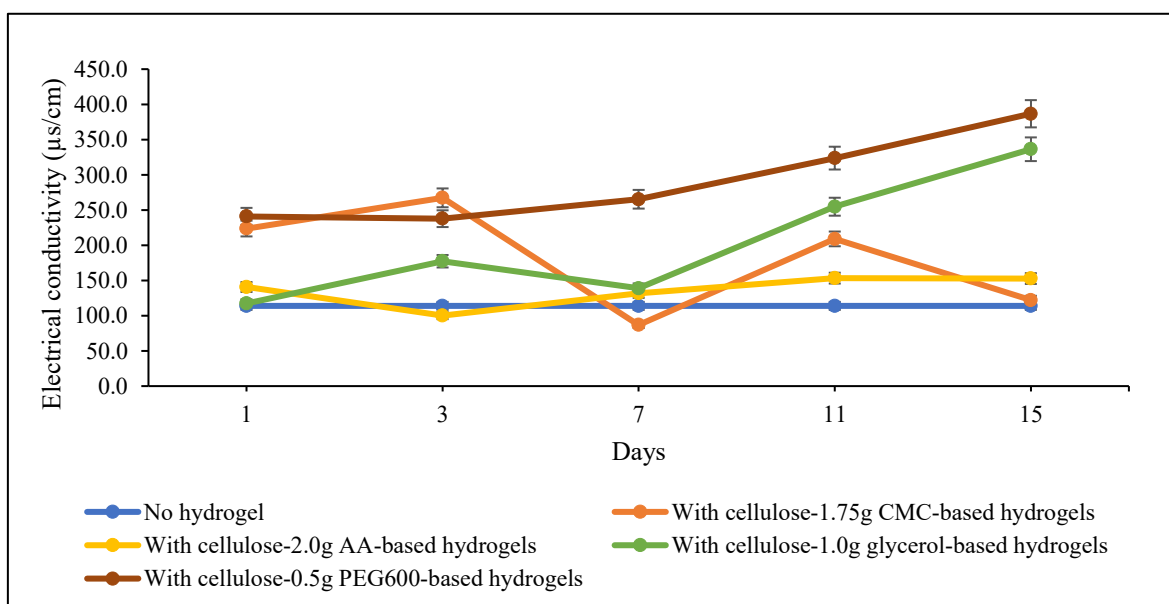


**Figure 4.39:** The electrical conductivity of clayey soil with types of cellulose-based hydrogel. The error bar represents the standard deviations from the triplicate (n=3)

In conclusion, the results of the salinity of each of the soil types indicate that the cellulose-based hydrogels with 1.75g CMC, 1.0g glycerol, and 0.5g PEG600 improved the electrical conductivity of the sandy soil, topsoil, gley soil, and clayed soil, which indicates that hydrogels can help all the soil types retain water without evaporating and increase the

soil salinity of each soil type. Thus, these hydrogels are an ideal soil conditioner to improve the soil properties such as soil moisture, pH, and electrical conductivity.

#### **4.8 Seed germination on hydrogels with various gelling agents**

Hydrogel is a new technology that can produce soilless conditions for germinating seeds. Our previous study proved that the cellulose-based hydrogel plasticised with carboxymethylcellulose (CMC) performed better than germinating paddy seeds in the soil. (Jong et al., 2024). To further study the effectiveness of cellulose-based hydrogel on germinating the common vegetable like Choy Sum vegetable, this study demonstrated and compared the effectiveness of types of cellulose-based hydrogel plasticised with alginic acid (AA), sodium carboxymethylcellulose (CMC), glycerol and polyethylene glycol (PEG) on the seed germination of Choy Sum vegetable. The results of the seed germination on types of cellulose-based hydrogels were compared with each other and also compared with the result of the seed germination without any hydrogel. Table 4.3 shows the effect of cellulose-based hydrogels containing various gelling agents on each germination parameter.

**Table 4.2:** The effect of cellulose-based hydrogels containing various gelling agents on germination parameters

| Germination Parameters                 |                 |                            |                               |                        |                      |                       |
|----------------------------------------|-----------------|----------------------------|-------------------------------|------------------------|----------------------|-----------------------|
| Treatment                              | Germination (%) | Mean Daily Germination (%) | Peak Value of Germination (%) | Germination Energy (%) | Energy Period (Days) | Germination Value (%) |
| No hydrogel                            | 27.00           | 3.57                       | 4.00                          | 25.00                  | 7                    | 2.25                  |
| Cellulose-2.0g AA-based hydrogel       | 40.00           | 4.29                       | 5.60                          | 30.00                  | 7                    | 4.58                  |
| Cellulose-1.75g CMC-based hydrogel     | 51.00           | 5.14                       | 5.67                          | 36.00                  | 7                    | 5.28                  |
| Cellulose-1.0g glycerol-based hydrogel | 65.00           | 8.71                       | 11.40                         | 61.00                  | 7                    | 16.91                 |
| Cellulose-0.5g PEG600-based hydrogel   | 49.00           | 6.29                       | 6.29                          | 44.00                  | 7                    | 6.89                  |

All cellulose hydrogels can last over a week without watering, as shown in Figure 4.40. The Choy Sum vegetable seeds at the cellulose hydrogels plasticised with 2.0g AA and 1.75g CMC were withered at day 14 (Figure 4.40). However, seeds germinating on cellulose-0.5g PEG600-based hydrogel began to wilt on day 14, while those germinating on cellulose-1.0g glycerol-based hydrogel grew healthy. This is because the water stored in the hydrogel is evaporated or absorbed by the seeds to complete the germination process, and the water swelling in its structure is greater. The water retention of the cellulose-1.0g glycerol-based hydrogel is the main factor that helps the seeds have enough water, as shown in the study

done by Qin et al. (2022), which states that the hydrogel with glycerol has hydrophilic properties and can provide the roots with additional moisture. Based on the observations in this study, cellulose-based hydrogels containing gelling agents can create ideal germination conditions for plants, maintaining sufficient moisture for over a week without additional watering.

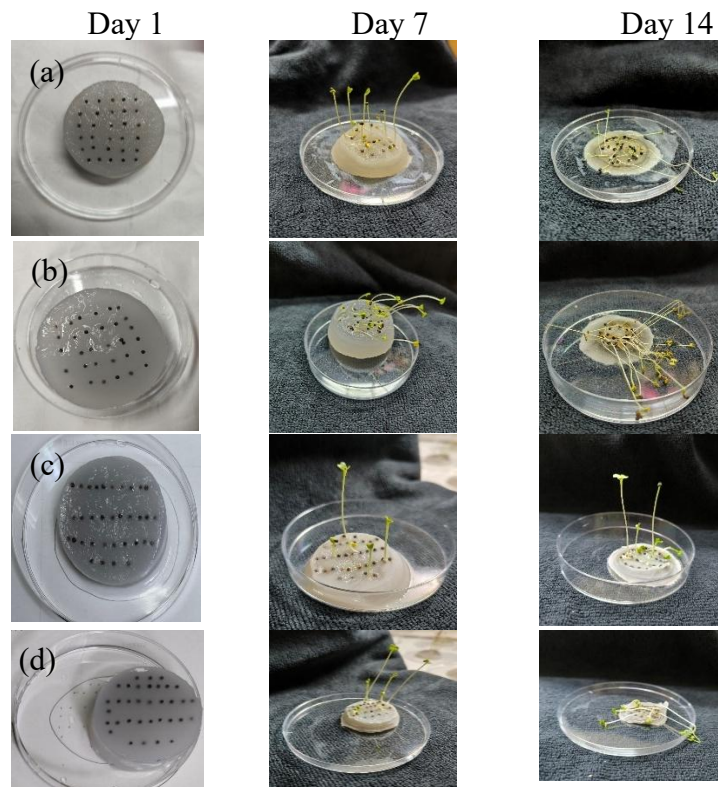


**Figure 4.40:** The observations of (a) the cellulose-AA-based hydrogel, (b) the cellulose-CMC-based hydrogel, (c) the cellulose-glycerol-based hydrogel, (d) the cellulose-PEG-based hydrogel, along the germination period without watering

The seed germination of the Choy Sum vegetable on the cotton pad (without hydrogel) with watering every day is only 27 %, as shown in Figure 4.41, and its germination value is only 2.25 %. During this study, the seeds also germinated without hydrogel and daily watering. However, no seed germination was observed due to insufficient moisture, as the water from the cotton pad had evaporated. Based on this situation, seeds germinating on cotton must be watered daily. Without regular watering, the seeds will fail to germinate

because the water evaporates, leaving insufficient moisture to support germination. Therefore, the traditional method of germinating seeds on cotton is inadequate unless a consistent and sufficient water supply is maintained. However, most of the seeds germinated on the types of swollen cellulose-based hydrogels, which do not provide any water during the germination period, were successfully germinated.

Based on the results in Figure 4.41, 65 % of the seeds germinated on the cellulose-1.0g glycerol-based hydrogel, followed by 51 % of the seeds germinated on the cellulose-1.75g CMC-based hydrogel, and 49 % of the seeds germinated on the cellulose-0.5g PEG600-based hydrogel. The cellulose-2.0g AA-based hydrogel had the lowest seed germination rate of Choy Sum vegetable among the selected cellulose hydrogels in this study at 40 %. When the overall seed germination rate of all types of the cellulose-based hydrogel without watering compared to the seed germination of Choy Sum vegetables in the condition of no hydrogel and watering every day, most of the Choy Sum vegetable seeds on those selected hydrogels in this study are successfully germinated with the seed germination rate at 40 % and above. The observed results can be attributed to the inherent water-retention capacity of the hydrogels, which maintained adequate moisture levels to support seed germination. Consequently, the lack of supplemental water during the germination period did not significantly affect the germination rate. Moreover, cellulose-based hydrogels effectively establish a soilless medium conducive to seed germination. Moreover, the cellulose-1.0g glycerol-based hydrogel had the highest seed germination rate at 61 % compared to the seed germination rate among all the selected cellulose hydrogels. The enhanced seed germination observed with this cellulose hydrogel is likely due to its superior water retention capacity, attributed to the abundance of hydrophilic groups within its structure. This characteristic, as demonstrated by Qin et al. (2022), enables the hydrogel to

continuously supply water to the seeds throughout the germination process.

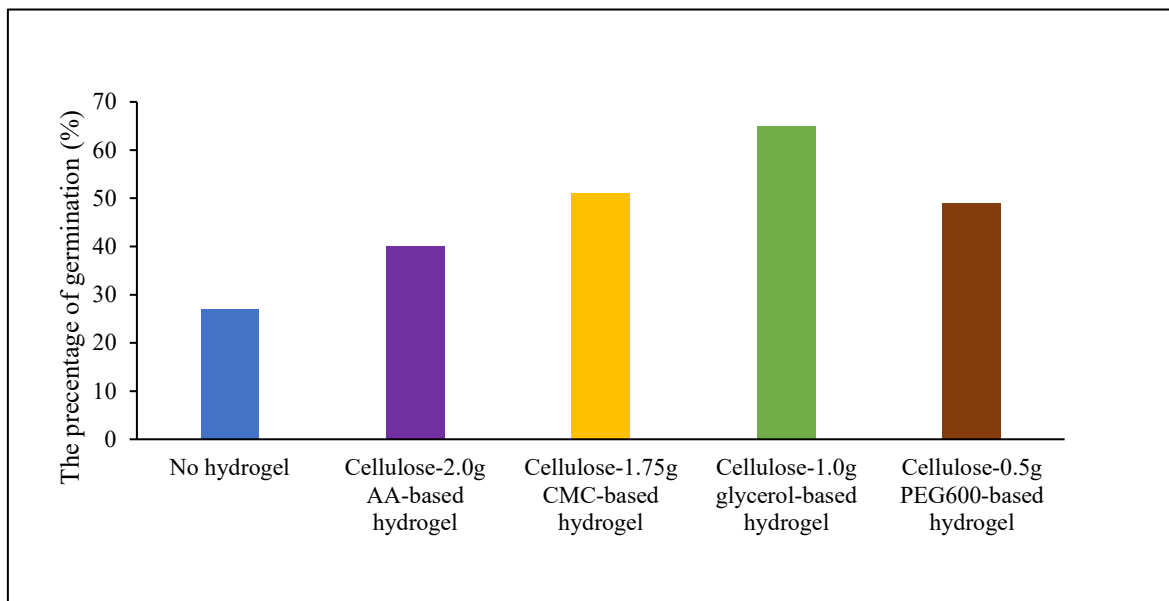


**Figure 4.41:** The germination of the Choy Sum vegetable seeds during the observation period

According to the seed germination value and the mean daily germination percentage of all the conditions in Figures 4.42 and 4.43, the hydrogel with 1.0g glycerol as the gelling agent had the most outstanding seed germination value of 16.91 % and had the most seeds starting to germinate on day 3. This proved that the seeds that germinated on cellulose-glycerol-based hydrogel are earlier and faster because the moisture provided by the hydrogel improves the metabolic activity of the seeds and causes the improvement in the germination process with earlier and better germination (Mote et al., 2018; Sahoo et al., 2022). The germination value of the rest of the cellulose hydrogels with 1.75g CMC, 0.5g PEG600, and 2.0g AA is between 4.5 % and 6.9 %, but it also promoted the earliest germination on day 3. Those hydrogels can improve the early seed germination process and perform better than the seeds that germinate on cotton without any hydrogel because the water uptake capacities of those hydrogels are the main factor that promotes the earlier seed germination process.

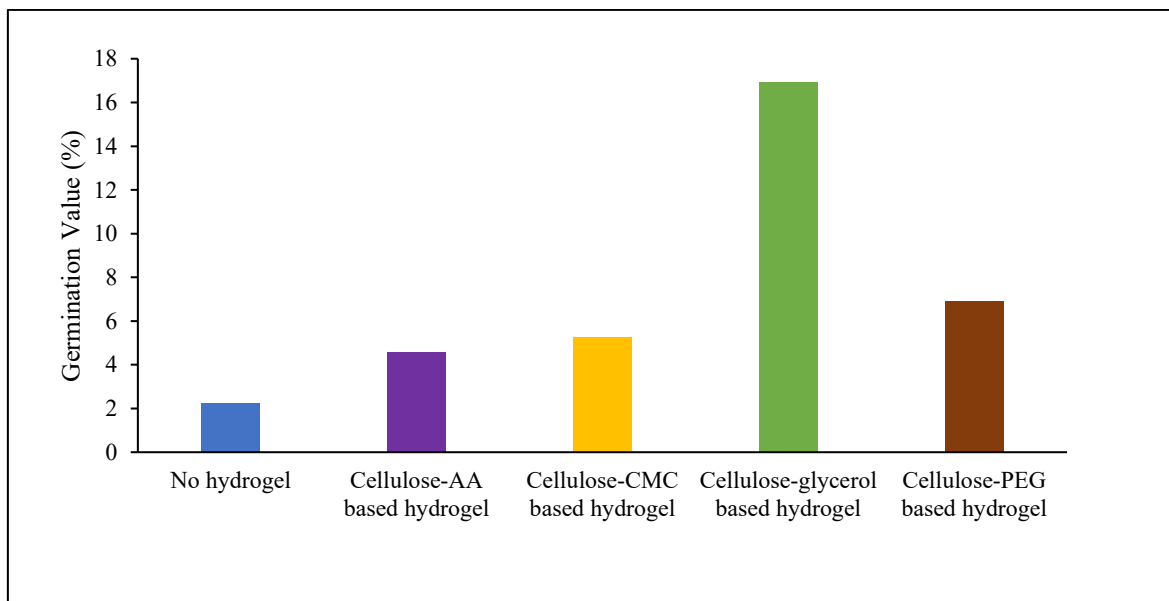


**Figure 4.42:** The germination value of Choy Sum vegetable seeds during the observation period

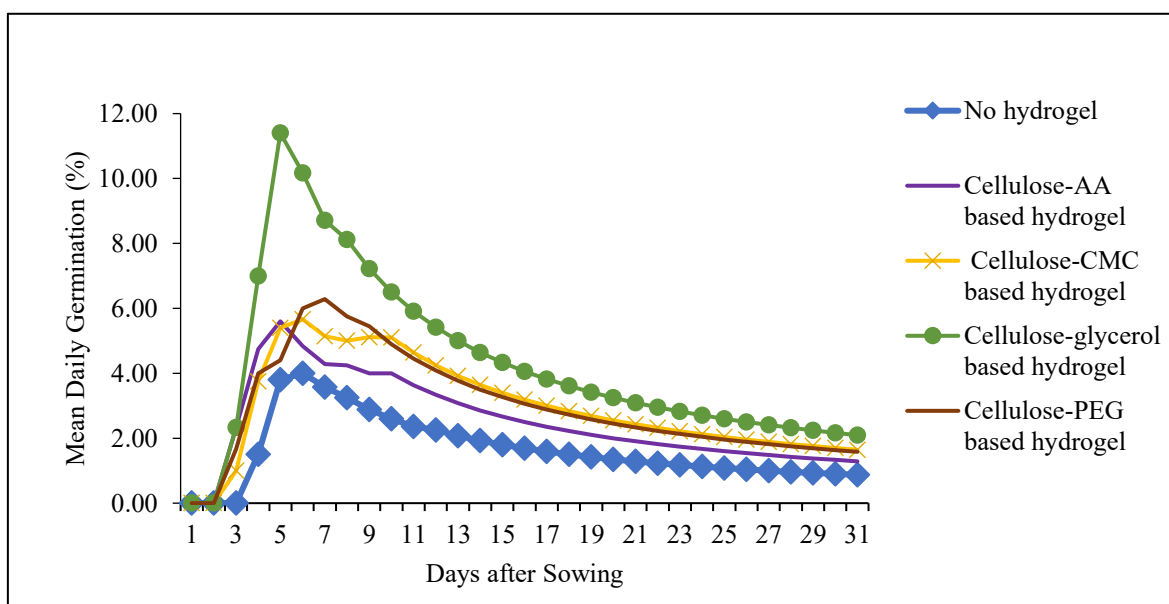


**Figure 4.43:** The mean daily germination percentage of the Choy Sum vegetable seeds

In conclusion, the cellulose-1.0g glycerol-based hydrogel is an ideal seed germination medium for germinating any seeds due to its excellent water uptake capacity. It can help the seeds thrive based on the water retained in the structure after 2 weeks without watering.

Thus, this hydrogel can be used as a soilless medium to germinate seeds, grow plants, and reduce the drought stress of the plant and seeds.

#### **4.9 Biodegradability of the hydrogel with various gelling agents**

This study evaluates the biodegradability of each cellulose-based hydrogel plasticised with those four types of gelling agents in typical garden soil, known as loamy soil. The experiment was set up as shown in Figure 4.45, starting on the first day of the experiment. Figure 4.44 demonstrates the percentage of weight loss of each cellulose hydrogel over 60 days. According to the data in Figure 4.44, the cellulose hydrogels containing CMC and alginic acid had already lost their weight by over 60 % after 14 days. However, the weight loss of the cellulose-glycerol-based hydrogels and cellulose-PEG-based hydrogels is not over 20 %, 18.99 % and 6.14 %, respectively. After 28 days, the hydrogels with glycerol and PEG had composted over 60 %. These results indicate that the cellulose hydrogels containing CMC and AA exhibit faster biodegradability compared to the other two cellulose hydrogels. However, the starting materials and the method used to form the hydrogel are the same. This may be because these two gelling agents, belonging to the biopolymer, produce a polymer that is easy to compost when buried in the loamy soil (Akalin & Pulat, 2018; Sreekumar & Bindhu, 2020). Moreover, the hydrogels containing 1.75g CMC and 2.0g AA had composted up to 96 % of the weight loss after being placed in the regular garden soil for 60 days. Meanwhile, the cellulose hydrogels contained glycerol, and PEG only composted up to 87 % of the weight loss after 60 days. The cellulose-PEG-based hydrogels are composted more slowly because PEG is a non-degradable polymer (Behjat et al., 2009). While glycerol, as the gelling agent in the formation of cellulose hydrogel, enhances the elasticity of the hydrogel, its elasticity may affect the slower rate of degradation (Pan et al., 2018). However, all types of cellulose-based hydrogels had a high degree of

biodegradability, and all the degradation of the hydrogels had reached 85 % and above on day 60, as shown in Figure 4.46. This finding is also supported by Tefera et al. (2022). This may be due to the glycosidic bonds present in the polysaccharide chains in the cellulose fibres. All the gelling agents used are biodegradable, which promotes enzymatic degradation, and the common enzymes can be found in garden soil, like amylase (Berradi et al., 2023). In conclusion, all the hydrogels with various gelling agents are biodegradable because all the hydrogels in this study are classified as biopolymers, which are derived from cellulosic waste.

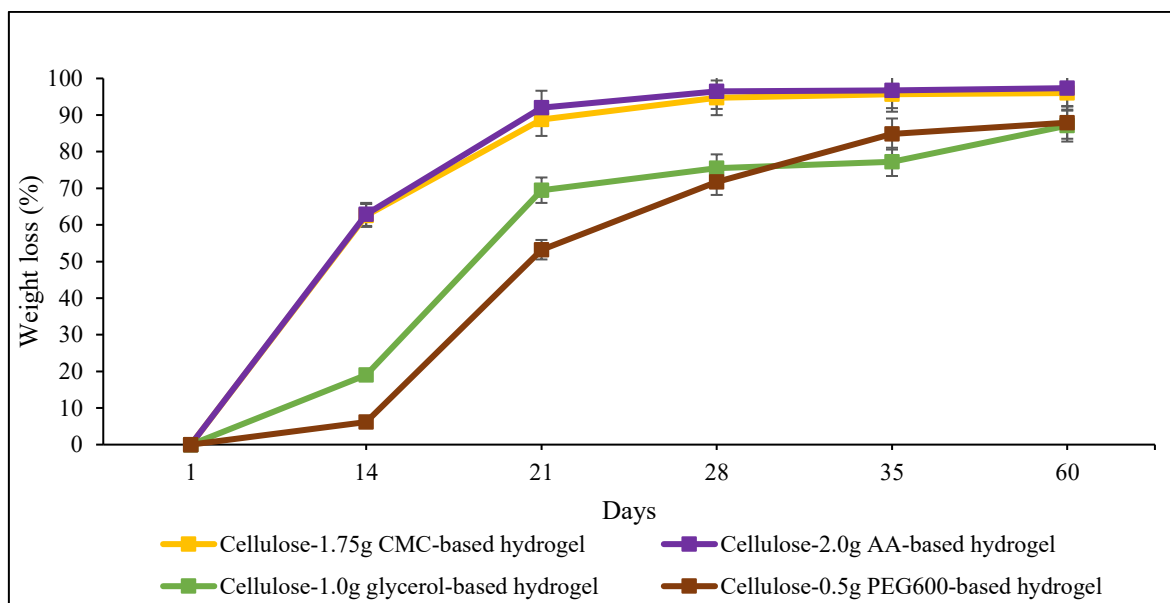


**Figure 4.44:** The biodegradability of the different types of cellulose-based hydrogels. The error bar represents the standard deviations from the duplicate (n=2)

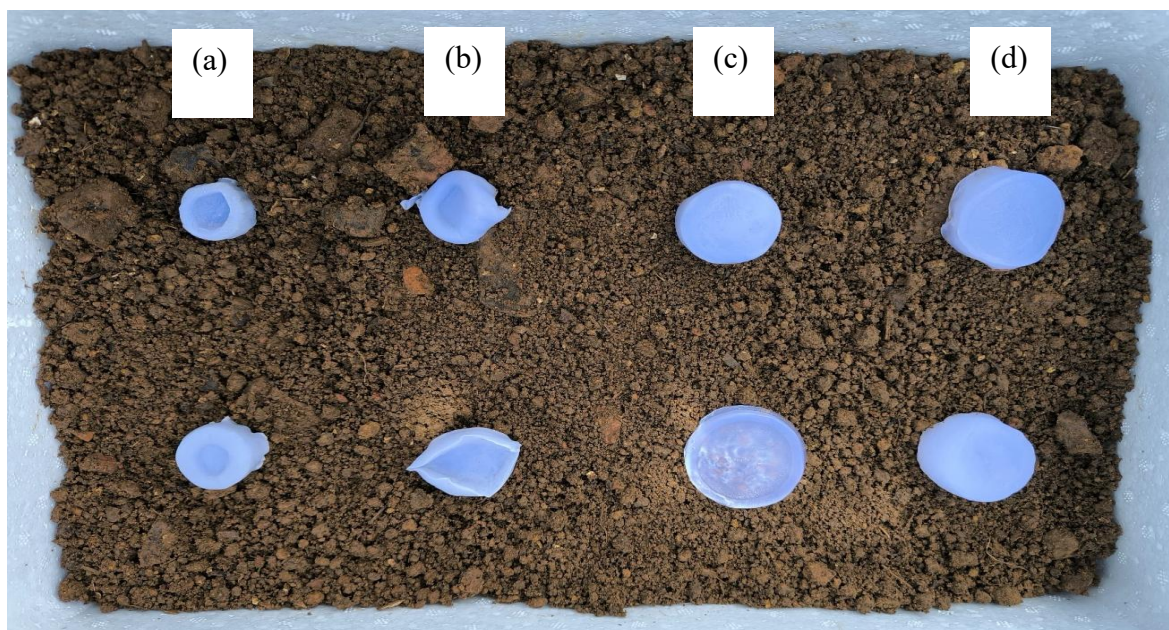


**Figure 4.45:** The observation of cellulose-based hydrogels with (a) 0.5g PEG600, (b) 1.0g glycerol, (c) 1.75g CMC, and (d) 2.0g AA on Day 1

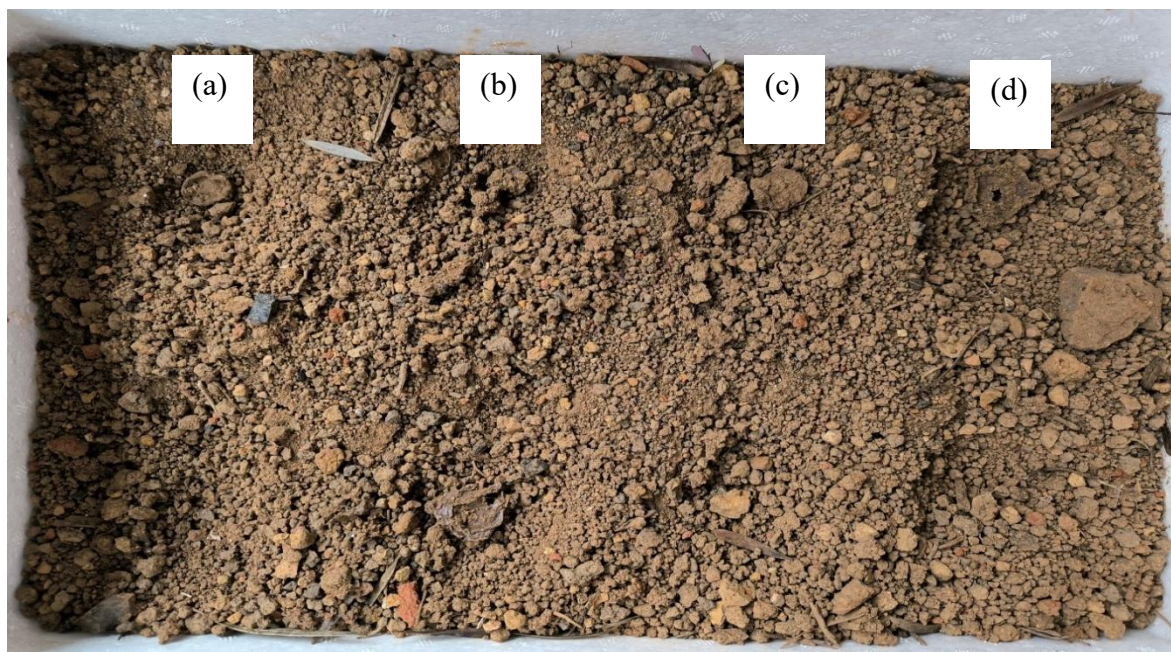


**Figure 4.46:** The observation of cellulose-based hydrogels with (a) 0.5g PEG600, (b) 1.0g glycerol, (c) 1.75g CMC, and (d) 2.0g AA on Day 60

#### 4.10 Chapter Summary

This chapter proved that the cellulose-based hydrogels plasticised with various gelling agents were successfully synthesised in this study. Each type of cellulose-based hydrogel has an excellent water uptake capacity and water retention rate with a high porosity structure in its surface morphologies. From the performances of types of cellulose-based hydrogel in agricultural applications, the cellulose hydrogels plasticised with CMC, glycerol, and PEG increase the soil moisture of various soil types and improve the seed germination rate of Choy Sum vegetables compared to the traditional germination medium.

## CHAPTER 5

### CONCLUSION AND RECOMMENDATIONS

#### 5.1 Conclusion

This study successfully synthesised cellulose-based hydrogels using various gelling agents, which are alginic acid (AA), carboxymethylcellulose (CMC), glycerol, and polyethylene glycol (PEG) with different concentrations (0.25g – 2.50g) of each gelling agent through chemical crosslinking. The cellulose was successfully extracted from the wastepaper through the alkaline treatment process in this study. FTIR analysis determined that lignin and hemicellulose in the cellulose had been removed during the delinking process.

In addition, the physical and chemical properties of the cellulose-based hydrogels were also determined, whereby the surface morphology of each hydrogel with different amounts of each gelling agent was successfully observed using the Field Emission Scanning Electron Microscope (FESEM) analysis. Fourier-transform infrared spectroscopy (FTIR) analysis successfully determined the presence of each hydrogel's functional groups. Furthermore, the swelling ratio and the water retention of types of cellulose hydrogels were also determined in this study. The cellulose-2.0g AA-based hydrogel, the cellulose-1.75g CMC-based hydrogel, the cellulose-1.0g glycerol-based hydrogel, and the cellulose-0.5g PEG600-based hydrogel had the excellent water uptake capacity, which with 808.30%, 2603.24%, 3562.97%, and 2697.49%, when compared among all the concentration of the gelling agents in each category. The cellulose-0.5g PEG600-based hydrogel had the highest water retention with 33.47% among all the gelling agents due to its smallest pore size of 11.95nm based on the Brunauer-Emmett-Teller (BET) analysis.

Lastly, the potential application of cellulose-based hydrogels as soil conditioners under various soil types (sand, topsoil, gley soil, and clayey soil) and seed germination was successfully evaluated. The cellulose-0.5g PEG600-based hydrogels mixed with sand, gley, and clayey soil had the highest water-holding capacity after 15 days, with 32.2 %, 41.3 %, and 20.5 %, respectively. Meanwhile, the cellulose-1.75g glycerol-based hydrogels mixed with topsoil had the highest water-holding capacity after 15 days, at 21.3 %. In addition, most of the Choy Sum seeds germinated on all the hydrogels with no water applied during the germination period. The hydrogel with 1.0g glycerol has the potential to become a water reservoir and seed germination medium for germinating the seeds because it has the highest seed germination with 65 % among all the hydrogels. The biodegradability of all types of cellulose hydrogel was also determined, and all the cellulose hydrogels plasticised with different gelling agents decomposed more than 85% in the loamy soil after 60 days, showing that all the hydrogels are biodegradable.

## **5.2 Recommendations**

For further studies on cellulose-based hydrogels, cellulose hydrogels plasticised with different gelling agents (alginic acid (AA), sodium carboxymethylcellulose (CMC), glycerol, and PEG) in this study can be used to evaluate their effect on the releasing rate of the fertiliser, either total nitrogen or using Nitrogen, Phosphorus, and Potassium (NPK) for their nutrient delivery ability to compare which hydrogel has the better releasing rate. Besides, for the seed, cultural applications can further study the plant growth assay and monitor the plant height, the size of the leaf, and the length of the stem to determine whether all types of hydrogels can produce soilless conditions for plants. Moreover, cellulose hydrogel frequently exhibits low mechanical strength, leading to its rupture when subjected to excessive water loading or significant external force. Therefore, future studies can apply

the cellulose hydrogel to a double crosslink network structure by combining it with another polymer material or nanoparticle without influencing their excellent water uptake and retention capacity.

Besides agricultural applications, cellulose-based hydrogels in this study can also be used for further studies in the biomedical field on drug delivery systems. The traditional medical hydrogel is often used to release active ingredients absorbed through our skin. Its purpose is to relieve some medical symptoms, like coughing, more directly than through our mouth. There is also a lack of research on cellulose hydrogels to replace traditional medical hydrogels. Synthetic polymers, biopolymers such as alginate, and semi-biopolymers are often used in making medical hydrogel. The cellulose-based hydrogels plasticised with various gelling agents in this study have the potential to replace medical hydrogels because of their excellent water uptake and retention ability. They may be used to test the drug delivery rate as a medical hydrogel to promise a sustainable and cost-effective alternative to making relief patches in future.

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## APPENDICES

### Appendix A: Journal Publications

1. **Jong, S. J., KarunaKaran, K., Wasli, M. E., Musa, Z., & Chin, S. F. (2024).** Cellulose-based hydrogel as a natural medium for paddy seed germination. *Starch/Stärke*, 76(1–2), 1–8. <https://doi.org/10.1002/star.202200234>

#### RESEARCH ARTICLE

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### Cellulose-Based Hydrogel as a Natural Medium for Paddy Seed Germination

Shu-Jun Jong, Kanchana KarunaKaran, Mohd Effendi Wasli, Zaki Musa, and Suk-Fun Chin\*

Cellulose hydrogel derived from cellulose fibers extracted from printed paper waste is prepared. The hydrogel derived from 3 wt% of the cellulose concentration exhibits a maximum water absorption capacity of 465.5%. The effects of the cellulose hydrogel on the soil moisture are evaluated using four types of soils namely river sand, clayey soil, loamy soil, and gley soil (the wetland soil). The potential application of cellulose-based hydrogel as an eco-friendly seed germination media of the paddy seeds under the soil-less condition is evaluated. This study shows that paddy seeds exhibit a higher seed germination rate of 53% when cellulose hydrogel is used as a seed germination media as compared to seed germination rate ranging from 14% to 35% when various types of soil are used as the germination media. The prepared cellulose hydrogel could be applied as an eco-friendly and cost-effective seed germination media to enhance the paddy seeds' germination rate.

#### 1. Introduction

Using swelling in its three-dimensional network, the crosslinked polymer known as hydrogel is able to absorb a significant quantity of water.<sup>[1]</sup> Cellulose is a favorable precursor material for hydrogel preparation due to its nature of being non-toxic, renewable, biodegradable, and abundantly available.<sup>[2,3]</sup> In line with the sustainable development concept, many researchers have attempted to prepare cellulose hydrogels using cellulose fibers isolated from cellulosic wastes like cotton linters, wastepaper, wood chips, and sago pith wastes.<sup>[4–7]</sup> The production of cellulosic

biomass, such as wastepaper, is around 57 000 tonnes per month in Malaysia.<sup>[8]</sup> Due to the fact that waste papers wind up in drains and rivers, they can cause significant environmental issues including flash floods and stagnant pools where mosquitoes thrive. Due to the use of renewable resources and potential to lessen environmental problems, waste paper that contains cellulose can serve as a starting point for agronomic cultivation without soil. Therefore, the superabsorbent hydrogel for the paddy germination medium was made using the cellulose fiber extracted from the wastepaper as the starting material in this study.

The amazing and unique characteristics of cellulose hydrogels allow them to become suitable materials in multidisciplinary areas, especially biomedical and agricultural applications.<sup>[9]</sup> In agriculture, cellulose-based hydrogels have been investigated as the water reservoir for soil moisture retention.<sup>[10–12]</sup> The cellulose hydrogel can absorb and store water molecules in the interstitial sites of their polymer matrix due to hydrophilic groups such as carboxyl groups and hydroxyl groups.<sup>[13]</sup> Thus, cellulose hydrogel could prevent rapid water loss via the evaporation process, thereby increasing water use efficiency, and reducing the irrigation frequency. So, hydrogels have been investigated as a seed germination medium because maintaining an optimum and consistent amount of moisture is crucial for seed germination. Cao and Li found that rapeseed seed germination and seedling development were enhanced in activated-carbon-filled agarose hydrogel growing medium.<sup>[14]</sup> In another study, Ismail et al. reported a starch/polyethylene glycol (PEG) hydrogel has a positive effect on the germination of maize seeds.<sup>[14–16]</sup> Cellulose anionic hydrogels also have been demonstrated to be a good simulator for sesame seed germination and growth.<sup>[14]</sup> In addition to serving as a seed germination medium and soil conditioner, cellulose-based hydrogel is frequently employed as a fertilizer releaser.<sup>[17]</sup>

So in this work, the cellulose-based hydrogel was synthesized by cellulose fibers isolated from printed paper wastes with cellulose derivative (sodium carboxymethylcellulose, NaCMC) was added as a plasticizer to promote gel formation and epichlorohydrin (ECH) was used as a crosslinker in the formation of hydrogel. The potential application of the cellulose hydrogel as the seed germination media for paddy seeds was evaluated.

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## Appendix B:

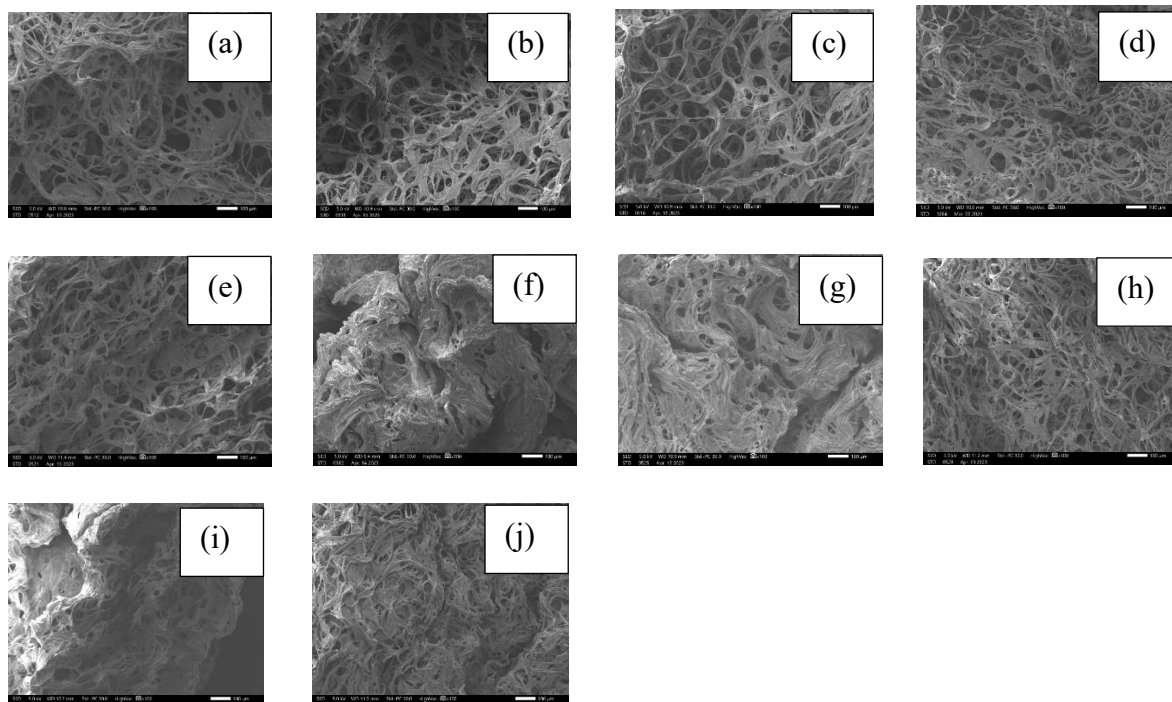


**Figure 1:** The surface morphology of the cellulose-based hydrogel with (a) 0.25g, (b) 0.5g, (c) 0.75g, (d) 1.0g, (e) 1.25g, (f) 1.50g, (g) 1.75g, (h) 2.0g, (i) 2.25g, (j) 2.5g of alginic acid at magnification 100×

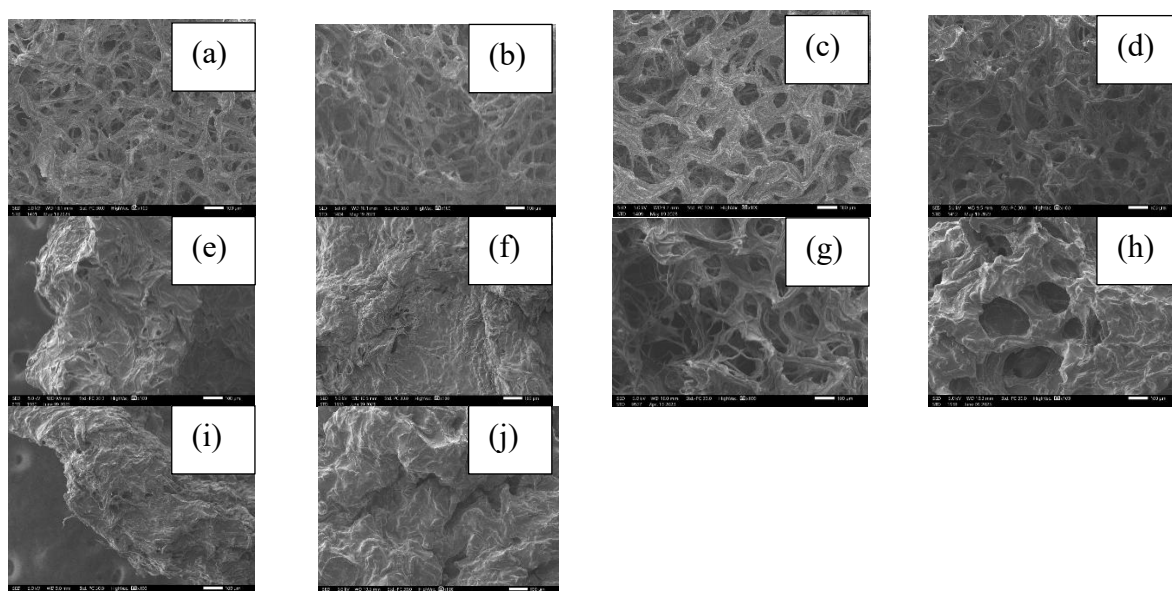


**Figure 2:** The surface morphology of the cellulose-based hydrogel with (a) 0.25g, (b) 0.5g, (c) 0.75g, (d) 1.0g, (e) 1.25g, (f) 1.50g, (g) 1.75g, (h) 2.0g, (i) 2.25g, (j) 2.5g of CMC at magnification 100×

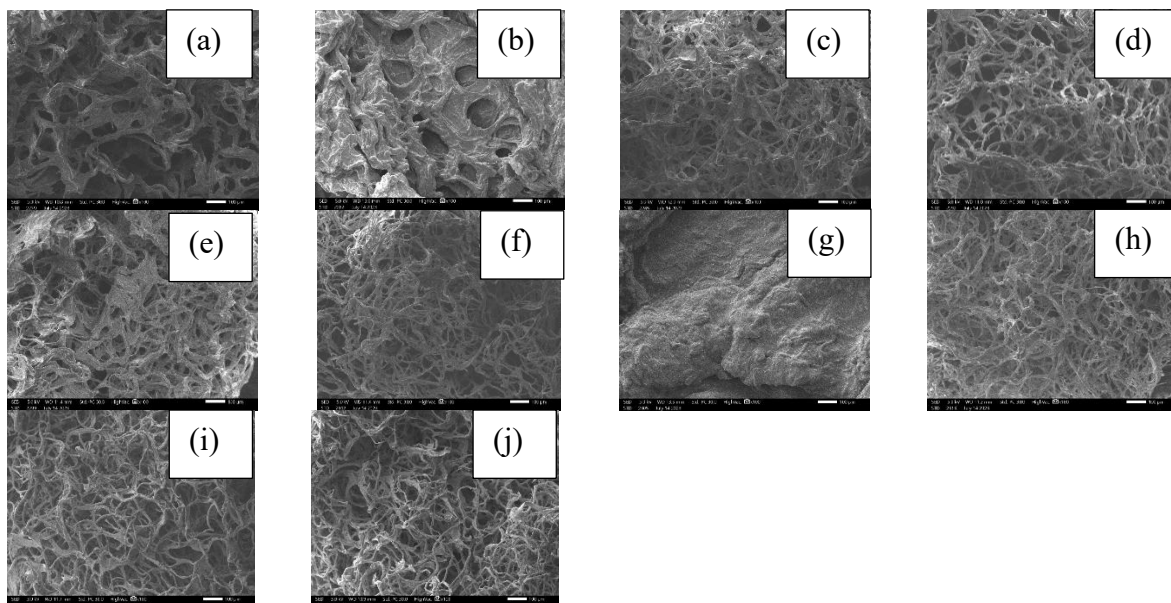


**Figure 3:** The surface morphology of the cellulose-based hydrogel with (a) 0.25g, (b) 0.5g, (c) 0.75g, (d) 1.0g, (e) 1.25g, (f) 1.50g, (g) 1.75g, (h) 2.0g, (i) 2.25g, (j) 2.5g of glycerol at magnification 100×

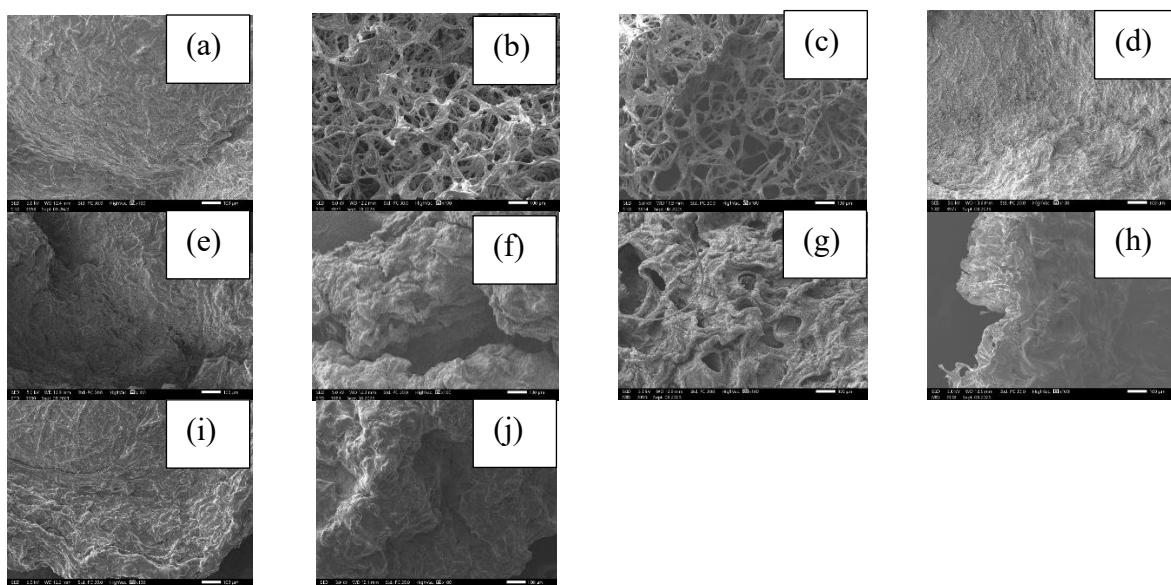


**Figure 4:** The surface morphology of the cellulose-based hydrogel with (a) 0.25g, (b) 0.5g, (c) 0.75g, (d) 1.0g, (e) 1.25g, (f) 1.50g, (g) 1.75g, (h) 2.0g, (i) 2.25g, (j) 2.5g of PEG400 at magnification 100×

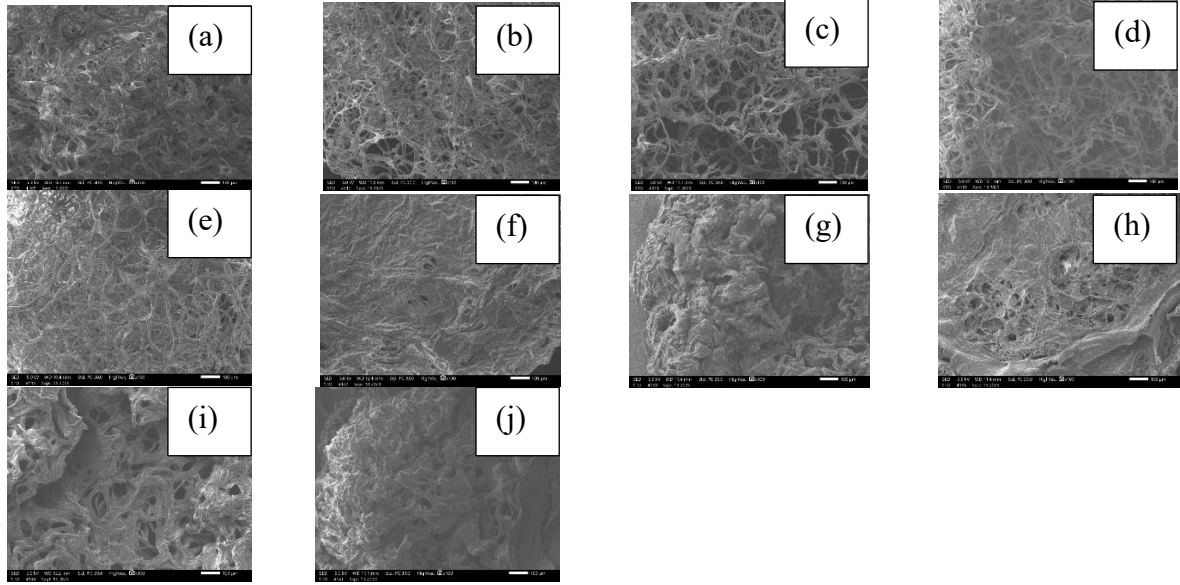


**Figure 5:** The surface morphology of the cellulose-based hydrogel with (a) 0.25g, (b) 0.5g, (c) 0.75g, (d) 1.0g, (e) 1.25g, (f) 1.50g, (g) 1.75g, (h) 2.0g, (i) 2.25g, (j) 2.5g of PEG600 at magnification 100×

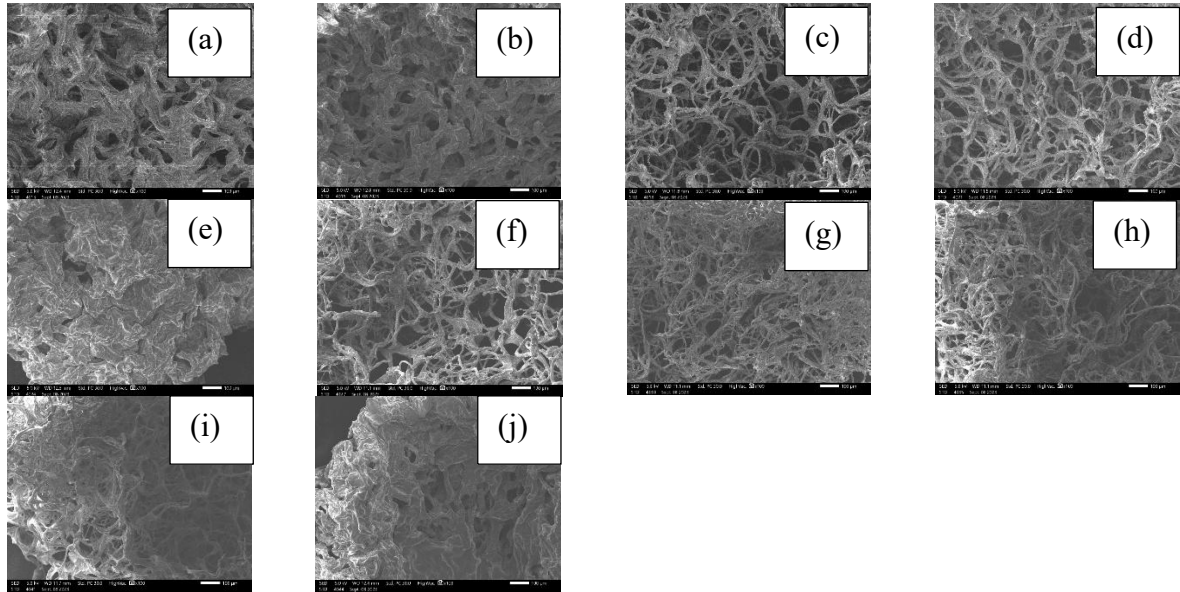


**Figure 6:** The surface morphology of the cellulose-based hydrogel with (a) 0.25g, (b) 0.5g, (c) 0.75g, (d) 1.0g, (e) 1.25g, (f) 1.50g, (g) 1.75g, (h) 2.0g, (i) 2.25g, (j) 2.5g of PEG1000 at magnification 100×

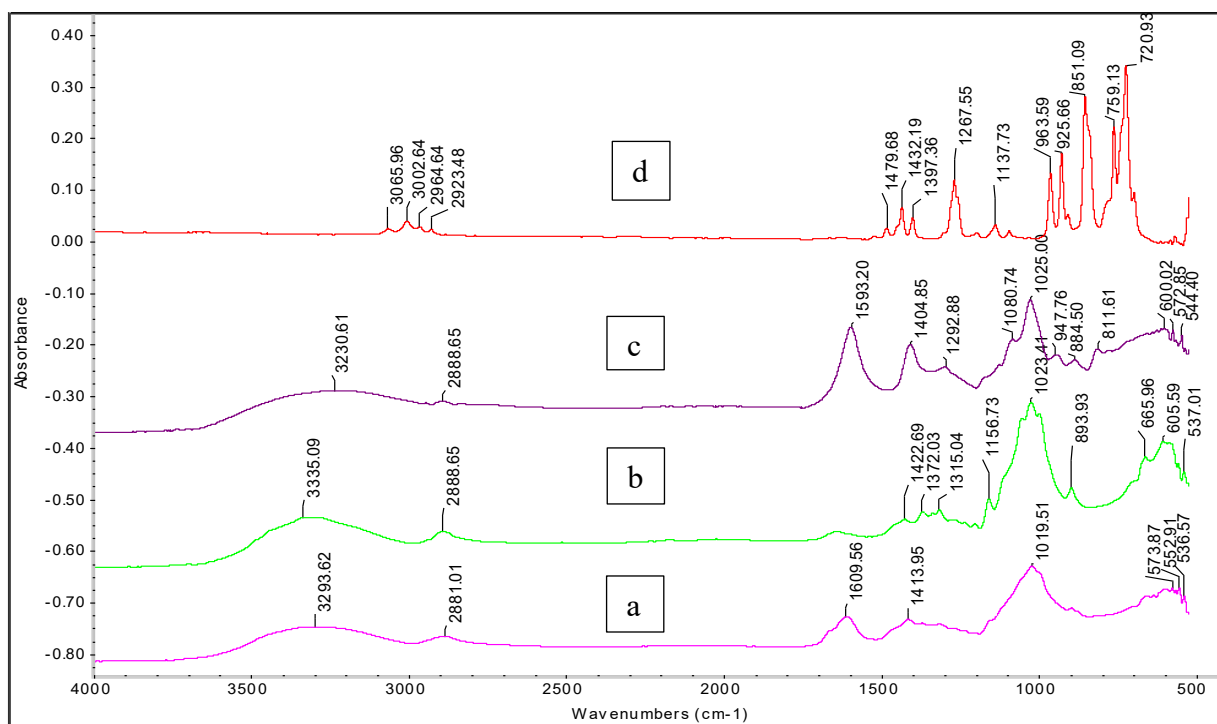


**Figure 7:** The FTIR spectra of (a) cellulose-AA-based hydrogel, (b) cellulose fibre extracted from wastepaper, (c) AA, (d) ECH

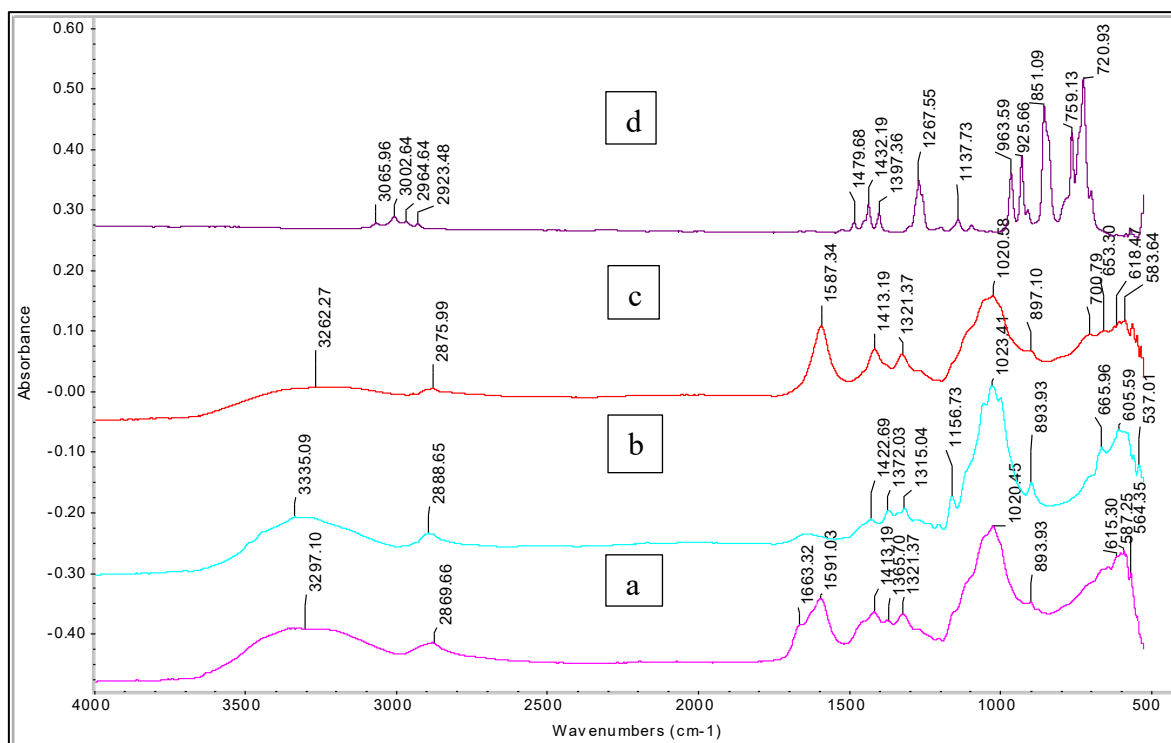


**Figure 8:** The FTIR spectra of (a) cellulose-CMC-based hydrogel, (b) cellulose fibre extracted from wastepaper, (c) CMC, (d) ECH

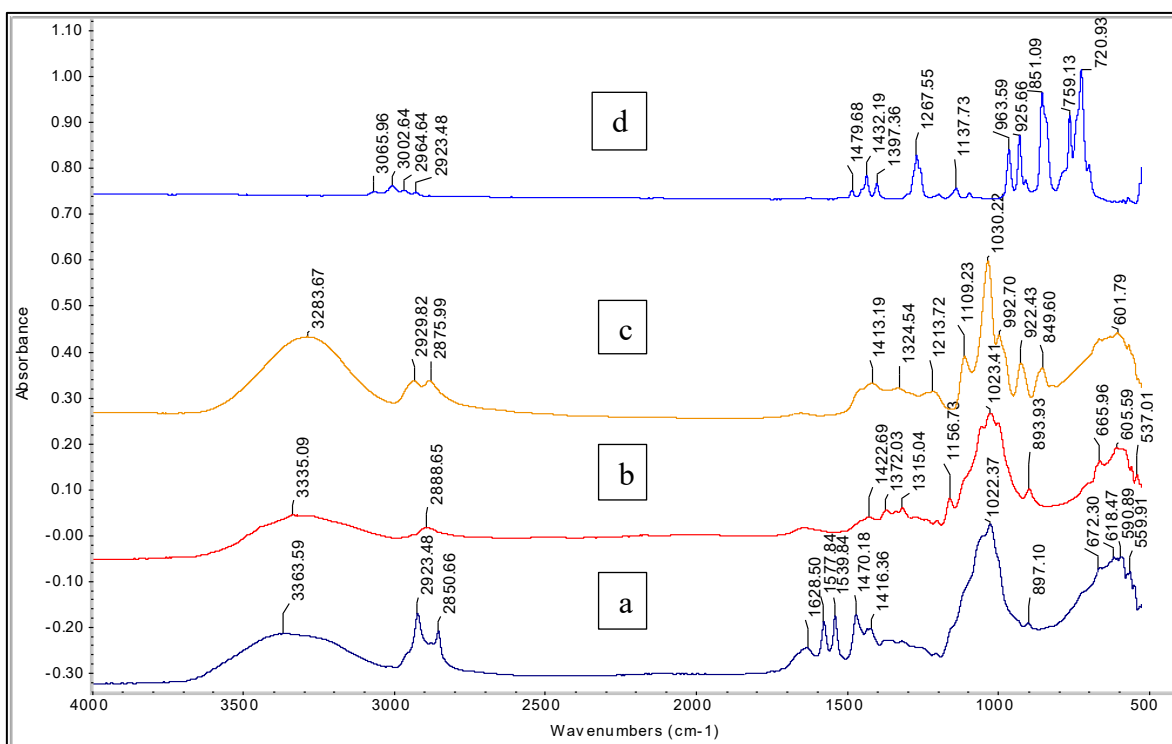


**Figure 9:** The FTIR spectra of (a) cellulose-glycerol-based hydrogel, (b) cellulose fibre extracted from wastepaper, (c) glycerol, (d) ECH

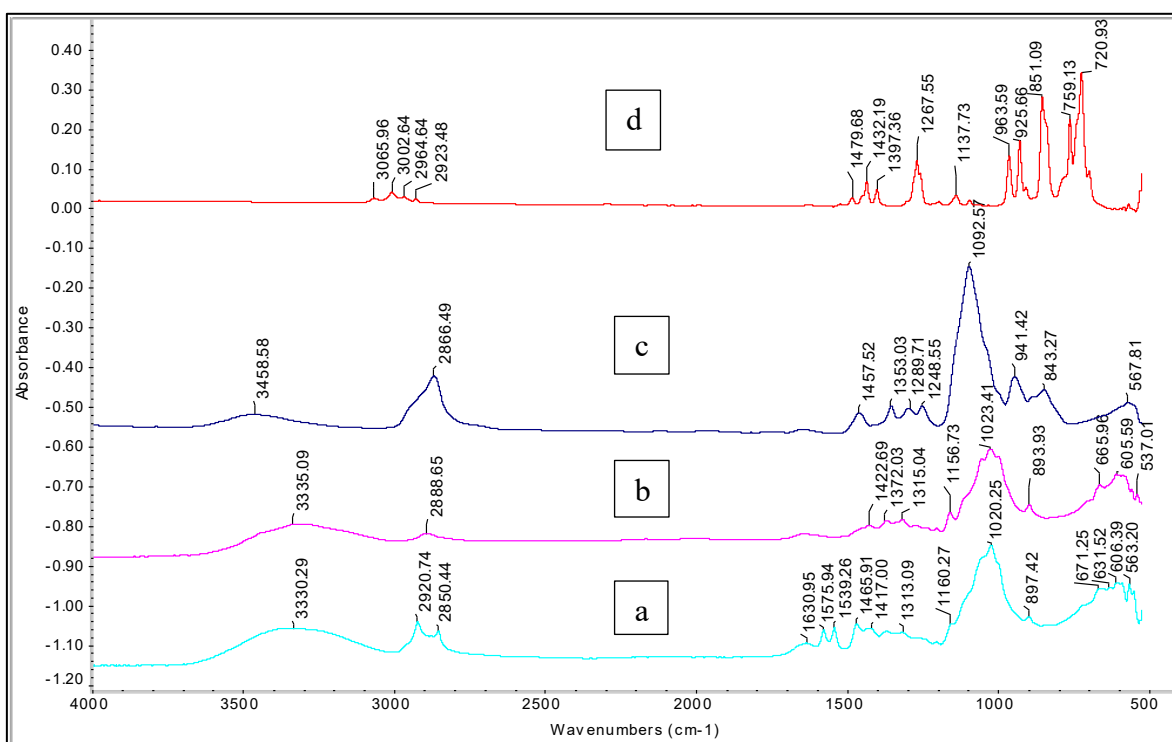


**Figure 10:** The FTIR spectra of (a) cellulose-PEG-based hydrogel, (b) cellulose fibre extracted from wastepaper, (c) PEG, (d) ECH

**Table 1:** The swelling ratio of cellulose-based hydrogels plasticised with different concentrations of AA.

| Time (h) | 0.25wt%                    | 0.50wt% | 0.75wt% | 1.00wt% | 1.25wt% | 1.50wt% | 1.75wt% | 2.00wt% | 2.25wt% | 2.50wt% |
|----------|----------------------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
|          | Average swelling ratio (%) |         |         |         |         |         |         |         |         |         |
| 1.0      | 356.27                     | 389.85  | 244.42  | 299.23  | 297.54  | 307.99  | 344.80  | 269.25  | 345.08  | 320.24  |
| 2.0      | 426.41                     | 448.36  | 333.38  | 377.61  | 347.94  | 372.47  | 507.91  | 458.64  | 389.37  | 378.57  |
| 3.0      | 456.94                     | 477.91  | 399.05  | 426.08  | 386.33  | 398.51  | 592.74  | 606.59  | 428.41  | 405.95  |
| 4.0      | 485.16                     | 497.59  | 449.22  | 472.47  | 405.77  | 410.85  | 657.15  | 730.28  | 408.57  | 425.79  |
| 5.0      | 497.07                     | 509.78  | 495.57  | 516.61  | 424.84  | 422.35  | 685.74  | 808.30  | 414.29  | 410.71  |

**Table 2:** The swelling ratio of cellulose-based hydrogels plasticised with different concentrations of CMC.

| Time (h) | 0.25wt%                    | 0.50wt% | 0.75wt% | 1.00wt% | 1.25wt% | 1.50wt% | 1.75wt% | 2.00wt% | 2.25wt% | 2.50wt% |
|----------|----------------------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
|          | Average swelling ratio (%) |         |         |         |         |         |         |         |         |         |
| 1.0      | 2179.41                    | 2008.12 | 2317.92 | 2320.27 | 1370.56 | 1695.82 | 1883.91 | 1202.72 | 1276.56 | 1364.62 |
| 2.0      | 2350.88                    | 2155.19 | 2437.44 | 2405.07 | 1313.35 | 1722.37 | 2057.39 | 1200.91 | 1401.49 | 1451.78 |
| 3.0      | 2528.22                    | 2344.30 | 2485.93 | 2618.17 | 1468.70 | 1733.62 | 2102.95 | 1284.40 | 1430.58 | 1453.75 |
| 4.0      | 2459.45                    | 2664.05 | 2698.81 | 2710.03 | 1486.52 | 1756.16 | 2246.93 | 1256.50 | 1575.33 | 1533.49 |
| 5.0      | 2355.79                    | 2360.71 | 2479.63 | 2603.24 | 1318.84 | 1539.96 | 2176.81 | 1176.19 | 1239.91 | 1296.47 |

**Table 3:** The swelling ratio of cellulose-based hydrogels plasticised with different concentrations of glycerol.

| Time (h) | 0.25wt%                    | 0.50wt% | 0.75wt% | 1.00wt% | 1.25wt% | 1.50wt% | 1.75wt% | 2.00wt% | 2.25wt% | 2.50wt% |
|----------|----------------------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
|          | Average swelling ratio (%) |         |         |         |         |         |         |         |         |         |
| 1.0      | 2667.31                    | 1047.83 | 3042.28 | 3356.24 | 2731.31 | 3457.88 | 814.53  | 2512.13 | 2594.89 | 2458.18 |
| 2.0      | 3259.39                    | 1268.05 | 3117.16 | 3314.56 | 2626.36 | 3557.37 | 934.13  | 2624.18 | 2640.53 | 2598.19 |
| 3.0      | 2908.01                    | 1531.69 | 3324.94 | 3549.68 | 2935.61 | 3515.36 | 1001.27 | 2622.80 | 2670.83 | 2728.15 |
| 4.0      | 3058.50                    | 1494.45 | 3374.72 | 3677.77 | 3007.16 | 3655.41 | 974.22  | 2668.30 | 2927.30 | 2895.73 |
| 5.0      | 3149.67                    | 1457.87 | 3309.31 | 3562.97 | 3117.67 | 4192.17 | 969.68  | 2682.90 | 2932.84 | 2930.41 |

**Table 4:** The swelling ratio of cellulose-based hydrogels plasticised with different concentrations of PEG 400.

| Time (h) | 0.25wt%                    | 0.50wt% | 0.75wt% | 1.00wt% | 1.25wt% | 1.50wt% | 1.75wt% | 2.00wt% | 2.25wt% | 2.50wt% |
|----------|----------------------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
|          | Average swelling ratio (%) |         |         |         |         |         |         |         |         |         |
| 1.0      | 845.77                     | 2414.96 | 2315.70 | 868.35  | 847.42  | 931.77  | 913.08  | 1078.22 | 990.80  | 980.31  |
| 2.0      | 992.45                     | 2365.75 | 2481.30 | 1014.06 | 1038.58 | 1039.58 | 999.69  | 1256.21 | 1102.71 | 1098.18 |
| 3.0      | 1067.10                    | 2502.30 | 2342.87 | 1045.26 | 1004.14 | 998.45  | 957.23  | 1100.96 | 1083.68 | 1103.00 |
| 4.0      | 1098.58                    | 2417.80 | 2351.70 | 1125.20 | 1045.32 | 1007.52 | 953.42  | 1167.64 | 1111.91 | 1118.74 |
| 5.0      | 1135.83                    | 2461.56 | 2460.33 | 1112.89 | 1089.61 | 1035.60 | 1045.00 | 1187.97 | 1124.03 | 1146.22 |

**Table 5:** The swelling ratio of cellulose-based hydrogels plasticised with different concentrations of PEG 600.

| Time (h) | 0.25wt%                    | 0.50wt% | 0.75wt% | 1.00wt% | 1.25wt% | 1.50wt% | 1.75wt% | 2.00wt% | 2.25wt% | 2.50wt% |
|----------|----------------------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
|          | Average swelling ratio (%) |         |         |         |         |         |         |         |         |         |
| 1.0      | 2417.93                    | 2407.90 | 2256.79 | 2247.08 | 2259.33 | 1263.08 | 1113.02 | 987.49  | 1212.37 | 1114.38 |
| 2.0      | 2496.44                    | 2499.83 | 2363.97 | 2295.94 | 2488.83 | 1401.36 | 1162.60 | 1098.72 | 1293.53 | 1200.49 |
| 3.0      | 2537.89                    | 2570.42 | 2424.05 | 2436.47 | 2555.95 | 1389.88 | 1198.01 | 1133.21 | 1397.71 | 1210.11 |
| 4.0      | 2550.30                    | 2470.26 | 2442.96 | 2461.15 | 2501.84 | 1430.58 | 1223.22 | 1120.74 | 1405.38 | 1275.00 |
| 5.0      | 2672.79                    | 2697.49 | 2488.85 | 2434.77 | 2582.92 | 1480.38 | 1280.21 | 1162.21 | 1374.95 | 1262.01 |

**Table 6:** The swelling ratio of cellulose-based hydrogels plasticised with different concentrations of PEG 1000.

| Time (h) | 0.25wt%                    | 0.50wt% | 0.75wt% | 1.00wt% | 1.25wt% | 1.50wt% | 1.75wt% | 2.00wt% | 2.25wt% | 2.50wt% |
|----------|----------------------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
|          | Average swelling ratio (%) |         |         |         |         |         |         |         |         |         |
| 1.0      | 1548.73                    | 1089.71 | 2655.82 | 2725.34 | 715.24  | 2430.08 | 2425.01 | 2502.97 | 2537.36 | 1249.65 |
| 2.0      | 1552.27                    | 1259.56 | 2722.31 | 2784.05 | 876.27  | 2803.01 | 2674.49 | 2719.33 | 2780.67 | 1358.34 |
| 3.0      | 1639.31                    | 1265.21 | 2756.36 | 2785.20 | 967.39  | 2768.45 | 2621.33 | 2755.72 | 2822.29 | 1342.69 |
| 4.0      | 1653.74                    | 1354.16 | 2723.18 | 2692.43 | 1035.39 | 2837.17 | 2702.66 | 2669.89 | 2683.63 | 1279.44 |
| 5.0      | 1716.18                    | 1343.15 | 2792.06 | 2757.07 | 1036.78 | 2716.32 | 2519.69 | 2618.15 | 2646.92 | 1287.75 |

**Table 7:** The water retention rate of the cellulose-AA-based hydrogels.

| Time (h) | 0.25 wt%                         | 0.50wt% | 0.75wt% | 1.00wt% | 1.25wt% | 1.50wt% | 1.75wt% | 2.00wt% | 2.25wt% | 2.50wt% |
|----------|----------------------------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
|          | Average water retention rate (%) |         |         |         |         |         |         |         |         |         |
| 0        | 100.00                           | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  |
| 24       | 18.07                            | 17.18   | 40.00   | 35.42   | 13.20   | 19.93   | 46.44   | 76.06   | 17.01   | 10.44   |
| 48       | 0.50                             | 3.31    | 9.35    | 5.27    | 0.00    | 0.50    | 14.37   | 55.33   | 0.65    | 1.16    |
| 72       | 0.00                             | 0.12    | 0.63    | 0.00    | 0.00    | 0.00    | 2.24    | 35.51   | 0.00    | 1.16    |
| 96       | 0.00                             | 0.12    | 0.09    | 0.00    | 0.00    | 0.00    | 0.08    | 9.24    | 0.00    | 0.93    |

**Table 8:** The water retention rate of the cellulose-CMC-based hydrogels.

| Time (h) | 0.25 wt%                         | 0.50wt% | 0.75wt% | 1.00wt% | 1.25wt% | 1.50wt% | 1.75wt% | 2.00wt% | 2.25wt% | 2.50wt% |
|----------|----------------------------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
|          | Average water retention rate (%) |         |         |         |         |         |         |         |         |         |
| 0        | 100.00                           | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  |
| 24       | 69.99                            | 78.46   | 77.94   | 73.08   | 62.01   | 52.44   | 82.77   | 63.03   | 60.00   | 64.76   |
| 48       | 39.63                            | 63.80   | 64.96   | 62.17   | 28.89   | 15.34   | 60.67   | 30.60   | 25.16   | 33.17   |
| 72       | 1.30                             | 48.50   | 28.29   | 46.58   | 4.50    | 0.16    | 40.68   | 5.53    | 2.82    | 7.39    |
| 96       | 0.12                             | 0.04    | 0.28    | 0.27    | 0.42    | 0.02    | 11.95   | 0.10    | 0.38    | 0.06    |

**Table 9:** The water retention rate of the cellulose-glycerol-based hydrogels.

| Time (h) | 0.25 wt%                         | 0.50wt% | 0.75wt% | 1.00wt% | 1.25wt% | 1.50wt% | 1.75wt% | 2.00wt% | 2.25wt% | 2.50wt% |
|----------|----------------------------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
|          | Average water retention rate (%) |         |         |         |         |         |         |         |         |         |
| 0        | 100.00                           | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  |
| 24       | 82.48                            | 70.40   | 83.40   | 80.79   | 77.62   | 82.82   | 58.85   | 79.32   | 78.04   | 77.56   |
| 48       | 58.28                            | 9.90    | 65.97   | 66.45   | 56.35   | 55.65   | 1.06    | 35.53   | 31.97   | 29.46   |
| 72       | 16.55                            | 1.75    | 27.92   | 31.82   | 16.38   | 24.01   | 0.23    | 14.53   | 9.49    | 5.54    |
| 96       | 0.08                             | 0.00    | 4.32    | 10.48   | 0.13    | 4.32    | 0.00    | 8.10    | 2.57    | 1.55    |

**Table 10:** The water retention rate of the cellulose-PEG 400-based hydrogels.

| Time (h) | 0.25 wt%                         | 0.50wt% | 0.75wt% | 1.00wt% | 1.25wt% | 1.50wt% | 1.75wt% | 2.00wt% | 2.25wt% | 2.50wt% |
|----------|----------------------------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
|          | Average water retention rate (%) |         |         |         |         |         |         |         |         |         |
| 0        | 100.00                           | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  |
| 24       | 73.37                            | 85.27   | 84.65   | 77.22   | 72.23   | 71.46   | 72.41   | 77.60   | 74.34   | 74.38   |
| 48       | 41.19                            | 66.23   | 65.32   | 57.91   | 38.75   | 36.28   | 37.25   | 47.55   | 40.92   | 42.86   |
| 72       | 23.56                            | 48.39   | 47.09   | 21.40   | 11.92   | 9.16    | 9.87    | 19.52   | 13.14   | 15.91   |
| 96       | 0.37                             | 34.31   | 32.80   | 4.88    | 0.63    | 0.28    | 0.38    | 3.02    | 0.51    | 1.52    |

**Table 11:** The water retention rate of the cellulose-PEG 600-based hydrogels.

| Time (h) | 0.25 wt%                         | 0.50wt% | 0.75wt% | 1.00wt% | 1.25wt% | 1.50wt% | 1.75wt% | 2.00wt% | 2.25wt% | 2.50wt% |
|----------|----------------------------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
|          | Average water retention rate (%) |         |         |         |         |         |         |         |         |         |
| 0        | 100.00                           | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  |
| 24       | 83.24                            | 85.42   | 85.86   | 85.22   | 85.40   | 77.55   | 73.60   | 72.88   | 75.42   | 74.10   |
| 48       | 60.76                            | 66.38   | 66.86   | 71.28   | 66.16   | 46.48   | 38.60   | 37.25   | 43.05   | 39.80   |
| 72       | 40.11                            | 48.43   | 48.62   | 46.52   | 47.89   | 18.48   | 8.72    | 9.43    | 13.64   | 11.21   |
| 96       | 22.61                            | 33.47   | 33.34   | 31.18   | 33.16   | 1.53    | 0.00    | 0.00    | 0.09    | 0.00    |

**Table 12:** The water retention rate of the cellulose-PEG 1000-based hydrogels.

| Time (h) | 0.25 wt%                         | 0.50wt% | 0.75wt% | 1.00wt% | 1.25wt% | 1.50wt% | 1.75wt% | 2.00wt% | 2.25wt% | 2.50wt% |
|----------|----------------------------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
|          | Average water retention rate (%) |         |         |         |         |         |         |         |         |         |
| 0        | 100.00                           | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  | 100.00  |
| 24       | 72.22                            | 70.94   | 83.40   | 83.70   | 60.29   | 82.70   | 81.79   | 83.00   | 82.41   | 73.02   |
| 48       | 37.34                            | 35.16   | 62.75   | 62.64   | 18.37   | 61.84   | 60.34   | 62.39   | 61.15   | 39.73   |
| 72       | 7.28                             | 6.72    | 42.77   | 42.05   | 0.13    | 40.81   | 39.73   | 43.08   | 41.15   | 10.53   |
| 96       | 0.24                             | 0.24    | 26.07   | 24.85   | 0.04    | 23.85   | 22.74   | 26.61   | 24.06   | 0.21    |

**Table 13:** The water retention rate of types of cellulose-based hydrogels

| Types of cellulose-based hydrogels     | Water retention rate after 96 hours |
|----------------------------------------|-------------------------------------|
| Cellulose-2.0g AA-based hydrogel       | 9.24%                               |
| Cellulose-1.75g CMC-based hydrogel     | 11.95%                              |
| Cellulose-1.0g glycerol-based hydrogel | 10.48%                              |
| Cellulose-0.5g PEG600-based hydrogel   | 33.47%                              |

**Table 14:** The effect of types of cellulose-based hydrogel on the soil moisture in the sand.

| Day                                     | Soil moisture (%) |      |      |      |      |
|-----------------------------------------|-------------------|------|------|------|------|
|                                         | 1                 | 3    | 7    | 11   | 15   |
| No hydrogel                             | 0.0               | 0.0  | 0.0  | 0.0  | 0.0  |
| Cellulose-1.75g CMC-based hydrogels     | 20.0              | 21.9 | 21.1 | 20.9 | 24.5 |
| Cellulose-2.0g AA-based hydrogels       | 19.3              | 14.4 | 18.5 | 15.9 | 15.6 |
| Cellulose-1.0g glycerol-based hydrogels | 10.9              | 12.3 | 19.9 | 19.9 | 21.1 |
| Cellulose-0.5g PEG600-based hydrogel    | 12.8              | 14.7 | 21.4 | 24.2 | 32.2 |

**Table 15:** The effect of types of cellulose-based hydrogel on soil moisture in the topsoil.

| Day                                     | Soil moisture (%) |      |      |      |      |
|-----------------------------------------|-------------------|------|------|------|------|
|                                         | 1                 | 3    | 7    | 11   | 15   |
| No hydrogel                             | 0.0               | 0.0  | 0.0  | 0.0  | 0.0  |
| Cellulose-1.75g CMC-based hydrogels     | 12.3              | 15.2 | 14.6 | 15.6 | 16.4 |
| Cellulose-2.0g AA-based hydrogels       | 12.0              | 13.1 | 11.9 | 18.1 | 8.9  |
| Cellulose-1.0g glycerol-based hydrogels | 9.6               | 13.6 | 29.2 | 18.5 | 21.3 |
| Cellulose-0.5g PEG600-based hydrogel    | 14.1              | 10.5 | 19.9 | 19.0 | 16.6 |

**Table 16:** The effect of types of cellulose-based hydrogel on the soil moisture in the gley soil.

| Day                                     | Soil moisture (%) |      |      |      |      |
|-----------------------------------------|-------------------|------|------|------|------|
|                                         | 1                 | 3    | 7    | 11   | 15   |
| No hydrogel                             | 0.0               | 0.0  | 0.0  | 0.0  | 0.0  |
| Cellulose-1.75g CMC-based hydrogels     | 11.3              | 13.0 | 14.4 | 22.6 | 21.1 |
| Cellulose-2.0g AA-based hydrogels       | 12.9              | 13.6 | 18.1 | 16.2 | 10.7 |
| Cellulose-1.0g glycerol-based hydrogels | 12.7              | 20.0 | 19.6 | 18.1 | 22.8 |
| Cellulose-0.5g PEG600-based hydrogel    | 16.5              | 10.8 | 18.5 | 21.6 | 41.3 |

**Table 17:** The effect of types of cellulose-based hydrogel on the soil moisture in the clayed soil.

| Day                                     | Soil moisture (%) |      |      |      |      |
|-----------------------------------------|-------------------|------|------|------|------|
|                                         | 1                 | 3    | 7    | 11   | 15   |
| No hydrogel                             | 0.0               | 0.0  | 0.0  | 0.0  | 0.0  |
| Cellulose-0.75g CMC-based hydrogels     | 11.4              | 12.1 | 15.8 | 18.9 | 16.7 |
| Cellulose-2.0g AA-based hydrogels       | 13.0              | 9.7  | 5.7  | 12.7 | 14.0 |
| Cellulose-1.0g glycerol-based hydrogels | 2.8               | 15.2 | 8.6  | 17.4 | 14.6 |
| Cellulose-0.5g PEG600-based hydrogel    | 5.1               | 12.7 | 19.6 | 21.4 | 20.5 |

**Table 18:** The effect of types of cellulose-based hydrogel on the sand's pH.

| Day                                          | pH   |      |      |      |      |
|----------------------------------------------|------|------|------|------|------|
|                                              | 1    | 3    | 7    | 11   | 15   |
| No hydrogel                                  | 7.20 | 7.20 | 7.20 | 7.20 | 7.20 |
| with cellulose-1.75g CMC-based hydrogels     | 7.07 | 8.51 | 8.72 | 8.35 | 8.02 |
| with cellulose-2.0g AA-based hydrogels       | 7.61 | 8.27 | 8.55 | 8.76 | 8.16 |
| With cellulose-1.0g glycerol-based hydrogels | 8.00 | 9.48 | 9.44 | 9.41 | 9.08 |
| with cellulose-0.5g PEG600-based hydrogels   | 7.69 | 9.11 | 9.45 | 9.19 | 8.79 |

**Table 19:** The effect of types of cellulose-based hydrogel on the topsoil's pH

| Day                                          | pH   |      |      |      |      |
|----------------------------------------------|------|------|------|------|------|
|                                              | 1    | 3    | 7    | 11   | 15   |
| No hydrogel                                  | 5.00 | 5.00 | 5.00 | 5.00 | 5.00 |
| With cellulose-1.75g CMC-based hydrogels     | 5.88 | 6.28 | 6.67 | 6.10 | 6.53 |
| With cellulose-2.0g AA-based hydrogels       | 5.75 | 5.35 | 5.92 | 6.22 | 5.69 |
| With cellulose-1.0g glycerol-based hydrogels | 7.11 | 7.92 | 8.57 | 8.22 | 7.77 |
| With cellulose-0.5g PEG600-based hydrogels   | 6.03 | 6.61 | 8.07 | 8.19 | 7.84 |

**Table 20:** The effect of types of cellulose-based hydrogel on the gley soil's pH.

| Day                                          | pH   |      |      |      |      |
|----------------------------------------------|------|------|------|------|------|
|                                              | 1    | 3    | 7    | 11   | 15   |
| No hydrogel                                  | 4.58 | 4.58 | 4.58 | 4.58 | 4.58 |
| With cellulose-1.75g CMC-based hydrogels     | 5.30 | 6.27 | 6.00 | 6.00 | 6.31 |
| With cellulose-2.0g AA-based hydrogels       | 4.95 | 5.37 | 5.44 | 5.28 | 5.57 |
| With cellulose-1.0g glycerol-based hydrogels | 5.62 | 6.72 | 7.31 | 7.99 | 7.84 |
| With cellulose-0.5g PEG600-based hydrogels   | 4.95 | 6.29 | 6.38 | 6.84 | 7.44 |

**Table 21:** The effect of types of cellulose-based hydrogel on the clayey soil's pH.

| Day                                          | pH   |      |      |      |      |
|----------------------------------------------|------|------|------|------|------|
|                                              | 1    | 3    | 7    | 11   | 15   |
| No hydrogel                                  | 5.12 | 5.12 | 5.12 | 5.12 | 5.12 |
| With cellulose-1.75g CMC-based hydrogels     | 5.27 | 5.45 | 5.97 | 6.55 | 5.94 |
| With cellulose-2.0g AA-based hydrogels       | 5.36 | 5.67 | 6.37 | 6.45 | 6.43 |
| With cellulose-1.0g glycerol-based hydrogels | 6.82 | 7.08 | 8.21 | 7.50 | 6.94 |
| With cellulose-0.5g PEG600-based hydrogels   | 5.11 | 6.88 | 7.30 | 7.45 | 7.14 |

**Table 22:** The effect of types of cellulose-based hydrogel on the sand's electrical conductivity.

| Day                                          | Electrical conductivity ( $\mu\text{s}/\text{cm}$ ) |       |       |       |       |
|----------------------------------------------|-----------------------------------------------------|-------|-------|-------|-------|
|                                              | 1                                                   | 3     | 7     | 11    | 15    |
| No hydrogel                                  | 63.9                                                | 63.9  | 63.9  | 63.9  | 63.9  |
| With cellulose-1.75g CMC-based hydrogels     | 64.8                                                | 53.0  | 101.8 | 377.7 | 803.3 |
| With cellulose-2.0g AA-based hydrogels       | 47.2                                                | 63.9  | 88.9  | 97.7  | 113.9 |
| With cellulose-1.0g glycerol-based hydrogels | 526.3                                               | 99.2  | 235.7 | 288.3 | 144.3 |
| With cellulose-0.5g PEG600-based hydrogels   | 81.7                                                | 153.5 | 168.7 | 278.0 | 257.3 |

**Table 23:** The effect of types of cellulose-based hydrogel on the topsoil's electrical conductivity.

| Day                                          | Electrical conductivity ( $\mu\text{s}/\text{cm}$ ) |       |       |       |       |
|----------------------------------------------|-----------------------------------------------------|-------|-------|-------|-------|
|                                              | 1                                                   | 3     | 7     | 11    | 15    |
| No hydrogel                                  | 44.2                                                | 44.2  | 44.2  | 44.2  | 44.2  |
| With cellulose-1.75g CMC-based hydrogels     | 81.8                                                | 99.5  | 136.5 | 98.0  | 136.6 |
| With cellulose-2.0g AA-based hydrogels       | 50.0                                                | 41.7  | 38.1  | 91.0  | 50.3  |
| With cellulose-1.0g glycerol-based hydrogels | 217.3                                               | 557.0 | 494.0 | 601.7 | 572.0 |
| With cellulose-0.5g PEG600-based hydrogels   | 80.9                                                | 157.1 | 683.3 | 716.0 | 689.7 |

**Table 24:** The effect of types of cellulose-based hydrogel on the gley soil's electrical conductivity.

| Day                                          | Electrical conductivity ( $\mu\text{S}/\text{cm}$ ) |       |       |       |       |
|----------------------------------------------|-----------------------------------------------------|-------|-------|-------|-------|
|                                              | 1                                                   | 3     | 7     | 11    | 15    |
| No hydrogel                                  | 55.0                                                | 55.0  | 55.0  | 55.0  | 55.0  |
| With cellulose-1.75g CMC-based hydrogels     | 80.3                                                | 126.2 | 121.1 | 145.7 | 127.8 |
| With cellulose-2.0g AA-based hydrogels       | 51.2                                                | 55.6  | 84.2  | 95.0  | 110.0 |
| With cellulose-1.0g glycerol-based hydrogels | 115.1                                               | 233.7 | 271.7 | 366.7 | 338.0 |
| With cellulose-0.5g PEG600-based hydrogels   | 78.9                                                | 147.8 | 277.7 | 377.7 | 446.7 |

**Table 25:** The effect of types of cellulose-based hydrogel on the clayey soil's electrical conductivity.

| Day                                          | Electrical conductivity ( $\mu\text{S}/\text{cm}$ ) |       |       |       |       |
|----------------------------------------------|-----------------------------------------------------|-------|-------|-------|-------|
|                                              | 1                                                   | 3     | 7     | 11    | 15    |
| No hydrogel                                  | 113.9                                               | 113.9 | 113.9 | 113.9 | 113.9 |
| With cellulose-1.75g CMC-based hydrogels     | 223.7                                               | 267.3 | 86.8  | 209   | 122.1 |
| With cellulose-2.0g AA-based hydrogels       | 140.7                                               | 100.1 | 131.9 | 153.3 | 152.7 |
| With cellulose-1.0g glycerol-based hydrogels | 117.3                                               | 177.3 | 138.8 | 254.7 | 336.3 |
| With cellulose-0.5g PEG600-based hydrogels   | 241.0                                               | 237.7 | 265.3 | 323.7 | 386.7 |

**Table 26:** The germination rate of the Choy Sum vegetable seeds during the observation period.

| Types of cellulose-based hydrogel      | Germination rate (%) |
|----------------------------------------|----------------------|
| No hydrogel                            | 27                   |
| Cellulose-2.0g AA-based hydrogel       | 40                   |
| Cellulose-1.75g CMC-based hydrogel     | 51                   |
| Cellulose-1.0g glycerol-based hydrogel | 65                   |
| Cellulose-0.5g PEG600-based hydrogel   | 49                   |

**Table 27:** The germination value of Choy Sum vegetable seeds during the observation period.

| Types of cellulose-based hydrogel      | Germination value (%) |
|----------------------------------------|-----------------------|
| No hydrogel                            | 2.25                  |
| Cellulose-2.0g AA-based hydrogel       | 4.58                  |
| Cellulose-1.75g CMC-based hydrogel     | 5.28                  |
| Cellulose-1.0g glycerol-based hydrogel | 16.91                 |
| Cellulose-0.5g PEG600-based hydrogel   | 6.89                  |

**Table 28:** The mean daily germination percentage of the Choy Sum vegetable seeds.

| Days after Sowing | Mean daily germination percentage (%) |                                  |                                    |                                        |                                      |
|-------------------|---------------------------------------|----------------------------------|------------------------------------|----------------------------------------|--------------------------------------|
|                   | No hydrogel                           | Cellulose-2.0g AA based hydrogel | Cellulose-1.75g CMC based hydrogel | Cellulose-1.0g glycerol based hydrogel | Cellulose-0.5g PEG600 based hydrogel |
| 1                 | 0.00                                  | 0.00                             | 0.00                               | 0.00                                   | 0.00                                 |
| 2                 | 0.00                                  | 0.00                             | 0.00                               | 0.00                                   | 0.00                                 |

**Table 28: Continued**

|    |      |      |      |       |      |
|----|------|------|------|-------|------|
| 3  | 0.00 | 2.33 | 1.00 | 2.33  | 1.67 |
| 4  | 1.50 | 4.75 | 3.75 | 7.00  | 4.00 |
| 5  | 3.80 | 5.60 | 5.40 | 11.40 | 4.40 |
| 6  | 4.00 | 4.83 | 5.67 | 10.17 | 6.00 |
| 7  | 3.57 | 4.29 | 5.14 | 8.71  | 6.29 |
| 8  | 3.25 | 4.25 | 5.00 | 8.13  | 5.75 |
| 9  | 2.89 | 4.00 | 5.11 | 7.22  | 5.44 |
| 10 | 2.60 | 4.00 | 5.10 | 6.50  | 4.90 |
| 11 | 2.36 | 3.64 | 4.64 | 5.91  | 4.45 |
| 12 | 2.25 | 3.33 | 4.25 | 5.42  | 4.08 |
| 13 | 2.08 | 3.08 | 3.92 | 5.00  | 3.77 |
| 14 | 1.93 | 2.86 | 3.64 | 4.64  | 3.50 |
| 15 | 1.80 | 2.67 | 3.40 | 4.33  | 3.27 |
| 16 | 1.69 | 2.50 | 3.19 | 4.06  | 3.06 |
| 17 | 1.59 | 2.35 | 3.00 | 3.82  | 2.88 |
| 18 | 1.50 | 2.22 | 2.83 | 3.61  | 2.72 |
| 19 | 1.42 | 2.11 | 2.68 | 3.42  | 2.58 |
| 20 | 1.35 | 2.00 | 2.55 | 3.25  | 2.45 |
| 21 | 1.29 | 1.90 | 2.43 | 3.10  | 2.33 |
| 22 | 1.23 | 1.82 | 2.32 | 2.95  | 2.23 |
| 23 | 1.17 | 1.74 | 2.22 | 2.83  | 2.13 |
| 24 | 1.13 | 1.67 | 2.13 | 2.71  | 2.04 |
| 25 | 1.08 | 1.60 | 2.04 | 2.60  | 1.96 |
| 26 | 1.04 | 1.54 | 1.96 | 2.50  | 1.88 |

**Table 28:** Continued

|    |      |      |      |      |      |
|----|------|------|------|------|------|
| 27 | 1.00 | 1.48 | 1.89 | 2.41 | 1.81 |
| 28 | 0.96 | 1.43 | 1.82 | 2.32 | 1.75 |
| 29 | 0.93 | 1.38 | 1.76 | 2.24 | 1.69 |
| 30 | 0.90 | 1.33 | 1.70 | 2.17 | 1.63 |
| 31 | 0.87 | 1.29 | 1.65 | 2.10 | 1.58 |

**Table 29:** The biodegradability of the different types of cellulose-based hydrogels.

| Day                                          | Weight loss (%) |       |       |       |       |       |
|----------------------------------------------|-----------------|-------|-------|-------|-------|-------|
|                                              | 1               | 14    | 21    | 28    | 35    | 60    |
| Cellulose-1.75g<br>CMC-based hydrogel        | 0               | 62.58 | 88.74 | 94.71 | 95.7  | 96.01 |
| Cellulose-2.0g AA-<br>based hydrogel         | 0               | 62.87 | 92.04 | 96.5  | 96.73 | 97.35 |
| Cellulose-1.0g<br>glycerol-based<br>hydrogel | 0               | 18.99 | 69.48 | 75.49 | 77.22 | 87.12 |
| Cellulose-0.5g<br>PEG600-based<br>hydrogel   | 0               | 6.14  | 53.23 | 71.77 | 84.82 | 87.92 |