PREPARATION AND CHARACTERIZATION OF CELLULOSE HYDROGEL DERIVED FROM PINEAPPLE LEAF FIBER

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DEDICATION

This thesis is dedicated to my parents who taught me that the best kind of knowledge to have is that which is learned for its own sake. To my families, supervisors and friends thank you for all your support along the way.

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ABSTRACT

In this study, hydrogel was prepared by using cellulose extracted from pineapple leaf fiber (PALF). PALF has higher cellulose content and better tensile properties compared to other cellulosic agro wastes such as sugarcane bagasse. However, up to date most of the researches are only focusing on the effect of alkali treatment on the properties of fiber regenerated from PALF and none has reported on the preparation of hydrogel from PALF. Besides, studies also showed that the cellulose concentration affected the properties of hydrogel. In this study, the effects of alkaline treatment on the lignin removal to obtain the PALF cellulose and the properties of PALF cellulose hydrogel were investigated. Then, the effect of cellulose concentration using the selected cellulose obtained from the treatment on the properties of the resultant PALF cellulose hydrogel were investigated. For the alkali treatment, PALF has been treated with different concentrations of sodium hydroxide (NaOH) (2-10 wt%) at two different temperatures (room temperature and 80 °C). Then, PALF was dissolved in *N,N'*-dimethyl acetamide and lithium chloride solvent and underwent a phase inversion to change from liquid into a solid cellulose hydrogel. Later, after the optimum concentration and temperature for alkaline treatment were obtained, the cellulose hydrogel was prepared with different cellulose concentrations (0.5–2.0 wt%). From the Fourier transform infrared results, lignin was removed starting at the concentrations of 6 wt% NaOH at room temperature and 4 wt% NaOH at 80 °C, respectively. The thermal stability of fiber was increased at higher NaOH concentration and the crystallinity of cellulose was also increased (71-79%) after the treatment. Higher NaOH concentration and treatment temperature have increased the swelling equilibrium of the hydrogel but adversely affected the gel fraction. Besides, the ultraviolet-visible spectra showed that the highest transmittance (87.8 %) and the lowest intensity of absorbance (at 280 nm corresponding to lignin) were obtained by the cellulose hydrogel prepared with cellulose treated at 8 wt% NaOH and 80 °C. Later, the designated cellulose was chosen to prepare the hydrogel at different cellulose concentrations. It was found that, the swelling and the transparency of hydrogel decreased with increasing of cellulose concentration. On the other hand, the viscosity of the solution, the gel fraction and the tensile strength of the hydrogel increased as the cellulose concentration increased. The highest tensile strength (1 MPa) was obtained by the hydrogel at 2.0 wt% cellulose concentration. The increase in these properties were most probably due to the increased of entanglement of the cellulosic chains due to the formation of the hydrogen bonding at higher cellulose concentration. From the rheological measurement, the elastic modulus (G′) of all the hydrogels were higher than the loss modulus (G″) regardless of the cellulose concentration and both parameters were independent to the measured frequency. This showed that the PALF hydrogels possessed the ideal rubber characteristics. The mesh sizes of the hydrogel decreased with increasing cellulose concentrations. Clearly, this revealed that the condition of the alkaline treatment affected the cellulose extraction and the hydrogel properties. However, the cellulose concentration has greater effect towards the physical and tensile properties of the hydrogel. Ultimately, cellulose hydrogels obtained from this study have promising potential to be used as biomaterials in many applications.

ABSTRAK

Dalam kajian ini, hidrogel telah disediakan menggunakan selulosa yang di ekstrak dari serat daun nanas (PALF). PALF mempunyai kandungan selulosa yang lebih tinggi dan kekuatan tegangan yang lebih baik berbanding dengan sisa agro selulosa lain seperti tebu. Walau bagaimanapun, kajian terkini mengenai PALF hanya berfokus kepada kesan rawatan alkali terhadap sifat serat yang diperoleh dari PALF dan tiada kajian terhadap penyediaan hidrogel daripada PALF. Selain itu, kajian juga menunjukkan kepekatan selulosa boleh mempengaruhi sifat hidrogel yang terhasil. Dalam kajian ini, kesan rawatan alkali terhadap penyingkiran lignin untuk memperoleh PALF selulosa dan terhadap sifat PALF selulosa hidrogel telah disiasat. Kemudian, kesan kepekatan selulosa menggunakan selulosa yang terpilih selepas rawatan terhadap sifat PALF selulosa hidrogel yang terhasil disiasat. Untuk rawatan alkali, PALF telah dirawat dengan kepekatan natrium hidroksida (NaOH) yang berbeza (2-10 wt%) pada dua suhu yang berbeza (suhu bilik dan 80°C). Kemudian, PALF telah dilarutkan dalam pelarut *N*, *N′*-dimetil asetamida dan litium klorida dan mengalami penyongsangan fasa untuk berubah dari cecair kepada pepejal hidrogel selulosa. Kemudian, setelah kepekatan dan suhu optimum rawatan alkali diperoleh, hidrogel selulosa telah disediakan dengan menggunakan kepekatan selulosa yang berbeza (0.5-2.0 wt%). Dari hasil inframerah transformasi Fourier, lignin akan mula disingkirkan masing-masing pada kepekatan 6 wt% NaOH pada suhu bilik dan 4 wt% NaOH pada 80 °C. Kestabilan terma serat meningkat apabila dirawat dengan kepekatan NaOH yang lebih tinggi dan penghabluran selulosa juga meningkat (71-79%) setelah rawatan. Kepekatan NaOH dan suhu rawatan yang lebih tinggi telah meningkatkan keseimbangan pembengkakan hidrogel yang dihasilkan namun memberi kesan yang sebaliknya terhadap pecahan gel. Selain itu, spektra ultralembayung-nampak menunjukkan transmitans tertinggi (87.8%) dan intensiti keserapan terendah (pada 280 nm sepadan dengan lignin) dihasilkan oleh hidrogel selulosa yang disediakan pada 8 wt% NaOH pada 80°C. Selulosa yang terpilih ini, kemudian digunakan untuk penyediaan hidrogel pada kepekatan selulosa yang berbeza. Keputusan menunjukkan bahawa, pembengkakan dan ketelusan hidrogel telah menurun dengan peningkatan kepekatan selulosa. Sebaliknya, kelikatan larutan, pecahan gel dan kekuatan tegangan hidrogel meningkat apabila kepekatan selulosa meningkat. Kekuatan tegangan yang tertinggi (1 MPa) telah diperoleh melalui hidrogel dengan 2.0 wt% kepekatan selulosa. Peningkatan sifat-sifat ini adalah disebabkan meningkatnya penggabungan rantaian selulosa disebabkan pembentukan ikatan hidrogen pada kepekatan selulosa yang tinggi. Melalui pencirian pengukuran reologi, modulus elastik (G′) semua hidrogel lebih tinggi daripada modulus kehilangan (G″) tanpa mengira kepekatan selulosa dan kedua-dua parameter ini tidak bergantung kepada ukuran frekuensi yang digunakan. Hal ini membuktikan bahawa, hidrogel PALF mempunyai sifat getah ideal. Saiz jaringan hidrogel didapati menurun dengan peningkatan kepekatan selulosa. Kajian ini jelas menunjukkan bahawa rawatan alkali memberikan kesan kepada pengekstrakan selulosa dan sifat hidrogel. Namun, kesan kepekatan selulosa adalah lebih besar terhadapat sifat fizikal dan kekuatan tegangan hidrogel. Hidrogel selulosa yang diperoleh dalam kajian ini terjamin berpotensi untuk digunakan sebagai biomaterial dalam banyak aplikasi.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURE

LIST OF ABBREVIATIONS

PALF	-	Pineapple leaf fiber
pHEMA	-	polyhydroxyethylmethacrylate
DMAc	-	N,N-dimethyl acetamide
LiCl	-	Lithium chloride
NaOH	-	Sodium hydroxide
PEG	-	Poly ethylene glycol
NaOCl	-	Sodium Hypochlorite
3D	-	Three dimensional
KOH	-	Potassium Hydroxide
$Ca(OH)_2$	-	Calcium Hydroxide
Na_2CO_3	-	Sodium Carbonate
H_2SO_4	-	Sulfuric Acid
NMMO	-	N-methylmorpholine-N-oxide
ILs	-	Ionic Liquids
TBAF	-	Tetrabutylammonium fluoride
DMSO	-	Dimethyl Sulfoxide
NMR	-	Nuclear Magnetic Resonance
FTIR	-	Fourier Transform Infrared
NTP	-	Non-treated PALF
XRD	-	X-Ray Diffraction
TGA	-	Thermal Gravimetric Analysis
CI	-	Crystallinity index
RT	-	Room Temperature
LVR	-	Linear Viscoelastic Region

LIST OF SYMBOL

ºC	-	Degree celcius
H	-	hour
Vol %	-	Volume percent
wt %	-	Weight percent
kN	-	Kilo newton
N	-	Newton
mm^2	-	Square millimeter
mm	-	Millimeter
s	-	second
min	-	minute
G′	-	Storage modulus/elastic modulus
G″	-	Loss modulus/ viscous modulus
MPa	-	Megapascal
nm	-	Nanometer

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Hydrogel can be defined as a material that consists of a three-dimensional network of polymer chains and water that fills the space between the networks (Ahmed, 2015). Hydrogels are made up of hydrophilic polymers and do not dissolve when cross-linked. The ability of the hydrogels to retain a large amount of water or biological fluids make it similar to living tissues (Ullah et al., 2015). Due to this unique characteristic of the hydrogel, many research and exploitation on hydrogel have been done.

In recent years, researchers have contributed much attention to the study of the natural polymers such as chitosan (Ahmadi et al., 2015), gelatin (Bakravi et al., 2018), starch (Ismail et al., 2013) and cellulose (Dutta et al., 2019) as the resources for the hydrogel synthesis. Cellulose is one of the most abundant natural polymers and it has attracted many scientists because of their unique properties such as biodegradability, low toxicity, and biocompatibility. Furthermore, cellulose is available worldwide and it has combination of hydrophilicity with good mechanical properties. (Navarra et al., 2015). Therefore, cellulose can be classified as a special and interesting materials to be used as the resource for the preparation of hydrogel.

There are several methods to extract cellulose from a plant which can be classified as physical, chemical and physicochemical methods. Acid treatment is one of the methods for fibers pre-treatment. Dilute acid treatment is used to break the rigid structure of the lignocellulosic materials while removing the hemicellulose only (Brodeur et al., 2011). Alkaline treatment is notorious for providing the efficient delignification of lignocellulose and is generally more effective on herbaceous crops and agricultural residues (Yamashita et al., 2010). Alkaline treatment eliminates the non-cellulosic content that covers the external surface of the fiber cell wall, thus increase the amount of exposed cellulose on the fiber surface (Zin et al., 2018). Therefore, some researchers have reported on the extraction of cellulose using acid treatment followed by alkaline treatment for more effective extraction of cellulose (Tovar-carillo et al., 2013). In addition, it can degrade the ester bonds and cleavage of glycosidic linkages in the lignocellulosic cell wall matrix, thus modify the lignin structure, reduce the lignin–hemicellulose complex, and leading to cellulose decrystallization (Cheng et al., 2010).

Many researchers have synthesized hydrogels from various cellulose resources such as sugarcane bagasse fiber, bamboo fiber and agave tequilana weber bagasse fiber (Nakasone, 2015; Tovar-Carrillo et al., 2014; Tovar-carrillo et al., 2013). Pineapple leaf fibers (PALF) is another source of cellulose which has not yet been used for the preparation of cellulose hydrogels. The total production of pineapple in Malaysia for 2019 is about 357,805 metric tonnes (Department of Agriculture, 2019). However, there is no appropriate method in handling residue produced from pineapple planting. The abundance of pineapple residues opens many doors for new research to be explored. According to Asim et al. (2015), PALF contains higher cellulose content (67-85%) and extraction of cellulose from PALF can be carried out at relatively mild condition. Therefore, the cellulose in PALF is suitable to be extracted for the usage in the preparation of cellulose hydrogels since it is available in abundance in Malaysia.

Hydrogels are physically cross-linked when there are no cross-linkers during synthesis (Akhtar et al., 2016). After the extraction process, cellulose need to be dissolved in an appropriate solvent before it can be made into hydrogel. In a recent study, *N,N'*-dimethyl acetamide (DMAc) with lithium chloride (LiCl) was used as the solution for cellulose dissolution as it is one of the best solvent used for high-molecular-weight cellulose (Nakasone, 2016; Zhang et al., 2014). Then, phase inversion method is used for the formation of cellulose hydrogel from a cellulose solution. It is a process where, polymer transforms from a liquid phase into the solid phase. In recent years, Tovar-Carillo et al. (2014) had successfully prepared cellulose hydrogel from Agave tequilana Weber bagasse fibers. In their study, besides acid and alkaline treatments, they had to perform bleaching at various sodium hypochlorite concentrations (0-10 vol% NaOCl) as alkali treatment alone was not effective enough to remove the non-cellulosic part in the fibers. In another study, three steps of treatment process were also required to obtain the cellulose from sugarcane bagasse fibers as the raw material for the preparation of cellulose hydrogel (Nakasone, Ikematsu and Kobayashi, 2016). As for their study, bleaching treatment as the third treatment step was conducted at 40 and 50 °C, while for alkaline treatment, they had kept it constant at 10 wt% NaOH, 80 °C and 12 hours treatment.

In this study, cellulose hydrogel from PALF was prepared via the phase inversion method. Cellulose was extracted from PALF using alkaline treatment method at various NaOH concentrations. Since there is no study has been done on the cellulose from PALF, the best treatment parameter was identified to get pure cellulose for the hydrogel preparation. This study emphasis on the effect of the alkaline treatment on the lignin removal from PALF and resultant hydrogel properties produced from PALF cellulose. The hydrogel produced was characterized based on equilibrium swelling ratio, gel fraction and transparency. The cellulose concentration also can affect the properties of the cellulose hydrogel. Therefore, the effect of cellulose hydrogel concentrations towards the cellulose hydrogel properties also was analysed so that hydrogel with the best properties in terms of gel fraction, swelling, transparency, tensile properties, viscosity and rheological properties can be produced and identified.

1.2 Problem Statement

Cellulose is the most abundant resources on earth among all biopolymers. The unique properties of cellulose make it suitable to be utilized as raw material for hydrogel preparation. The properties of biopolymer-based hydrogel such as biocompatible and biodegradable cannot be found in the synthetic based hydrogel. Thus, it has acquired increasing attentions from researchers. Biopolymer-based hydrogel components are mostly non-toxic and bio-inert; these properties contribute to the overall biocompatibility of hydrogel matrices with surrounding tissue (Reddy et al., 2021). Nevertheless, very limited research has been performed on using cellulose fiber for the preparation of hydrogel film.

Currently, the utilization of cellulosic agro-waste as the raw materials for hydrogel preparation only focused on several fiber resources such as from Agave tequilana Weber bagasse, bamboo and sugarcane bagasse. However, the cellulose content for all those fibers are low; i.e., 43% (Li et al., 2012), 33-51% (Wahab et al., 2013) and 40-50% (Nakasone et al., 2016) for Agave tequilana Weber bagasse fibers, bamboo fibers and sugarcane bagasse fibers, respectively. PALF contains higher cellulose content which is 67-85% (Asim et al., 2015) and no study has been reported yet on the preparation of hydrogel from PALF cellulose. The preparation of hydrogel from PALF cellulose will be more efficient as high cellulose content of PALF provides more cellulose compared to fibers with lower cellulose content. Besides, the tensile strength (350-700 MPa) (Malou et al., 2017) of PALF was reported to be higher than the tensile strength of sugarcane fibers (249-468 MPa) (Fiore et al., 2015), bamboo fibers (100-350 MPa) (Sugiman et al., 2019) and agave bagasse (41-58 MPa) (Kestur G. et al., 2013).

The temperature and concentration of the treatment of sugarcane and agave bagasse reported to affect the properties of cellulose hydrogel (Nakasone et al., 2016; Tovar-Carillo et.al, 2014). In those studies, the increase in temperature during treatment has decreased the molecular weight of the cellulose and mechanical properties of the resultant hydrogels but has increased the crystallinity and the lignin removal of the cellulose fibers. On the other hand, the mechanical properties of the resultant hydrogels increased with increasing NaOCl concentration. Over the decades, there are many studies have been reported on the effect of the alkaline treatment onto the properties of fiber regenerated from PALF (Asim et al., 2018; Fareez et al., 2018; Malou et al., 2017). Particularly, about 2 to 6 wt% of NaOH concentration was used for the alkaline treatment of PALF. Besides, there are also studies reported on the effect of the alkaline treatment onto the PALF to be used as reinforced in composites (Motaleb, 2018; Zin et al., 2018; Siregar et al., 2010). The NaOH concentration used was in the range of 2-8% and the best treatment parameters reported depends on the properties of the composites obtained. However, up to date, there are still no studies reported on the effects of alkali treatment on the properties of cellulose based hydrogel regenerated from PALF. Thus, it is compulsory to evaluate the best alkaline treatment parameters that can result in the best properties of cellulose hydrogel obtained from PALF. Poor treatment conditions can result in un-pure cellulose obtained as lignin is still presence in the fibers. The presence of lignin in the fibers also will result in poor dissolution of cellulose. On the other hand, too harsh treatment can result in degradation of cellulose thus will affect the properties of the hydrogel obtained (Nakasone and Kobayashi, 2015).

According to Navarra et al., (2015) the swelling capabilities and mechanical properties of hydrogel can be affected by the cellulose concentration. Ishii et al. (2006) reported that, cellulose concentration effects the dissolution state of cellulose in LiCl/DMAc. Besides, Mendoza et al. (2018), Qiao et al. (2016) and Wu et al. (2014) reported on the effects of cellulose concentration on the rheological properties of hydrogel. Current study only reported on the effect of cellulose concentration obtained from commercial cellulose. Besides, different cellulose resources might result in difference of hydrogel properties as the properties of cellulose itself is varied depending on its resources. Therefore, it is essential to evaluate the effect of cellulose concentration from PALF onto the properties of cellulose hydrogel generated since this is the first research reported on the preparation of cellulose hydrogel from PALF.

1.3 Objective of Study

The aim of this research is to synthesize cellulose hydrogel from renewable bio-waste of PALF. The aim can only be achieved by following these few objectives which are:

1) To investigate the effect of alkali treatment on the removal of lignin from PALF based on FTIR, TGA and XRD and resultant properties of cellulose hydrogel in terms of gel fraction, swelling, transparency and lignin content.

2) To investigate the effect of different cellulose concentrations on the viscosity, swelling, gel fraction, transparency, mesh size, tensile and rheological properties of cellulose pre-gel solutions and cellulose hydrogels.

1.4 Scope of Study

In this study, cellulose hydrogel was prepared from PALF. The PALF has undergo treatment process which consists of acid treatment and alkali treatment. For acid treatment, PALF was treated with 4 vol% of 300 ml sulphuric acid solution at 80 °C for 1.5 hrs. Next, PALF was treated with different concentration of sodium hydroxide solution (2-10 wt%) for 6 hrs at room temperature (RT) and 80 °C. The obtained cellulose after each treatment was further characterized with Fourier Transform Infrared spectroscopy (FTIR), Thermogravimetric analysis (TGA) and X-ray Diffraction (XRD).

Then, treated PALF (6-10 wt% NaOH) was dissolved in DMAc/LiCl solvent under stirring condition at RT until all the cellulose had dissolved. The last part was the preparation of cellulose hydrogel by phase inversion method using cellulose solution. For phase inversion method, cellulose solution was poured into a petri dish and was left in a container containing ethanol. The cellulose solution was solidified and become hydrogel.

The optimized treatment conditions that was used to investigate the effect of cellulose concentration on the hydrogel properties was determined based on the gel fraction, swelling, transparency and lignin content in the hydrogel. Then, cellulose hydrogels were prepared with different cellulose concentrations (0.5-2.0 wt%).

The scope of the work also included characterization of physical and functional properties of cellulose solution and cellulose hydrogels. The cellulose solutions were prepared with different cellulose concentrations (0.5-2.0 wt%). Then, cellulose hydrogels were obtained by phase inversion. Viscosity test was done for the characterization of the cellulose solution. Cellulose hydrogel was characterized by using gel fraction, equilibrium swelling ratio and transparency. Mechanical properties of cellulose hydrogels were studied by using texture analyser and oscillatory rheometer. Texture analyser was used to characterize the tensile properties of cellulose hydrogel. Rheology test was done to determine the hydrogel viscoelasticity and microstructure. Mesh sizes of the hydrogels were calculated from rheology measurement.

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