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REVIEW: SYNTHESIS OF STARCH BASED AEROGELS VIA AMBIENT PRESSURE DRYING

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SYNTHESIS OF STARCH BASED AEROGELS

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ABSTRACT

Porous material made from biomaterial like aerogels has been getting numerous attentions in the research arena lately. Aerogel is classified as a material of light weight and open pores accompanied with high specific surface area. Starch based aerogels are a class of advanced biomaterial made in the same way as bio-aerogels based on other polysaccharides via starch gelatinization, retrogradation, organic solvent exchange, and process of drying. Essentially, there are three most common drying mechanism of aerogel available which are supercritical carbon dioxide drying, freeze drying and ambient pressure drying. This paper reviews the potential outcome of synthesis of starch aerogel via ambient pressure drying based on similar studies conducted previously. This review summarizes the potential functional properties of starch-based aerogel based on similar polysaccharide aerogels like cellulose and chitosan aerogels which were synthesized via ambient pressure drying. Comparative analysis between similar porous materials was executed to reveal any advantages of the former. Thus, through this detailed review on previous polysaccharide aerogels despite having limited studies on starch-based aerogel via ambient pressure drying, the expected result of starch-based aerogel synthesized via this route can be forecasted. This paper analyses essential properties of starch-based aerogel reflected through this processing route such as morphology and microstructure, density and shrinkage, mechanical properties and thermal conductivity of the material. There are numerous ways to achieve variety of morphology and properties by varying the preparation condition to yield varying results tunable to the desired application. Therefore, with chemical and physical modification open up the possibilities of wide range of aerogels' properties and applications.

ABSTRAK

Bahan berpori yang terbuat dari bahan bio seperti *aerogel* telah mendapat perhatian di arena penyelidikan akhir-akhir ini. *Aerogel* diklasifikasikan sebagai bahan ringan dan mempunyai liang terbuka disertai dengan jumlah luas permukaan yang tinggi. *Aerogel* berasaskan kanji adalah dalam kategori bahan bio yang canggih yang dibuat dengan cara yang sama seperti *bio-aerogel* berdasarkan polisakarida lain melalui gelatinisasi kanji, retrogradasi, pertukaran pelarut organik, dan proses pengeringan. Pada asasnya, terdapat tiga mekanisma pengeringan *aerogel* seperti pengeringan karbon dioksida superkritik, pengeringan beku dan pengeringan tekanan ambien. Makalah ini mengkaji kemungkinan hasil sintesis *aerogel* berasaskan kanji melalui pengeringan tekanan ambien berdasarkan kajian serupa yang dilakukan sebelumnya. Ulasan ini meringkaskan potensi keupayaan berfungsi *aerogel* berasaskan kanji berdasarkan *aerogel* polisakarida yang serupa seperti *aerogel* selulosa dan citosan yang disintesis melalui pengeringan tekanan ambien. Analisis perbandingan antara bahan berliang yang serupa dilakukan untuk melihat beberapa kelebihan yang penting pada struktur ini. Oleh itu, tinjauan terperinci berdasarkan kajian yang terdahulu terhadap penghasilan aerogel berasaskan kanji melalui proses pengeringan boleh membantu kita meramal struktur yang terhasil. Makalah ini menganalisis beberapa sifat penting *aerogel* berasaskan kanji seperti morfologi dan struktur mikro, ketumpatan dan pengecutan, sifat mekanik dan kekonduksian haba bahan. Terdapat banyak teknik untuk menghasilkan pelbagai morfologi dan sifat seperti dengan mempelbagaikan keadaan persekitaran sebelum pemprosesan hingga sehingga proses selesai. Faktor tersebut aan menghasilkan sifat produk yang berbeza. Pengubahsuaian kimia dan fizikal mampu menghasilkan aerogel yang mempunyai pelbagai sifat dan seterusnya menjurus kepada kepelbagaian aplikasi *aerogel.*

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LIST OF SYMBOLS

W	Watts
K	Thermal Conductivity
°C	Celcius
Q	Heat Flow
ρ	Density
m	Mass
V	Volume

LIST OF ABBREVIATIONS

APD	Ambient Pressure Drying
SCD	Supercritical Drying
FD	Freeze Drying
ASTM	American Standard Test Methods
SEM	Scanning Electron Microscopy
BET	Brunauer–Emmet–Teller
BJH	Barret–Joyner–Halenda
CNF	Cellulose Nanofibrils
PMSQ	Polymethylsilsesquioxane
TMCS	Trimethylchlorosilane

CHAPTER 1

INTRODUCTION

1.1 Background

The popularity of aerogels is increasing and continue growing over the years due to its amazing properties and various applications ranging from home to space applications. Today, there are various types of aerogels such as silica aerogels, cellulose aerogels, oxide aerogels and alumina and other oxide aerogels. According to Koh et al. (2018), aerogels are lightweight materials with low densities $\left(0.003 - 0.5 \frac{g}{cm^3}\right)$, low thermal conductivities (0.005 – 0.045 W/m·K) and high porosity. Due to this mechanical properties and its versatility, aerogels are widely used for various technical applications laser experiments, ultrasonic and gas sensors, nuclear particle detection, thermal insulation, waste management gas absorption, radioactive waste confinement, optics and light-guides, electronic devices, energy storage, imaging devices, catalysts and X-ray laser research (Hrubesh, 1998)

Starch is a promising source of aerogel formation at low cost, sustainable, abundant and bio-based (Ubeyitogullari & Ciftci, 2016). According to Maningat et al. (2009), among various starch sources, wheat starch is the world's third most produced type of starch which has the potential to form three dimensional polymeric network structures of starch hydrogels whilst playing a crucial role in food. Maningat et al. (2009) further explained that wheat starch has a composition of 25% amylose and 75% amylopectin.

To date, wheat starch has restricted usage and application which is mainly for the production flour (Ubeyitogullari & Ciftci, 2016). In the recent years, various materials have been used to develop aerogels with enhance properties which includes corn starch (García-González et al., 2015; Kenar et al., 2014) and hybrid (composed of inorganic and organic combination) materials such as silica-cellulose (Demilecamps et al., 2015).

1.2 Problem Statement

Generally, aerogels exhibit countless of outstanding properties which often leads to their potential application in various fields. In a manufacturing study conducted by Dorcheh and Abbasi (2008), the production of silica aerogel utilizes toxic chemicals, high consumption of solvent in their diffusion-controlled processes and high pressure vessels running for an extended period of time depending on the drying process.

According to Long et al. (2018), the precursors of synthetic polymer-based aerogels are non-degradable and toxic. In another study conducted by Filipe (2015) stated that the production of inorganic aerogels (from extraction of raw materials to production phase) in comparison with monolithic hybrid aerogels posed the highest environmental impact in terms of abiotic depletion, photochemical oxidation and global warming photochemical oxidation indicators.

Therefore, it is safe to deduce that a more environmental-friendly and greener approach to synthesize aerogels derived from organic source is highly demanded to replace the existing conventional methods.

1.3 Objectives

The objectives of this study are stated as follows;

i. To review the synthesis starch-based aerogels via ambient pressure drying (APD),
ii. To review the morphology and microstructures of starch-based aerogels of different starch concentrations in the dispersion, the gelatinization temperature, the incorporation of non-starch material (as cross-linking agents), the incorporation of surface modification agents and the drying temperature, and
iii. To review the physical properties of starch-based aerogels of different starch concentrations in the dispersion, the gelatinization temperature, the incorporation of non-starch material (as cross-linking agents), the incorporation of surface modification agents and the drying temperature.

1.4 Research Scope

Wheat starch is used as the source of starch to synthesize the starch-based aerogels. Ambient pressure drying (APD) is used as the mechanism to dry the aerogels. A scanning electron microscope is used to carry out the micrography analysis.

1.5 Thesis Outline

This thesis is divided into five chapters which comprise of the overall details of the review paper. In Chapter 1, the introduction of the review is explained by clarifying the background, problem statements, objectives and the scopes of the proposed methodology.

Moreover, Chapter 2 elaborates on the literature review of the paper. In this chapter, concepts and findings of a particular field related to the review are clarifies. The general concept of aerogels is explained with an overview of aerogels and types of aerogels. The synthesis and method to produce aerogels based on previous studies are also included in this review paper. The available methods to produce aerogels are supplemented with the properties of aerogels and the wide range of application.

Chapter 3 focuses on the proposed methodology of the project. The methodology is briefly illustrated in a flowchart supplemented in this chapter to explain on the planning and organizing process of executing the project. This chapter is also accompanied by the characterization methods of samples to obtain the results. The calculation for the concerned parameter is also included as to cover the detailed explanation of the matter.

Moreover, Chapter 4 showcases the expected result and discussion of starch-based aerogel. A collective result from previous studies of similar scopes and spectrum is used to achieve consistency in result expectation and forecast. The highlighted parameters include the morphology and microstructure of aerogels, density and shrinkage, mechanical properties and finally thermal conductivity of aerogels.

Lastly, Chapter 5 provides the overall conclusion and future research direction of this study. This section emphasizes on the expected outcomes and provide structured research direction for further works in the recurring field of study.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter reviews the types of aerogels exist in the commercial market today and its various commercial and technical applications. This chapter will also review the synthesis of aerogels, mechanical properties of aerogels and biodegradable aerogels.

2.2 Types of Aerogels and Application

There are three types of aerogels commonly found which includes silica, carbon and metal oxides. In practical applications, the most common among the three types of aerogels is the silica aerogel. This is because silica aerogels exhibit amazing properties which opens to windows of many potential applications.

The pilot production of silica aerogels started in 1930s, but saw little development for several decades (Soleimani et al., 2008). Silica aerogels has found its way in many commercial applications. One of the most interesting commercial application of silica aerogels include molds for casting aluminum metal, wastewater treatment, heat storage device for automobiles (Ahmed & Attia, 1995; Alkemper et al., 1995; Herrmann et al., 1995). It was also reported that silica aerogel are involved in the application of transparent window components, building walls transparent heat insulator or vacuum insulation (Herrmann et al., 1995).

Another type of aerogels is carbon aerogels. Carbon aerogels are considered as a very promising type of materials for various energy related applications due to their high mass-specific surface area and electrical conductivity, environment benignity and inertness of chemical (Biener et al., 2011). It has been reported that a recent developed carbon aerogels with a new class of ultra-high surface area are used as hydrogen physisorbents (Kabbour et al., 2006). A process called thermal activation is used to prepare this material at which involving the controlled incineration of carbon from aerogel structure in an oxidizing atmosphere like carbon dioxide (Biener et al., 2011). Biener et al. (2011) also reported that through the carbon removal, new micropores are created within the microstructures and thus resulted in the increment of overall surface area. Another application of carbon aerogels is in the field of green technologies as electrical double-layer capacitor, EDLC (Pekala et al., 1998). It was reported that in the device, the storage of the charge was in the form of ions amassed on the surface of the material and subsequently resulted in the formation of a midway between electrostatic capacitors and batteries (Conway, 1991). Beyond its application as energy storage, carbon aerogels are also used to boost the performance of other solid-state hydrogen storage materials especially multifaceted hybrids (Biener et al., 2011). Biener et al. (2011) also reported that carbon aerogels is one of the most potential candidates for this application due to their tunable porosities, outsized pore volumes and its ability to adjust the surface characteristics of the carbon framework.

Metal oxide aerogels can be classified as inorganic cousins of silica aerogels which exhibits their own unique properties. This type of aerogels are very interesting nonporous materials used the application of thermal insulations, catalysts, sensors and etc. Examples of metal oxide aerogels includes Iron Oxide, Zirconia, Titania, Alumina, Chromia and Vandia. Some applications of various metal oxide aerogels includes filtration, sorption media or photocatalysts, in medical or electrochemical and optical application (Feinle & Hüsing, 2015).

2.3 Hydrophobic and Hydrophilic Aerogels

In the present market today, there are two existing types of aerogels which can be categorized as hydrophobic and hydrophilic aerogels. The following text will be only for silica hydrophobic and hydrophilic aerogels. Figure 2.1 and Figure 2.2 show the schematic diagram of hydrophilic silica aerogels and hydrophobic silica aerogels, respectively.

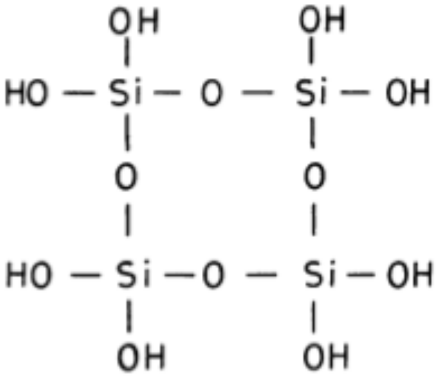

Figure 2.1 The schematic diagram for hydrophilic silica aerogels

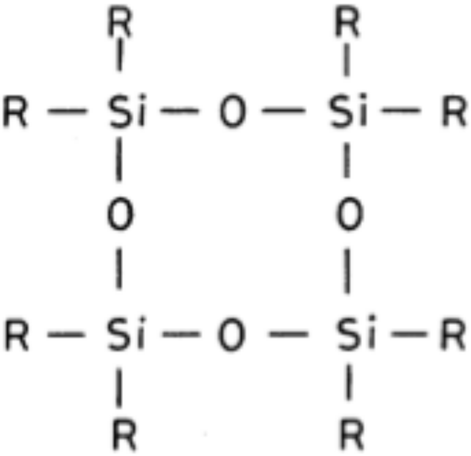

Figure 2.2 The schematic diagram for hydrophobic silica aerogels

The aerogel’s hydrophobicity originates from the chemical structure of the aerogel. the main source of hydrophilicity of the aerogel is the Si-OH structure as this particular chemical structure promotes the adsorption water (Wagh & Ingale, 2002). Wagh and Ingale (2002) also further clarified that when a hydrolytically stable Si-R (R=CH3) replaces the Si-OH structure, the aerogel will exhibit hydrophobicity as it prevents the water adsorption and will not be susceptive to water.

There are two methods to achieve hydrophobic aerogels namely (a) surface chemical modification of aerogels by gaseous reagents and (b) surface modification of the colloidal particles by the incorporation of certain hydrophobic reagents in the alcosol.

2.4 Synthesis of Aerogels

2.4.1 Sol Gel Process

There are two critical steps involved in preparation of aerogels. The first step is called a sol-gel process to form the gel followed by the second step which is the drying process to obtain the aerogel (Bangi et al., 2018). Bangi et al. (2018) also stated the fundamental step to synthesizing the aerogels is the sol-gel process which takes place via hydrolysis and polycondensation of precursor solutions to form gels. ‘The process of dispersion of nanoparticles in the liquid phase is called sol whilst the result of this process which leads to the aggregation to form a continuous three dimensional network is called gel (da Silva et al., 1992). The synthesis of aerogel through the sol-gel method can be categorized into a series of steps and depicted as in Figure 2.3 (Błaszczyński et al., 2013). The steps to synthesizing aerogels is shown is Figure 2.3.

Step 1: Formation of alkoxide or solvated metal precursor (sol).
Step 2: Polycondensation reactions to result in gelation (rapid viscosity increments of the solution) from an oxide- or alcohol bridged network (gel).
Step 3: Gelatinization; polycondensation reactions continue until the gel forms a solid mass, followed by contraction of gel network and removal of solvent from gel pores.
Step 4: This step involves shaping of the gel accompanied by the drying process. The manner solvent removal process dictates the porosity of the end material.

There are three known methods of drying aerogels which includes super critical drying (SCD), ambient pressure drying (APD) and freeze drying (FD). According to Bangi et al. (2018), SCD is a drying method which utilizes the drying of present liquid in the pores of the gel above the critical pressure and temperature while APD is a drying process which only uses the ambient pressure and temperature above 150 °C. Bangi et al. (2018) further elaborated that FD is a drying process of the frozen gel in a vacuum.

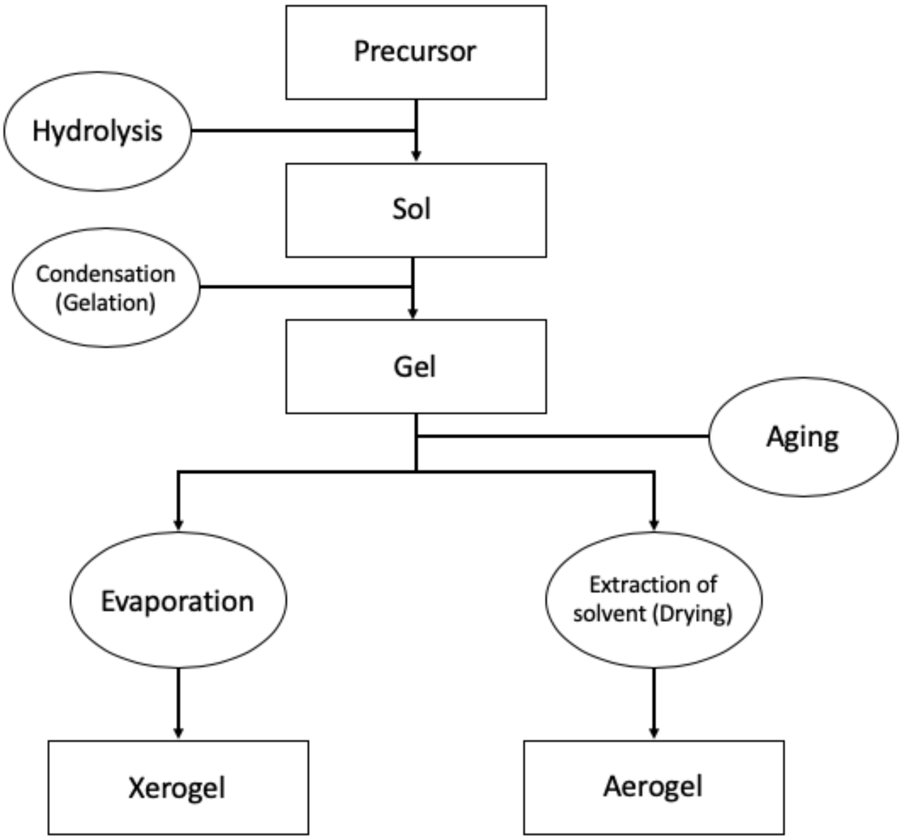

Figure 2.3 Steps to Synthesizing Aerogels (Błaszczyński et al., 2013)

SCD is a conventional way of aerogel drying process. It was first performed in 1931 by Kistler at Stanford University using sodium silicate (Na_2SiO_3) as a starting material (Kistler, 1931). This conventional drying method is then often used. This drying method is also frequently used in the process of drying the three dimensional scaffolds for the maintenance of macro- and nano- hydrogel structure (García-González et al., 2012). García-González et al. (2012) inferred that this approach does not lead to surface tension and vapor-liquid transition. This is due to the ability of this process to prevent the shrinkage of the porous structure (Yan et al., 2019). Yan et al. (2019), also further elaborate this process often comes with a high production cost which includes high equipment requirement, high energy usage and heavy operating hazard. Super critical drying involves a systematic solvent exchange to any solvent that are miscible with carbon dioxide such as acetone and ethanol (Darpentigny et al., 2019). Darpentigny et al. (2019) further elaborated that the process of solvent exchange with liquid carbon dioxide usually takes place at low temperatures and high-pressure prior achieving the supercritical state. Mild supercritical parameters of temperatures

APD is another method of aerogel drying process. This method is capable for large-scale application due to its advantages of simple operation and high safety (Rao et al., 2007). However, the conventional process of APD is tedious as it involves solvent replacement and hydrophobic modification and consequently leading to the higher cost and environmental pollution due to the large usage of organic solvents and modifiers (Yan et al., 2019). Yan et al. (2019) also mentioned the importance to design greener method of APD in order to achieve good formability of silica aerogels via APD. APD has a key advantage when it is compared to supercritical drying process as it is able to remove pore liquids from wet gels effectively without causing any shrinkage and creation of cracks which commonly due to capillary pressure and the condensation of surface silanol groups (Si-OH) (Hüsing & Schubert, 1998). There are two main approaches to prepare aerogels; first, the monolithic aerogels should be aged in a silane precursor to intensify the mechanical strength of aerogels in order to resist the capillary forces (Davis et al., 1992; Haereid et al., 1994). Secondly, the solvent exchange process should be followed by covering the surface of silanol groups by alkyl groups in order for the gel to experience "spring-back" effect after shrinkage caused by the repulsion between the alkyl groups (Patent No. US 5565142, 1996; Patent No. US 5948482A, 1999).

Freeze-drying process is considered to be the best method for solvent removal and obtain dried product at the highest quality (Ratti, 2012). It is further explained that this method is an outstanding route in order to get dried cellulosic cryogels and sponges with exceptional properties like high porosity, low density and high specific surface area (Zaman et al., 2019). This drying process comprises of three steps which includes freezing, primary drying and secondary drying. The freeze step is the most crucial step in the freeze-drying process and it should avoid having interstices with concentrate liquid to get all the drying is carried out by sublimation (Simón-Herrero et al., 2016). Simón-Herrero et al. (2016) also mentioned that the freezing temperatures range from -50 and -80 °C. Additionally, the freezing rate governs the size of ice crystals and thus the porosity of the dry layer which subsequently affect the drying time (Hammami & René, 1997). It was also mentioned that cellular membranes irreversibility and final product texture are affected by the extreme crystal size (Simón-Herrero et al., 2016).

Primary drying stage depends on the solid solvent sublimation. This step is the longest step of freeze-drying process as water present in the material is sublimated up to 95% and the pressure is decreased to a very low levels whilst the temperature in the shelves is increased to facilitate solvent sublimation (Kremer et al., 2009; Simón-Herrero et al., 2016).

The secondary drying step on the other hand is often carried out to remove unfrozen water molecules since most of the ice has been removed in the primary step. In order to facilitate both removal of residual solvent, the temperature of the shelves is raised to integers higher than those required in the primary drying step (Zhai et al., 2005). The vacuum pressure is also reduced in this step (Patapoff & Overcashier, 2002).

2.4.2 Advantages of Sol Gel

Sol-gel method has many advantages which makes it a frequent method to synthesize aerogels. According to Kumar et al. (2015), this method to synthesize nanomaterials can improve the adhesion between the substrate and the top coat. Materials can be easily moulded into complex geometry due to the gel state (Kumar et al., 2015). Another advantage of using this method to synthesize aerogels is homogenization can be achieved in a shorter period of time since the mixing involved low viscosity liquids (Mackenzie, 1988). Mackenzie (1988) further explained that since the homogenization can be achieved at molecular level in such a short interval of time, the reactants are most likely to be equally well mixed when the gel is formed. According to Wenzel (1985), this method enables the controllability of the microstructure of the gels. He further explained that this method enables the dry gels to be made with wide range of densities, surface areas and pore sizes.

2.5 Property of Aerogels

Generally, aerogels are a type of material that possesses numerous outstanding physical characteristics such as low mass densities, continuous porosities and high surface areas. The property of aerogel varies with the types of aerogel. However, the general agreement on the mechanical behavior and property for this literature review will be focusing on silica aerogel.

Aerogels are generally described as brittle materials like glasses and the stress-strain relation evolves like any other elastic material towards fracture (Woignier et al., 2009). Aerogel is a type of material which exhibit a very low density (0.004 – 0.500 g/cc) with an open crossed-linked network having a particle size of less that 10 nm and pore size of less than 50 nm (Aegerter et al., 2011; Mahadik et al., 2016). This material also possesses high porosity of 80 – 99.8%, surface area ($500 - 1200\ m^2/g$), low thermal conductivity (0.017 – 0.05 W/m K) and hydrophobicity (in terms of the contact angle of water with the aerogel surface > 90°) and etc. (Fricke, 1988; Kistler, 1931; Pajonk, 1998; Rao & Kulkarni, 2002). This material also exhibit low water vapor diffusion resistance and good fire ratings which makes it a favourable material for the application of thermal insulation application (Ganobjak et al., 2019). Ganobjak et al. (2019) further inferred that for building thermal insulation, aerogels are able to refurbish small space or where a thick insulation would alter the appearance of the building abruptly. Gonobjak et al. (2019) also demonstrated in their studies that aerogels are prone deterioration with a prolong exposure to temperatures above 70°C and at a high relative humidity of more than 90%.

2.6 Biodegradable Aerogels

Biodegradability has been widely discussed in the recent years. With the emerging of new technologies, there have been numerous researches on the synthesis of aerogels from biodegradable materials. To name a few are cellulose aerogels, alginate-based aerogels, and even aerogels derived from clay and biodegradable polymers.

2.7 Cellulose Aerogels

Cellulose is the most abundant natural polymers on this planet. It possesses distinctive properties from other petroleum-derived polymers like biocompatibility, biodegradability, thermal stability, chemical stability and cost effective (Habibi et al., 2010). In particular, besides its ability to biodegrade, cellulose also possess outstanding mechanical properties such as high porosity, large surface area and low density (Long et al., 2018). Due to these properties, cellulose has become a promising material for future researches. Figure 2.4 summarised the preparation and application of cellulose aerogels.