

UNIVERSITI TEKNOLOGI MARA

**SYNTHESIS AND
CHARACTERIZATION
OF β -CYLODEXTRIN/ALGINATE
AEROGEL
DRIED BY AMBIENT DRYING**

**NURLIYANA BINTI HAMSSIN
2016317755**

B. Eng. (Hons) Chemical

Faculty of Chemical Engineering

January 2020

ABSTRACT

Aerogel is an attractive candidate for the drug delivery system in order to increase dissolved compounds solubility because of its excellent features with large surface area and high pore volume. These properties can only be obtained when the materials are dried by supercritical CO₂ (SCCO₂) to remove liquids present in solid pores. However, this method is so power-intensive and dangerous that it is difficult to practice and sell. Therefore it is very important to synthesize aerogels at a reasonable cost using an ambient pressure drying technique. Even though aerogels have a comparatively elevated compression strength, the structural power of aerogels is not great, because it is very brittle owing to its very small tensile strength and because of the quantity of water in its framework of 99%, they break readily when the stress and heavy load pressure applied to it. The aim of this work were to determine the suitable ratio for the formation of aerogel and to study the morphology and properties of β -cyclodextrin (β -CD)/alginate aerogel. Percentage of shrinkage level for aerogel decrease as the amount of B-cyclodextrin increase. β -CD mixed with alginate to form aerogels enhanced the thermal stability and the rate of decomposition. The application of β -cyclodextrin results in more stable aerogel decomposition about 270 to 290°C for each sample except B1 and B2. The surface area of aerogel increase when β -CD introduced to sodium alginate at first but the surface area suddenly decrease when the amount of β -CD increase which also give a results to low porous structure of aerogel. Lastly, the pore volume of each sample increase as the amount of β -CD increase.

ACKNOWLEDGEMENT

First of all, I thank God for the good health and well-being needed to complete this thesis. Additionally, my sincere thanks to Dr. Ana Najwa, my supervisor who supports me throughout the study. I am also thankful to my co-supervisor at the Chemical Engineering Department, Dr. Suhaiza Hanim. I am extremely grateful and indebted to her for sharing knowledge and for providing to me genuine and meaningful advice and support. I would like to take this opportunity to thank all the laboratory assistant who helped me do the work. Finally, until this research is done, I would like to express gratitude to my parents for their support in terms of morals. I would like to take this opportunity to thank all the laboratory assistant who helped me along this research. Finally, I would like to express gratitude to my parents for their support in terms of morals until I manage to finish this research.

TABLE OF CONTENTS

ABSTRACT	3
ACKNOWLEDGEMENT	i
LIST OF TABLES	iv
LIST OF FIGURES	v
LIST OF SYMBOLS	vi
LIST OF ABBREVIATIONS	vii
CHAPTER ONE: INTRODUCTION	1
1.1 Research background	1
1.2 Problem statement	3
1.3 Objective	4
1.4 Scope of research	4
CHAPTER TWO: LITERATURE REVIEW	5
2.1 Alginate aerogel	5
2.1.1 Alginate	5
2.1.2 Aerogel	6
2.2 Drying method of aerogels	7
2.2.1 Ambient drying	7
2.2.2 Evaporative drying	8
2.2.3 Freeze drying	8
2.2.4 Supercritical drying	8
2.3 Gelation methods	9
2.4 Beta cyclodextrin	9
CHAPTER THREE: METHODOLOGY	14
3.1 Experimental procedure	14
3.1.1 Preparation of β -cyclodextrin/alginate aerogels	14
3.2 The morphology and structure characterization	15
3.2.1 Nitrogen adsorption-desorption analysis	15
3.2.2 Differential Scanning Calorimetry (DSC)	16
3.2.3 Scanning Electron Microscopy (SEM)	16
3.2.4 Thermogravimetric Analysis (TGA)	16
CHAPTER FOUR: RESULTS AND DISCUSSION	17

CHAPTER ONE

INTRODUCTION

1.1 Research background

Throughout recent years, biomaterials have been commonly used for continuous delivery systems to enhance drug safety, effectiveness and consumer enforcement. The efficacy of the drug molecules can be significantly increased by controlling their release and distribution within the body. According to (Gidwani and Vyas 2015) water solubility and dissolution rate are two critical factors that affect the medication process formulation and development and limit their therapeutic applications.

Among biomaterials, aerogels have gained a lot of attention in the field of pharmaceuticals. Due to its low density, high specific surface area, high porosity and other outstanding characteristics, aerogel has a broad variety of applications in catalysts, adsorbents, thermal or noise insulation material, drug carriers and other materials (Barrios, Fox et al. 2019). Aerogel has attracted more and more attention as a drug carrier. However, the two most important factors for drug carriers in the pharmaceutical industry are biocompatibility and biodegradability (Mehling, Smirnova et al. 2009).

In the past research in 1995, drug delivery system was prepared with hydrophilic and hydrophobic silica aerogels. The appropriate aerogels were balanced by drug solution adsorption, then filtered and dried as drug carrier to make loaded aerogels. Although silica aerogels have high particular surfaces and excellent biocompatibility, they are small in pharmaceutical applications due to bad biodegradability. To fix these issues, aerogels based on polysaccharides were first suggested for drug carriers in 1995 (Yun, Luo et al. 2014). Due to its low toxicity, reproduction and good biological performance, aerogel polysaccharides, such as sodium alginate (Veronovski, Knez et al. 2013) and chitosan (Assaad, Wang et al. 2011) are used as a drug carrier.

An alginate is described as a natural polysaccharide biopolymer extracted from brown algae. It includes α -L-guluronic acid and β -D-mannuronic acid (M) residues that are linearly linked to 1,4-glycosidic connection (Paques, van der Linden et al. 2014). It is used as a gelling agent, a stabilizer for colloids and a suspender. In various fields such as biomedical, pharmaceutical, absorption due to biodegradable, biocompatible, non-toxic, low cost and stable, natural biopolymer has been widely used. Alginate aerogel characterized by the combination of unique aerogel characteristics and particularly high surface polysaccharides, open porosity, good compatibility and biodegradable