SYNTHESIS, CHARACTERIZATION AND FABRICATION OF POROUS CARBOXYMETHYL CELLULOSE/STARCH BASED BIO-COMPOSITE MATERIALS

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NUR NADIA BINTI MOHD NASIR

Thesis submitted in fulfillment of the requirements

for the award of the degree of

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SUPERVISOR'S DECLARATION

I hereby declare that I have checked the thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Bachelor of Applied Science (Honour) Material Technology.

Signature

Name of Supervisor : DR FARAH HANANI BINTI ZULKIFLI

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STUDENT'S DECLARATION

I hereby declare that the work in this thesis is my own except for quotation and summaries which have been duty acknowledged. The thesis has not been accepted for any degree and is no concurrently submitted for award for other degree.

Signature

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DEDICATION

Dedicated to the strength of my weakness- my family, lecturers and my friends.

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LIST OF SYMBOLS

%	-	percent
°C	-	degree celcius
g	-	grams
cP	-	centi Poise
µm	-	micro meter
s^{-1}	-	seconds
mg	-	milligram

LIST OF ABBREVIATIONS

M_w	-	molecular weight
TGA	-	Thermogravimetric Analysis
SEM	-	Scanning Electrons Microscopy
ATR-FTIR	-	Attenuated Total Reflectance – Fourier Transformation Infrared
CMC	-	carboxymethyl cellulose
wt%	-	weight percentage

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ABSTRACT

Bio-composite polymer have an important role in many applications. Bio-composite consists of the perfect combination of natural polymers that are biodegradability, least expensive cost and non-toxicity material. The natural polymer combination that have been used in this research are modified cellulose, which is carboxymethyl cellulose (CMC), and starch. In this study, CMC and corn starch will be blend at different weight ratios (100:0, 50:50, 60:40, 30:70, 0:100) by using water as the only solvent. The CMC, corn starch and mixture of CMC/corn starch will be undergo freeze-drying process to produce highly porous biopolymers substrates. Freeze-drying is an easy and convenient technique to produce highly interconnected pores, favourable in tissue engineering. This methods involves the removal of water or any other solvent without damaging of a frozen product by a process called sublimation. All the samples will be physically and thermally characterized by using Rheometer, SEM, ATR-FTIR and TGA. ATR-FTR spectroscopy, has been used to study the functional group of CMC/starch blend polymer solution. In ATR-FTIR study, the changes to the infrared bands, associated with the C-O stretch coupled to the C-C stretch and O-H deformation, were significant and this further supports the strong hydrogen bonding of CMC to the solid surface. SEM have been used to investigate the functional group and porous structure characteristics of the prepared sample. Under SEM testing, the images of each sample shows the different porous structure were obtained by varying the different content of CMC/starch. The measurement of porous scaffolds were assessed with ImageJ, the results showed the increasing of pore size with the addition of CMC content while the granular morphology were showed that the distribution is increased with the amount of corn starch. . Next, TGA and derivatives thermogravimetry (DTG) were carried out for studying the thermal properties of CMC/starch freeze-dried scaffolds. TGA/DTG curve shows that the weight loss of the sample decreased due to the increasing of the temperature of heating. The viscosity of CMC/starch blends polymer solution has been testing by using a rheometer. The sample solution with higher content of CMC showed that the viscosity decrease due to the increasing of shear rate.

ABSTRAK

Bio-komposit polimer mempunyai peranan penting dalam pelbagai aplikasi. Bio-komposit terdiri daripada gabungan sempurna polimer semulajadi yang mudah terurai, kos rendah dan bahan tidak bertoksik. Gabungan polimer semula jadi yang telah digunakan dalam kajian ini diubah suai selulosa, yang carboxymethyl selulosa (CMC), dan kanji. Dalam kajian ini, CMC dan kanji akan menjadi gabungan pada nisbah berat yang berbeza (100: 0, 50:50, 60:40, 30:70, 0: 100) dengan menggunakan air sebagai pelarut. CMC, dan campuran CMC/kanji akan menjalani proses pembekuan dan pengeringan untuk menghasilkan biopolimer sangat berliang substrat. Freeze-pengeringan adalah teknik mudah untuk menghasilkan struktur liang yang sangat saling berkaitan, dan boleh diaplikasikan dalam bidang kejuruteraan tisu. Kaedah ini melibatkan proses pemejalwapan air atau pelarut lain tanpa merosakkan produk yang beku. Semua sampel akan diuji tahap fizikal dan haba dengan menggunakan reometer, SEM, ATR-FTIR dan TGA. ATR-FTR spektroskopi, telah digunakan untuk mengkaji kumpulan berfungsi bagi CMC / kanji penyelesaian gabungan polimer. Dalam kajian ATR-FTIR, berkaitan dengan regangan ikatan C-O ditambah dengan regangan ikatan C-C dan O-H berubah bentuk, adalah penting dan ini menyokong lagi ikatan hidrogen yang kuat CMC ke permukaan pepejal. SEM telah digunakan untuk menyiasat kumpulan dan struktur berliang bagi setiap sampel yang disediakan. Dalam ujian SEM, imej setiap sampel menunjukkan struktur berliang yang berbeza telah diperolehi dengan mengubah kandungan yang berbeza CMC/kanji. Pengukuran struktur berliang dinilai dengan menggunakan applikasi Imej J, keputusan menunjukkan peningkatan saiz liang dengan tambahan kandungan CMC manakala morfologi berbutir telah menunjukkan bahawa pembahagian itu meningkat dengan jumlah kanji. Seterusnya, TGA dan derivatif termogravimetri (DTG) telah dijalankan bagi mengkaji sifat haba CMC / kanji beku-kering. Lengkungan TGA / DTG menunjukkan bahawa kehilangan berat sampel berkurangan disebabkan oleh peningkatan suhu pemanasan. Kelikatan CMC / kanji polimer telah diuji dengan menggunakan reometer.

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

Nowadays, bio-composite polymer gained a lot of attention from researchers all around world. The bio-composite polymer are the natural reinforcement material that have been combined and produced an environmental friendly material. Bio-composite polymer is a material formed by a matrix, generally resin and a reinforcement element, usually made of natural fibres arising mostly from plants or cellulose. The reinforced material are composite material that are biodegradable and non-toxicity not harmful for environment and user.

Bio-composites can be classified into (i) matrix, (ii) natural fibers. Matrix fibers were further classified as biodegrabable (i.e., polyacytide, thermoplastic starch and cellulose) and non-biodegradable (i.e., polypropylene, polyester and polyethylene). In other hand, natural fibers could be utilized from agro wastes such as rise husk. Bagasse, coconut husks and wastes from rubber and palm oil industries that have become one of the most important engineering materials in various application. The unique properties of natural and biodegradable polymers fibers, for example, low economical production, light density with higher specific strength and stiffness safer handling and working conditions has increase their potential to various sectors, including aircraft, automotive, construction, medical and pharmaceutical (Malhotra et al., 2007).

Almost 80% of synthetic polymers available in market are produced from non-renewable petroleum, which was not only higher in price, but also unstable, not sustainable and non-biodegradable. Furthermore, the higher consume of synthetic polymers, specifically fiber composites in different sectors has led to disposal problems. The increasing pollution also caused by the uses of plastics and any others material that are non-biodegradable are very harmful for environment. Hence, many researchers are now interested towards biodegradable material which are more environmentally friendly.

Natural polymers like starch and cellulose have been significantly attract many attention in recent years. Furthermore, Malaysia is one of the main producer and supplier for exportation of the cellulose and starch such as (corn, tapioca, rice starch and bagasse.) in our global marketing. The safe bio-composite polymer can be more various in the application uses in Malaysia such as in water filter, biosensor, medical field, cosmetics and many others.

Malaysia is one of the main producer and supplier for exportation of the cellulose and starch-based plants. Starch (corn, tapioca and rice), and cellulose (carboxymethyl cellulose, polyvinyl alcohol) are regenerated from carbon dioxide (CO_2) and water (H_2O) by process of photosynthesis in green plants. Starch is one of the most abundant natural polysaccharides raw materials.

In order to solve the problems generated by non-biodegradable materials, many effort have been done to obtain an environmental friendly material. Most of researches are focused on substitution of the biodegradable materials with similar properties and low in cost. Bio-composite are composite material that is formed by a matrix and a reinforcement of natural fibres that are usually made from plants or cellulose. Starch and cellulose are one of the abundant natural biodegradable in wide variety of environments. Both of this natural resources are cheap, renewable, non-toxic and biodegradable

In this research, the natural polymer CMC/starch based composites are uses as the bio-composite polymer. The bio-composite polymer are completely biodegradable in soil and water, which is of great advantage from an environmental side and human point of view. (Ghasemlou et al., 2013)

1.2 PROBLEM STATEMENT

The development of biodegradable polymers is a high priority from the angle of environmental preservation, biodegradable polymers from corn chemistry have different purpose of benefits over other manufactured material (Malhotra et al., 2007). Now, the world eyes more focus on learning towards environmental friendly materials, and the need for bio-composite polymer materials. Therefore, there will always be demand on the non-toxicity, course effective and safe bio-composites materials for various industrial application in Malaysia.

In this study, the potential of porous starch and CMC bio-composite will be analysed and discussed. The characteristics of the surface morphology and thermal properties of bio-composites material will be mainly focuses. The effect of the weight ratio of CMC/starch towards the thermal properties and surface morphology of the resulting of bio-composite is also the main concerns in this case study.

1.3 OBJECTIVES OF THE STUDY

The objective of conducting this research are:

i. To prepare porous CMC/starch based composites by using freeze drying techniques.
ii. To characterize the morphology and thermal properties of bio-composites material.

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