STUDY OF THE THERMAL PROPERTIES OF CHITOSAN FILM

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UNIVERSITI TEKNIKAL MALAYSIA MELAKA
STUDY OF THERMAL PROPERTIES OF CHITOSAN FILM

This report submitted in accordance with the requirement of the Universiti Teknikal Malaysia Melaka (UteM) for the Bachelor Degree of Manufacturing Engineering (Engineering Materials) with Honours.

by

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JUDUL: Study of Thermal Properties of Chitosan Film

SESI PENGAJIAN: 2008/2009 Semester 2

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Date : 22nd MAY 2009
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ABSTRACT

Chitosan [(1-4)-2 amino-2-deoxy-d-glucose], the natural polyaminosaccharide derived from N-deacetylation of chitin [(1-4)-2 acetamide-2-deoxy-d-glucose], has recently received a lot of attention due to its huge potential in various fields of application such as water treatment, pulp and paper, biomedical devices and therapies, cosmetics, membrane technology and biotechnology and food applications. The purpose of this study was to determine the thermal and optical properties of chitosan films and chitosan-zeolite composite films by analytical and universal testing instrument such Fourier Transform Infrared Spectroscopy (FT-iR). Other than that, a Direct Current (DC) conductivity measurement test was also conducted at five different temperatures and applied voltage in the range of 0.5 V to 12 V to investigate the ion conductivity. Chitosan films was prepared by blending chitosan powder from Aldrich Chemical Co. in 50% acetic acid concentration, are then dried at room temperature by using the glass casting technique and left for a week. The effect of Zeolite 5A catalyst presence in the chitosan film was evaluated via fabrication of the chitosan-zeolite composite films, that prepared by adding of 0.25 g, 0.5 g, 0.75 g and 1.0 g of Zeolite 5A catalyst in chitosan powder before were blended. FT-iR scanning in wave number range from 4000 cm\(^{-1}\) to 400 cm\(^{-1}\) had detected some changes in chemical structure and bonding of the chitosan as the zeolite was added to the film. The increases of the zeolite content in the chitosan film also had increased the ion conductivity as can be observed in DC conductivity measurement.
ABSTRAK

Kitosan “[(1-4)-2 amino-2-deoxy-d-glucose]”, “polyaminosaccharide” asli diterbitkan daripada “N-deacetylation of chitin [(1-4)-2 acetamide-2-deoxy-d-glucose]” adalah digunakan secara meluas dalam pelbagai aplikasi seperti rawatan air, pulpa dan kertas, alat-alat dan terapi bioperubatan, kosmetik, teknologi membran dan bioteknologi dan juga aplikasi dalam teknologi makanan. Tujuan kajian ini adalah untuk menentukan sifat terma dan optikal filem kitosan dan filem komposit kitosan-zeolite secara analisis dan melalui alat ujian universal seperti “Fourier Transform Infrared Spectrometer” (FT-iR). Selain itu, Ujian Pengukuran Kekonduksian Arus Terus (DC conductivity measurement) turut dilaksanakan pada lima suhu berlainan dan pada voltan berbeza dia antara 0.5 V sehingga 12 V, untuk menguji kekonduksian ion. Melalui teknik tuangan kaca, filem kitosan disediakan dengan mengisar serbuk kitosan daripada Aldrich Chemical Co. dengan asid asetik yang mempunyai kepekatan 50%. dan kemudian ianya dikeringkan selama seminggu di dalam suhu bilik. Kesan kehadiran pemangkin “Zeolite” 5A dalam filem kitosan adalah dikenalpasti melalui penyediaan filem-filem komposit kitosan-zeolite dengan menambah 0.25 g, 0.5 g, 0.75 g dan 1.0 g pemangkin “Zeolite” 5A dalam serbuk kitosan sebelum ianya dikisar. Imbasan FT-iR telah mengenal pasti perubahan dan perbezaan yang terdapat di dalam struktur filem kitosan dan filem kitosan-zeolite. Melalui Ujian Pengukuran Kekonduksian Arus Terus dapat di simpulkan bahawa kehadiran dan pertambahan jumlah zeolite dalam filem kitosan telah meningkatkan kekonduksian ion elektrik.
DEDICATION

To my beloved parents who have always encouraged me to “Go for it!” and supported me wholeheartedly in everything I pursued.
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First and foremost, I would like to express my deepest sense of gratitude to my supervisor Encik Fairuz bin Dimin for his patient guidance, encouragement, detailed and constructive comments and excellent advice throughout this study. His understanding, encouraging and personal guidance have provided a good basis for the present report.

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<td>XRD</td>
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<td>(\alpha)-chitin</td>
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<td>(\mu m)</td>
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CHAPTER 1
INTRODUCTION

1.1 Background of Study

Chitosan, poly-(1-4)-β-glucosamine, constitutes a valuable raw material in the production of film, fibres, beads, sponges, microcrystalline chitosan and fibrils. Chitosan is a carbohydrate polymer that can be derived from crustacean seafood wastes such as shells of crabs, shrimps and crawfish. Chitosan has a wide range of applications in diverse fields ranging from medical sutures and seed coatings to dietary supplements and coagulants for waste treatment. Presently, chitosan is widely used in medicine, veterinary, husbandry, cosmetics and the food industry (Nadarajah, 2005).

Thermal properties of chitosans and their functionalities are affected by their sources (Rhazi et al., 2004) and the methods employed to extract them (Brine et al., 1981). Furthermore, many characteristics of the chitosan (molecular weight, its polydispersity, the purity) are greatly dependant on the method, the equipment used and also of the source of the shells. It is therefore, crucial to control precisely methods of production of the chitosan to obtain the exact characteristics needed for end use application of the product (Beaulieu, 2005). Hence, different thermal properties and functionalities can be expected from chitosans derived from shrimp shell by different composition.

Chitosan film is easily prepared due to its physicochemical properties such as adhesion property, permeability and tensile strength but difficult to handle due to curving during dying phase, if no assisting means and weights are used to keep it straight. Up to now, biocompatible chitosan film has been extensively applied as
biomedical materials and there have been many relative reports such as artificial skin and artificial kidney.

In addition, the intra-structure of macromolecular compound is very complex, and exist crystal phase region and non-crystal phase region. The crystallinity is the weight percent of crystal phase region, and the stable physicochemical properties of chitosan should be ascribed to its high crystallinity, which increases with the de-acetyl degree increasing (Miranda, 2004).

The properties of chitosan can be determined by using Fourier Transform Infrared Spectroscopy (FT-iR). FT-iR is an analytical technique used to identify organic (and in some cases inorganic) materials. This technique measures the absorption of various infrared light wavelengths by the material of interest. These infrared absorption bands identify specific molecular components and structures. It can be applied to the analysis of solids, liquids, and gasses (Bosscher, 2001).

The term Fourier Transform Infrared Spectroscopy refers to a fairly recent development in the manner in which the data is collected and converted from an interference pattern to a spectrum. Samples for FT-iR can be prepared in a number of ways. Alternatively, solid samples can be dissolved in a solvent such as methylene chloride, and the solution placed onto a single salt plate. The solvent is then evaporated off, leaving a thin film of the original material on the plate. This is called a cast film, and is frequently used for polymer identification (Stuart, 2005).

Other than that, the Direct Current (DC) Conductivity Measurement also can exhibit the thermal properties and the ability of the chitosan to conduct the ion. Most polymers are basically have very low electrical conductivities. Thus, under normal electrical fields the measured currents are usually very low. DC conductivity may be attributed to electrons or ions or both. Usually in naturally occurring polymers like chitosan studied here, the dominant conduction mechanism is ionic, the electronic conductivity being very small and very difficult to measure accurately.

Both ionic and electronic conductivities may be present in polymers simultaneously, but usually after a period of time, one type of charge carrier predominates. DC
conductivity studies can give an indication of the charge transfer mechanism involved. Measurements of DC conductivity are usually done while varying the temperature and this can provide further information on the nature of the transport mechanism, or the temperature-dependent characteristics of the conductivity.

1.2 Problem Statement

Proton exchange membrane fuel cell (PEMFC) is a type of fuel cell in which polymer membrane is used as electrolyte. Currently, the most popular membrane used in research and industry comprises of tetrafluorethylene that make it very expensive and hazardous to the environment. The interest of using the chitosan is because of its biodegradable properties and else, it can be easily got as it is just a waste material. Zeolites are hydrated alumino-silicates with symmetrically stacked alumina and silica tetrahedral which result in an open and stable three dimensional honey comb structure with a negative charge. This net negative charge makes zeolite posses high cation exchange capacity and motivates the interest in chitosan-zeolite composite membrane.

In this study, the chitosan-film and chitosan-zeolite film were prepared to investigate the optical and chemical properties of both films by using Fourier Transform Infrared Spectroscopy (FT-iR). Then both films were tested for their electrical conductivity by Direct Current (DC) Conductivity Measurement. In this test, a circular of silver conductive paint with 1.8 cm diameter was painted at each film before a voltage of range 0.5V to 12V was applied to the film to test the current conductivity. This test was conducted at five different temperatures which are at room temperature, 45°C, 55°C, 65°C and also 75°C for each sample.
1.3 Objectives

The objectives of this project are:
(a) To characterize the physical and thermal properties of chitosan film via analytical and universal testing instruments such Fourier Transform Infrared Spectroscopy (FT-iR) and Direct Current (DC) Conductivity Measurement.
(b) To study the effect of the zeolite to the chitosan film and the ability of it to conduct the electricity.
(c) To analyze the FT-iR spectrums of the chitosan film and chitosan-zeolite films.
(d) To investigate the ability of the chitosan-zeolite as a new material in the proton exchange membrane fuel cell (PEMFC).

1.4 Scope Of The Project

The scopes of the project are;
(a) Development of pure chitosan and chitosan-zeolite composites films.
(b) To study the optical properties and thermal properties of pure chitosan and chitosan-zeolite films by using FT-iR.
(c) Analysis of FT-iR spectrums of pure chitosan and chitosan-zeolite film.
(d) Investigate the ability of the chitosan and chitosan-zeolite films to conduct the ion by DC Conductivity Measurement.
2.1 Chitin

The basic principles of the chitin isolation are known since the beginning of 19th century (Beaulieu, 2005). Chitin was described for the first time in 1911 by Professor Henri Braconnot, who was professor of Natural History, director of the Botanical Garden and member of the Academy of Science of Nancy, France. In the pursuit of his researchers on mushroom, he treated Agaricus Volvaceus and other mushroom with diluted warm alkali and isolated chitin, possibly slightly contaminated with proteins. From the dry distillation of this product, called fungi, he obtained a liquid that, upon distillation with potassium hydroxide, yielded ammonia. He described the lack of reaction of chitin with diluted alkali solutions, the compound formed with tannin, the degradation produced by sulfuric acid, the release of acetic acid and several other reactions of chitin (Anonymous, 1998). In 1823, Odier renamed fungine as chitin (meaning tunic in Greek) almost 3 decades before the isolation of cellulose (Beaulieu, 2005).

Chitin is the hard outer shell or exoskeleton that consisted millions of tightly interwoven polymer strands. It can be found in animals (exoskeletons of crustacean and insects) as well as in fungi, mushrooms and yeasts. The exoskeleton is known to give protection to shrimps, crabs, lobsters, scorpions, insects and the others, from their predators (Beaulieu, 2005). Chitin is one of the most abundant polysaccharide in nature (Knorr, 1982) and can be described as a biopolymer composed of repeating units of beta (1-4) 2 - acetamido-2-deoxy-D-glucose (or N-acetyl-D-glucosamine) as shown in Figure 2.1. Its structure is similar to the structure of Cellulose, except that acetylamino groups have replaced the hydroxyl groups (Clarke et al., 2001).
A polysaccharide is a polymer with long-chain carbohydrate made up of hundreds or thousands of linked simple sugars (monosaccharide) such as glucose and closely related molecules. Chitin is similar to cellulose both in chemical structure and in biological function as a structural polymer (No et al., 1995). The crystalline structure of chitin has been shown to be similar to cellulose in the arrangements of inter- and intrachain hydrogen bonding as shown in Figure 2.2 below. Like cellulose, chitin is a fiber, but it has outstanding chemical and biological properties that can be used in a large number of industrial and medical applications (Clarke et al., 2001).

The increased use of chitin (and its derivates) is motivated by the fact that contrary to the petroleum derivatives, chitin is obtained from fisheries by-products, naturally renewable source, non-toxic, non-allergenic, anti-microbial and biodegradable (Peh et al., 2000). While there is exists many extraction methods of the chitin from the crustacean, the principles of chitin extraction are relatively simple (Beaulieu, 2005).
The crustacean waste is the most important chitin source for commercial use due to its high chitin content and ready availability.

However, chitin present in the crustacean waste is associated with proteins, minerals (mainly calcium carbonate) and lipids including pigments. Therefore, chitin purification passes through several steps which are the grinding of the shells to a uniform particle size, protein separation (deproteinization), mineral removal (demineralization) and elimination of pigments and lipids (Mahmoud et al., 2007). The proteins are removed by a treatment in a dilute solution of sodium hydroxide (1-10%) at high temperature (85-100°C). The process of demineralized is done by treating in a dilute solution of hydrochloric acid (1-10%) at room temperatures (Beaulieu, 2005). The processes involved in producing the chitin are shown in Figure 2.3 below.

![Figure 2.3: Process of producing the chitin from the crustacean shell](Source: http://www.france chitine.com/html)

Depending on the severity of these treatments such as temperature, duration, concentration of the chemicals, concentration and size of the crushed shell, the physio-chemical characteristics of the extracted chitin will vary. For instance, the three most important characteristics of the chitin such as the degree of the polymerization, acetylation and purity will be affected (Beaulieu, 2005). Chitin has several properties that define its general behavior and are related to methods of isolation. As a polyacetal it is sensitive to and easily hydrolyzed by acids. It is sensitive to heat even at modest temperature (50°C) and may denature by prolonged heat (Payne et al., 2007).