



Faculty of Resource Science and Technology

**ISOLATION OF CELLULOSE FROM WASTE PAPER FOR THE
PREPARATION OF CELLULOSIC BEADS**

Alice Sia Wei Chen

**Bachelor of Science with Honours
(Resource Chemistry)
2013**

Isolation of Cellulose from Waste Paper for the Preparation of Cellulosic Beads

Alice Sia Wei Chen (25854)

A final project report submitted in the fulfillment of the requirements for the degree of
Bachelor of Science with Honors
(Resource Chemistry)

Supervisor: Associate Professor Dr. Pang Suh Cem
Co-Supervisor: Dr. Chin Suk Fun

Resource Chemistry
Department of Chemistry
Faculty of Resource Science and Technology
Universiti Malaysia Sarawak
2013

Declaration

I hereby declare this thesis submitted is my original research work in support of an application for other degree of qualification or any university or private institution of higher level. This thesis has not used sources without declaration in the text and any quotation were inferred from literature review are clearly marked as such.

.....

(Alice Sia Wei Chen)

Resource Chemistry Programme

Department of Chemistry

Faculty science and Resource Technology

University Malaysia Sarawak

Acknowledgement

First of all, I would to thank to The Department of Chemistry, Universiti Malaysia Sarawak for giving an opportunity to fulfill my Final Year Project.

I would like to express my deepest gratitude to my supervisor, Associate Professor Dr. Pang Suh Cem for his guidance, encouragement and concern throughout the project. He is the one who constantly keep track on my progress and giving my ideas, information and advices on my project and report writing.

Besides that, I would to thank to the master student of Physical Chemistry Laboratory, Kak Fiona for her generosity in helping and giving my ideas and information throughout the project. She lowers my burden and helping me to solve problem that arise in my project. I also like to thank to my friends for their guide, and support. I appreciate the valuable experience, knowledge and laboratory skills that I have gained throughout this project.

Table of Content

Acknowledgement	I
Tables of content.....	II
List of Abbreviations.....	IV
List of Tables	V
List of Figures.....	VI
Abstract.....	1
1.0. Introduction	2
1.1. Objective	3
2.0. Literature Review	4
2.1. Isolation of cellulose from waste paper	4
2.1.1. Pulping process	4
2.1.2. Deinking process	5
2.1.3. Dissolution process	7
2.1.4. Regeneration process	10
3.0. Methodology	11
3.1. Materials	11
3.2. Sample preparation and pretreatment	11
3.2.1. Effect of acetic acid and hydrogen peroxide	11
3.2.3. Effect of acetic acid and sodium chlorite	11
3.3. Deinking of cellulose fibers	12
3.3.1. Effect of blending and sonicate	12
3.4. Dissolution of cellulose	12
3.4.1. Solubility of the cellulose solution	13

3.5. Regeneration of cellulose	13
3.5.1. Effect of heating	14
3.5.2. Effect of volume of cellulose solution	14
3.5.3. Effect of cellulose solution concentration	14
3.5.4. Effect of concentration of surfactant	14
3.6. Characterization technique	15
3.6.1. Scanning Electron Microscope	15
3.6.2. Fourier-Transform Infrared Spectroscopy	15
3.7. Flow chart for methodology.....	16
4.0. Results and Discussion	17
4.1. Pretreatment of waste paper	17
4.2. Deinking of cellulose cellulosic fibers	20
4.3. Dissolution of cellulose	23
4.4. Regeneration of cellulose	24
4.4.1. Effect of heating	24
4.4.2. Effect of volume of cellulose solution	25
4.4.3. Effect of cellulose solution concentration	28
4.4.4. Effect of surfactant concentration	31
5.0. Conclusion	36
6.0. References	37

List of Abbreviations

Sodium hydroxide	NaOH
Hydrogen peroxide	H ₂ O ₂
Carbon disulfide	CS ₂
Hydrogen sulphate	H ₂ S
N-methylmorpholine-N-oxide	NMMO
Scanning Electron Microscope	SEM
Fourier Transform Infrared Spectroscopy	FTIR

List of Tables

Table 1 Cellulose solubility in solvents with different composition.	9
Table 2 Different percentage of cellulosic sample (%w/v) dissolved in NTU solvent system	23
Table 3 Effect of different concentration of surfactant on the mean particles size of the cellulose beads	33

List of Figures

Figure 1	Flow chart for methodology.	16
Figure 2	SEM micrograph of cellulose; (a) wastepaper; (b) wastepaper after alkali pretreatment; and pretreated samples after maceration process; (c) using hydrogen peroxide; and (d) sodium chlorite.	17
Figure 3	FTIR spectra of raw material of cellulose; (a) wastepaper; (b) before alkali pretreatment; after maceration process using (c) hydrogen peroxide; and (d) sodium chlorite.	20
Figure 4	SEM micrograph of cellulosic sample after (a) deinking flotation; (b) blender flotation; and (c) sonicate followed by flotation.	21
Figure 5	Pulp before addition of acetic acid/hydrogen peroxide; (a) flotation; (b) blender followed with flotation; and (c) sonicate followed with flotation	21
Figure 6	FTIR spectra of cellulose; (a) after flotation; (b) sonicate; and (c) blender followed by flotation.	22
Figure 7	SEM micrograph of cellulose beads; (a) & (b) before heating; and (c) & (d) after heating at magnification of 100x and 5000x respectively.	25
Figure 8	SEM micrograph of cellulose beads with; (a) & (b) 1 ml; (c) & (d) 2 ml; (e) & (f) 3 ml; and (g) & (h) 5 ml of cellulose solution at magnification of 100x and 10000x respectively.	26
Figure 9	FTIR spectra of cellulose beads formed at different volume; a) 1ml; b) 2 ml; c) 3 ml; and d) 5 ml of cellulose beads.	28
Figure 10	SEM micrographs of cellulose beads formed with different cellulose concentration; (a) & (b) 1 %(w/v); (c) & (d) 3 %(w/v); (e) & (f) 4 %(w/v) of cellulose solution at magnification of 100x and 10000x	29

respectively.

Figure 11	Effect of cellulose solution concentration on the mean size particles of cellulose beads formed in water-in-oil-emulsion.	30
Figure 12	FTIR spectra of cellulose beads formed at different concentration; (a) 1 %(w/v); 3 %(w/v); (c) 4 %(w/v) of cellulose solution.	31
Figure 13	SEM micrograph of cellulose beads at different concentration of surfactant; a) & b) 0.3 %; c) & d) 0.2 %; e) & f) 0.1 %; g) & h) 0.01 % at magnification of 100x and 10000x respectively.	32
Figure 14	Effect of surfactant concentration on the mean particles sizes of cellulose beads formed in water-in-oil emulsion.	34
Figure 15	FTIR spectra of cellulose beads at different concentration of surfactant; a) 0.3 %; b) 0.2 %; c) 0.1 %, d) 0.01 % at magnification of 100x and 10000x respectively.	35

Isolation of Cellulose from Paper Waste for the Preparation of Cellulosic beads

Alice Sia Wei Chen

Resource Chemistry
Faculty of Resource Science and Technology
Universiti Malaysia Sarawak

ABSTRACT

Cellulose had been extracted from wastepaper through pretreatment, deinking and dissolution process for the preparation of cellulose beads. After dissolution of cellulose, regeneration of cellulose beads was conducted by water-in-oil emulsion. Cellulose solution was added drop by drop into the water-in-oil emulsion to form cellulose beads. The sizes of the cellulose beads were controlled by concentration of cellulose solution and concentration of surfactant. The physical and chemical properties of the cellulosic fibers and beads were investigated by FTIR and SEM. FTIR had been used to determine the presence of cellulose in cellulose beads. The surface morphology of the cellulose beads was characterized by SEM.

Keywords: Cellulose, emulsion, cellulose beads

ABSTRAK

Selulosa telah dikeluarkan dari kertas melalui proses prarawatan, deinking proses dan disolusi. Selepas selulosa dilarutkan, pertumbuhan semula manik selulosa telah dilakukan oleh kaedah emulsi air dalam minyak. Solusi selulosa ditambah setitik demi setitik ke dalam emulsi air dalam minyak untuk membentuk manik selulosa. Saiz manik selulosa dikawal oleh kepekatan larutan selulosa dan kepekatan surfaktan. Ciri-ciri fizikal dan kimia gentian selulosa dan manik telah disiasat oleh FTIR dan SEM. FTIR telah digunakan untuk menentukan kehadiran selulosa dalam manik selulosa. Morfologi permukaan manik selulosa telah disifatkan oleh SEM.

Kata Kunci: Selulosa, emulsi, manik

1.0.Introduction

In 21st century, the increased in education institutions and industries had increased the quantity of paper throughout the world. The attitude of the people in the world who do not concern about recycling the waste papers also increased the production of the waste paper.

About 42% of the industrial woods harvested from the forest world had been used to make paper. (Shapley, 2007). The continual wood harvesting activities had caused effect to environment such as global warming and forest degradation. This is because forest store 50% of the world's terrestrial carbon. Due to this reason, the paper industry had been the fourth largest contributor to greenhouse gases emission in the United State (Shapley, 2007). Besides, it also degraded the ecosystems from sustaining diverse plants and animals, and displaces traditional people, who's lives and cultures are intertwined with the local forest environment.

The paper waste produced are decomposed in the landfill and this led to the released of methane gases followed by the increased in greenhouse effect. In order to reduce the impact of paper-making process toward environment and the decreased in amount of waste paper, paper recycling was introduced. Paper that made through 100% recycled content uses 44% less energy, produces 38% less greenhouse gas emissions, 41% less particulate emissions, 50% less wastewater and 49% less solid waste compared of using virgin wood from the trees (Shapley, 2007). In Japan, 50% of waste newspapers and 90% of cardboard were recycled to solve environment problem (Park *et al.*, 2002).

Recycling of waste paper is defined as recycle the waste paper without additional reagents, additional processes, or additional pretreatment (Park *et al.*, 2002). Besides recycling paper waste into paper, it also can convert into functional cellulosic moldings which are biodegradable and environmental friendly. Some examples of functional cellulosic

moldings that had been produced are egg trays and beads. In this study, extraction of cellulose from waste paper is used to regenerate cellulose beads. Cellulose beads play an important role in various pharmaceutical and biotechnological applications as it is nontoxic and biocompatible material (Wang *et al.*, 2008).

1.1.Objectives

Among research objectives of the proposed study include

- a. To extract cellulose from waste paper by dissolution and regeneration.
- b. To determine the effectiveness of flotation deinking in removing ink particles from waste paper.
- c. To characterize the structure of regenerated cellulose beads.
- d. To control the sizes of regenerated cellulose beads.

2.0. Literature Review

Waste newspaper has been used as the raw material to produce activated carbon that is used as the electrode material for super capacitors like electrochemical double-layer capacitor (EDLCs) (Kalpana *et al.*, 2009). Recently, acid pretreated waste paper is studied as the feedstock for bioethanol production with xylose fermenting *Pichia stipitis* due to the presence of holocellulose in waste paper (Dubey *et al.*, 2012).

2.1. Isolation of Cellulose from Waste Paper

2.1.1. Pulping process

Pulping is a process used to convert raw plant material (wood, straw or bamboo) or recycled paper into a fibrous raw material known as pulp (Ince, 2004). Pulp had been primarily used to make paper and other products derived from cellulose such as film and fiber. In principle, pulp could be produced either from chemical or mechanical pulping or combination of both which known as chemical-mechanical pulping.

Chemical pulping involved the process of extracting cellulose from wood by dissolving the lignin that binds to the cellulose fibers with the use of chemical reactant. Typically, chemical pulping consumes large amounts of virgin wood material. This is due to the dissolution of the organic material into the delignification media, which can reduces the pulp yield to about half of the original raw material (Rousu *et al.*, 2002). Principally chemical pulping consists of alkaline sulfate (Kraft process), acid sulfite, and semi-chemical pulping (Ince, 2004). In Kraft process, the mixture of sodium hydroxide and sodium sulfide is used as the active chemical reagent. Sodium hydroxide is used to degrade lignin and sodium sulfide used to fastens cooking reactions. In acid sulfite, concentrated strong acid such as sulphuric acid and hydrochloric acid had been used widely to treat

lignocelluloses material. No enzymes were required for acid hydrolysis. However, some of the cellulose was hydrolyzed during acidic condition which result in weak fibers if compared with Kraft process and the concentrated acid used are corrosive and hazardous to environment.

Mechanical pulping is the process that produced fibers through mechanical method such as ground wood pulping, refining-refiner pulping. The combination of chemical and mechanical pulp production was called as chemi-mechanical pulp where pulp grounded mechanically after a chemical treatment. If compared to mechanical pulp, Kraft and sulfite pulping had higher market value because their fiber quality was better and more uniform, in which lignin or other wood constituents are less but more cellulose fiber (Ince, 2004).

Delignification of lignocelluloses also could be achieved by treatment with an oxidizing agent such as hydrogen peroxide, ozone, oxygen or air. Hydrogen peroxide was the commonly used oxidative compound as it could remove about 50% of lignin and hemicelluloses. Besides Kraft pulping, organosolv pulping was also a type of chemical pulping method in delignification where organic solvent (formic acid) was used with the mixture of inorganic catalyst (HCl or H₂SO₄ or peroxide). Formic acid allowed an efficient fractionation of raw material into a cellulose rich fraction, a water soluble fraction rich in sugars and lignin fraction (Li *et al.*, 2011b). In the present time, Kraft pulping was being replaced by organosolv pulping due to air pollution caused by the release of sulphur compounds and dust particles (Shirkolae *et al.*, 2007).

2.1.2. Deinking process

In paper waste recycling, deinking process was one of the important steps to remove ink particles. Conventionally, in viscose process, the chemicals needed in deinking process

were NaOH, H₂O₂, and sodium silicate. NaOH functioned as the solvent to adjust the pH of the aqueous medium. The aqueous medium must be in alkaline condition to soften and swollen the paper fibers in order to remove the ink particles from the fiber. Besides, the brightness of the paper also depended on the alkalinity in which the pH value was within the range of 9.5-10.2 (Venugopal, n.d.; Costa & Rubio, 2005). Continuous increased in pH value (>10.2) could led to the decrease in brightness due to the formation of chromophore in lignin. To minimize the formation of chromophores, bleaching agent such as hydrogen peroxide and chelating agent such as sodium silicate had been used (Venugopal, n.d.; Sarja, 2007). However, hydrogen peroxides were not eco-friendly and harmful to the environment. When it is exposed to the stream, it could cause heavy metal poisoning and severely affect aquatic life. Therefore, the usage of the harmful chemical should be avoided and a simple, environmental friendly technology method should be introduced in deinking process.

In order to reduce the effect on the environment and increase the removal of ink particles, two basic methods such as flotation and washing had been introduced. For washing method, the paper pulp was diluted with plenty of water and subjected to filtration repeatedly where the ink particles were removed from system through dewatering. However, this method was seldom used in industrial technology due to some disadvantages such as low efficiency in deinking, low fiber recovery and required large volumes of clean water to produce cleaner pulp (Nie *et al.*, 1997). Flotation is a solid-solid, liquid-liquid, solid-liquid or solid-liquid-liquid separation process, in which the aggregates of particles and bubbles with lower density rise to the surface and removed (Brabty & Marais, 1975). Flotation method had gained world concern due to its simplicity, low cost, less water consumption and its efficiency in removal of ink particles especially large ink particles (Nie *et al.*, 1997).

In flotation method, an appropriate foaming ability was required since the separated particles must be removed through adhesion. To increase foaming ability, surfactant such as foaming agent, collectors and dispersants were used in flotation deinking. The most common surfactant used in flotation was fatty acid soaps (detergents). For example, fatty acids reacted with calcium ions in the system to form calcium soaps which adsorb onto ink surface and provide the collector action. Although fatty acid soap was widely used in flotation deinking, it had given unfavourable effects on deinking performance. Calcium ions were believed to cause scaling and deposition problems on paper machines and other equipment in a deinking plant (Zhou *et al.*, 2004).

Therefore, Venditi (2004) had introduced a simple flotation deinking experiment which could be easily conducted in the laboratory. In his study, fine air bubbles were introduced into suspension that contain of hydrophobic contaminants (inks and toners). The fine air bubbles were attached to the interface of the contaminants and carried to the top of the surface and removed. Liu and Liu (2004) suggested that through air bubble formation, mineral filters and ink particles, with their size ranging between 5 and 20 μm could be effectively collected and removed.

2.1.3. Dissolution process

From decades, several ways to dissolve cellulose with different solvents had been done. In traditional viscose rayon process, dissolution of cellulose had been done by alkaline xanthogenation using carbon disulfide (CS_2) and producing H_2S (Wang, 2008). Besides, cuprammonium technology which generates heavy metal residue was one of the traditional techniques in dissolving cellulose. Due to its high toxicity and its effect to environment, a new organic solvent such as N-methylmorpholine-N-oxide (NMMO) was gradually used to

replace carbon disulfide (CS₂). Due to its strong N-O dipole, NMMO was able to dissolve cellulose effectively (Wang, 2008). In terms of wet and dry strength, and elasticity, NMMO/cellulose process was better than viscose rayon process, but NMMO/cellulose process system was complex and had multiple pathways in the system. Besides NMMO, a better solvent such as NMMO/H₂O had been identified. The product formed from the system was known as Tencel or Lyocell. The advantage of NMMO/H₂O solvent system was its ability to attain high cellulose concentration. But, these solvent systems required high temperature for dissolution, degradation of cellulose, side reaction of the solvent itself without antioxidant and the high cost were not recommended to replace viscose technology (Zhang *et al.*, 2008). Thus, a simple, environmental friendly and low cost cellulose solvents such as NaOH was introduced.

Dissolution of cellulose fiber in NaOH solution at low temperature had been studied by Wang (2008). His study focused on the interaction of cellulose with alkali solution at low temperature, the improvement of its solubility, and the effect of hemicelluloses and lignin. The presence of lignin had decreased the solubility whereas no effect occurred in the presence of hemicelluloses. The dissolving ability had shown to improve with decreasing temperature from -15 to 4 °C (Zhang *et al.*, 2010a).

Besides NaOH solvent, new non-derivative solvents such as NaOH/urea and NaOH/thiourea had been used to dissolve cellulose at low temperature (Cai & Zhang, 2006; Cai *et al.*, 2007; Ruan *et al.*, 2004; Cai & Zhang, 2005). These solvents were preferable because celluloses were able to dissolve easily, quickly and stable cellulose solution can be produced. However, spinning solutions containing high concentrations cellulose in these two solvents were unstable, which was a disadvantage in industrial applications.

Recently, Zhang *et al.* (2010a) had studied the solubility of cellulose in aqueous mixture of NaOH/urea/thiourea solvent. In dissolution of cellulose, NaOH play a role in penetrating into crystalline to destroy inter- and intra- hydrogen donor between cellulose bonds meanwhile urea and thiourea functioned as the hydrogen donor and receptor to prevent re-association of the cellulose molecular chains (Zhou & Zhang, 2000). The breakdowns of the hydrogen bond prevent re-association led to a good dispersion of cellulose to form solution. Zhang *et al.* (2010b) had studied that different composition of solvent could result in different cellulose solubility and the aqueous mixture of 8 wt% of NaOH, 8 wt% of urea and 6.5 wt% of thiourea composition had shown the most favourable alkaline aqueous solution in dissolving cellulose (Table 1).

Table 1: Cellulose solubility and solvents with different composition (Zhang *et al.*, 2008).

NaOH (wt%)	Urea (wt%)	Thiourea (wt%)	Solubility (wt%)
7	12	4.5	0.49
7.5	12	5	0.66
8	8	6.5	0.84
8.5	6	5.5	0.8
9	4	5.5	0.8
9.5	0	4.5	0.63
10	2	5.5	0.75

On a comparison of solubility of cellulose with other solvent system, this mixture solution (NaOH/urea/thiourea) had shown the most powerful solvent. This is because it was less toxicity and able to prepare more stable spinning solutions containing higher concentrations of cellulose than all aqueous NaOH solution (Jin *et al.*, 2007).

2.1.4. Regeneration of cellulose beads

Cellulose beads are particles that are used for various types of application, mainly for immobilization, purification, separation and filtration purposes. Cellulose beads could be regenerated either through chemically or mechanically. According to Bai and Li (2006), regeneration of cellulose beads could be done by dissolving cellulose derivative (viscose, cellulose ether, ether) in a coagulating bath which chemical composition was tuned to perform de-derivatisation.

Besides using “de-derivatisation” step, cellulose dissolve either by using N-oxide monohydrate (NMMO) or NaOH mixed with or not with urea or thiourea could be used to regenerate cellulose beads (Sescousse *et al.*, 2010). In chemical methods, cellulose solution was stirred in dispersion medium to disperse small droplets of the cellulose solution utilizing phase separation between a cellulose solution and a dispersion medium and coagulating the droplets to form cellulose beads. This was known as emulsion method. This was proven by Suvachitanont and Pookingdao (2011) when water in oil emulsification thermal regeneration method was used to regenerate into beads from cellulose that were extracted from corn.

Mechanically, cellulose beads could be made from solution by either using classical droplet-making machines like atomizer and jet splitters (Rosenberg *et al.*, 2007). Cellulose solution also could be sprayed with high pressure gas through nozzles and droplet formed was coagulated and dried to obtain cellulose beads (Morimoto *et al.*, 1997). Cellulose solution also could be sprayed with high pressure gas through nozzles and droplet formed was coagulated and dried to obtain cellulose beads (Morimoto *et al.*, 1997). In laboratory scale, beads could be made through a syringe or by pressing a solution through a perforated material (Linko *et al.*, 1977).

3.0. Methodology

3.1. Materials

The cellulosic sample used in this study was office paper waste. Chemicals used were sodium hydroxide, hydrochloric acid, acetic acid, hydrogen peroxide, urea, thiourea, absolute ethanol, acetone and Span 60.

3.2. Sample Preparation and Pretreatment

The waste paper samples were cut into small sizes and soaked in NaOH (12 % w/v) for 2 hours to enable the solvent to penetrate through the crystalline region of the cellulose. In order to remove the impurities and unwanted compound, acid pretreatment was carried out with 1M HCl for 2 hours and followed by alkali treatment using NaOH (2 % w/v) for 2 hours (Yek, 2010). Then, the pretreated cellulosic samples were filtered with vacuum pump and thoroughly washed with distilled water.

3.2.1. Effect of acetic acid and hydrogen peroxide

The cellulosic samples were soaked in the solution of acetic acid and hydrogen peroxide in the ratio of (1:1) for 4 days at temperature of 80 °C. The resulting cellulosic samples were then collected by vacuum filtration and washed with distilled water.

3.2.2. Effect of acetic acid and sodium chlorite

The cellulosic samples were pretreated by using 0.2 ml of acetic acid and 0.5 g of sodium chlorite at 70 °C for 2 hours. The resulting cellulosic samples were collected by vacuum filtration and washed with distilled water.

3.3. Deinking of Cellulosic Fibers

To remove the ink particles from cellulose fibers, the flotation de-inking process was carried out. The detergent which was acted as the collector was added into the beaker containing pretreated cellulose sample and the removal of ink particles was done by pumping air bubbles into beaker. Foam containing ink particles were taken off from the top of the beaker whereas the cleaned fibers were remained in the container (Venditti, 2004).

3.3.1. Effect of blending and sonicate

Before flotation, the cellulose samples were either blended by using machine blender or sonicated for half an hour to disperse the cellulosic fibers in order to enhance the removal of ink particles from the fibers.

3.4. Dissolution of cellulose

Cellulose solution was prepared by dissolving cellulose fibers in an aqueous-based solvent mixture of 8 wt% of NaOH, 8 wt% of urea, 6.5 wt% of thiourea. The sample of cellulose fibers were mixed well in the solvent and cooled to -20 °C in freezer overnight until they become a solid frozen solution (Zhang *et al.*, 2010a). The frozen solution was then thawed

under room temperature before underwent regeneration. A clear solution obtained would show the cellulose fibers had completely dissolved in the solution. When the cellulose fibers were slightly dissolved in the solution, vigorously stirring was done until the cellulose fibers dissolved.

3.4.1. Solubility of cellulose

Cellulose at 1 %w/v, 3 %w/v, 5 %w/v and 8 %w/v were dissolved in the 25ml solution of NTU respectively. The cellulose solutions were cooled to -20 °C in freezer overnight until they become a solid frozen solution. The frozen solution was then thawed under room temperature and the solubility of cellulose was determined.

3.5. Regeneration of cellulose

The cellulose solution was regenerated as cellulose beads using the water-in-oil emulsification regeneration method. A mixture containing 1:4 ratio of ethanol:oil and a small amount of Span 60 were mixed until completely dissolved. Then, an amount of cellulose solution was added dropwise into the mixture to form small droplet and stirred for 2 hours. After 2 hours, the cellulose particles were collected and washed with ethanol by centrifuge and acetone was used to wash the excess oil. After centrifuged, it was dried in the oven.

3.5.1. Effect of heating

A small amount of cellulose solution was added drop by drop into mixture of oil, ethanol and Span 60 and stirred for 2 hours. After stirring for 2 hours, the emulsion was heated at 80 °C for 1.5 hours. The resulting precipitate was collected and washed with ethanol by centrifuge. After centrifuged, it was dried in the oven.

3.5.2. Effect of volume of cellulose solution

Varying volume (1ml, 2ml, 3ml, 5ml) of 5% cellulose solution was added drop by drop into the water-in-oil emulsion for 2 hours. The resulting precipitate was collected and washed with ethanol by centrifuge. After centrifuged, it was dried in the oven.

3.5.3. Effect of cellulose solution concentration

After the mixture of ethanol, oil and Span 60 was dissolved, 1 ml cellulose solution of varying concentration (1 %w/v, 3 %w/v, and 5 %w/v) was added into the water-in-oil emulsion drop by drop and then stirred for 2 hours. The resulting precipitate were collected by and washed with ethanol by centrifuge. After centrifuged, it was dried in the oven.

3.5.4. Effect of surfactant concentration

Span 60 was used as the surfactant. Varying concentration of surfactant (0.3 %w/v, 0.2 %w/v, 0.1 %w/v and 0.01 %w/v) of was added into 20 ml of solution containing 16 ml of oil and 4 ml of ethanol. After addition of surfactant, the solution was stirred until all the surfactants were dissolved. 1 ml of 5 %w/v cellulose solution was then dropped into the