THE REMOVAL OF OIL FROM OIL-IN-WATER EMULSIONS BY MODIFIED KAPOK FIBER WITH HYDROCHLORIC ACID

LEE LI YING

UNIVERSIITI SAINS MALAYSIA

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by

LEE LI YING

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LIST OF SYMBOLS

Symbol	Description	Unit
A_i	Initial highest absorbance of absorbate	Dimensionless
A_f	Final highest absorbance of absorbate	Dimensionless

LIST OF ABBREVATIONS

ADT-$(CF_3)_2$	Bis(trifluoromethyl)benzenediazonium tosylate
BAT	Best available technologies
DC	Deposited carbon black
DT	Dodecanethoil
FA	Fly ash
FeNPs-$(CF_3)_2$	Iron nanoparticles
FTIR	Fourier transform infrared spectroscopy
GAC	Granular activated carbon
HCl	Hydrochloric acid
MF	Melamine
O/W	Oil-in-water
PAC	Powdered activated carbon
PDA	Polydopamine
PU	Polyurethane
SEM	Scanning electron microscopy
USEPA	United States Environment Protection Agency
UV-Vis	Ultraviolet-visible
W/O	Water-in-oil
WCA	Water contact angle

PENYINGKIRAN MINYAK DARIPADA EMULSI MINYAK DALAM AIR OLEH GENTIAN KAPUK YANG DIUBAH SUAI DENGAN ASID HIDROKLORIK

ABSTRAK

Serat kapok digunakan secara meluas dalam penyerapan minyak kerana ia menawarkan banyak faedah seperti kapasiti penyerapan minyak yang sangat baik, biodegradasi, kebolehgunaan semula, kelimpahan dan kos rendah. Rawatan serat kapuk dengan asid hidroklorik (HCl) timbul untuk meningkatkan penyerapan minyak. Oleh itu, kerja penyelidikan ini disasarkan untuk menghasilkan gentian kapuk yang diubah suai dengan larutan HCl. Dalam kajian ini, gentian kapuk telah disintesis pada kepekatan HCl, suhu rawatan dan masa rawatan yang optimum. Dengan gentian kapuk yang diubah suai, 94.90% penyingkiran minyak daripada emulsi minyak dalam air dicapai pada kepekatan minyak awal 1000 ppm. Kecekapan pengasingan telah dipertingkatkan dengan peningkatan kepekatan HCl, suhu rawatan dan masa rawatan. Ini disebabkan oleh peningkatan pembungkusan kristal dan kekasaran permukaan gentian kapuk. Walau bagaimanapun, apabila parameter ini meningkat melebihi nilai optimumnya iaitu 1.5%, 70 °C dan 50 minit, prestasi gentian kapuk menurun kerana struktur kapuk yang rosak dalam keadaan yang melampau. Kapasiti penyerapan gentian kapuk yang diubah suai juga dipertingkatkan dengan mengurangkan kepekatan minyak awal kerana kawasan permukaan yang lebih besar untuk menyerap minyak kekal. Hasilnya membuktikan bahawa serat kapok adalah penyerap yang menjanjikan untuk mengeluarkan minyak daripada emulsi minyak dalam air dalam air sisa industri.

THE REMOVAL OF OIL FROM OIL-IN-WATER EMULSIONS BY MODIFIED KAPOK FIBER WITH HYDROCHLORIC ACID

ABSTRACT

Kapok fiber is widely used in oil absorption as it offers many benefits such as excellent oil absorption capacity, biodegrability, reusability, abundancy and low cost. The treatment of kapok fiber with hydrochloric acid (HCl) arises to enhance the absorption of oil. Hence, this research work targeted to generate modified kapok fiber with HCl solution. In this study, kapok fiber was synthesized at an optimized HCl concentration, treatment temperature and treatment time. With the modified kapok fiber, 94.90% of oil removal from oil-in-water emulsions was reached at an initial oil concentration of 1000 ppm. The separation efficiency was improved with increasing of HCl concentration, treatment temperature and treatment time. This was due to the enhancement of crystalline packing and surface roughness of kapok fiber. However, when these parameters increased beyond their optimum values which are 1.5%, 70 °C and 50 minutes, the performance of kapok fiber dropped because of the damaged kapok structure under extreme conditions. The absorption capacity of modified kapok fiber was also improved by decreasing the initial oil concentration due to larger surface area for absorbing oil remained. The results proved that kapok fiber is a promising absorbent to remove oil from oil-in-water emulsion in industrial wastewater.

CHAPTER 1

INTRODUCTION

1.1 Background

Every year, a huge amount of oily wastewater is produced from various types of modern manufacturing processes such as petrochemicals, food production, paper, textile, metal processing industries, etc. (Elanchezhiyan et al., 2018). The oily wastewater pollution has become a major concern over the world as it consists of various toxic substances such as polyaromatic hydrocarbons, petroleum hydrocarbons and phenols These toxic substances can act as inhibitors to animal and plant growth (Alade et al., 2011). Therefore, the oily wastewater has caused severe ecological problem to all the living organisms by affecting crop production, drinking water, aquatic resources and human health (Elanchezhiyan et al., 2018; Yu et al., 2017). Thus, it is crucial to remove the oil from the wastewater before discharging it into the environment. However, oil separation has always been a time consuming process as the oily wastewater usually consists of oil-in-water emulsions in a very high volume (Primasari et al., 2011). In these emulsions, the oil droplets spontaneously coalesce into larger flocs, therefore, causing it more difficult to degrade their aliphatic and aromatic components (Elanchezhiyan et al., 2018; Primasari et al., 2011).

Oil-in-water emulsions are usually formed due to the presence of emulsifiers (Cassiday, 2014). Emulsifier is commonly used in food industry to improve the appearance, texture, stability and shelf life of food product (Atchley, 2019). It is a surfactant which has a hydrophobic head and a hydrophilic tail. Hence, when the emulsifiers are added to the oily water, their hydrophobic heads are attracted to the non-polar components (oil droplets) whereas their hydrophilic tails are attracted to the polar components (water). Therefore, the interfacial tension between the oil and water will be reduced causing the oil droplets to be more stable (Cassiday, 2014). Thus, it is difficult to separate oil from the emulsions so many researchers have carried out some studies to solve this problem.

There are many conventional technology available for the removal of oil from oil-in-water emulsions, for example, membrane bioreactor, reverse osmosis, chemical dosing, ultrafiltration, air flotation, gravity separation etc. (Okiel et al., 2011). However, among all the existing techniques, sorption is the most preferable due to its simplicity, low cost, eco-friendly, high separation efficiency and easy recovery (Akpomie and Conradie, 2021; Elanchezhiyan et al., 2018).

For sorption technique, sorbent materials used for oil removal can be classified into three categories: natural organic materials (kapok fiber, cotton fiber, risk husk, banana peel and milkweed), natural inorganic materials (zeolite, silica, activated carbon and graphite) and synthetic organic polymers (polyurethane foam, polyethylene, polypropylene and polyacrylate) (Akpomie and Conradie, 2021; Dong et al., 2015; Wang et al., 2012). Among all these materials, kapok fiber is the most preferred because it is cheap, hydrophobic, biodegradable, reusable and capable of absorbing high amount of oil (Wang et al., 2012).

Kapok fiber is a natural product obtained from the fruits of silk-cotton tree. It contains 64% of cellulose, 13% of lignin and 23% of polysaccharides. There is a small amount of waxy coating on its surface, thus, making it hydrophobic (Dong et al., 2015). To improve the oil absorbency of kapok fiber, it can be treated with hydrochloric acid

(HCl) to modify its hollow structure, surface, surface wax and crystallinity of fiber wall matrix. It was reported that the modified kapok fiber could achieve approximately 35 g/g of toluene absorbency. However, the performance of kapok fiber is strongly affected by the concentration of HCl solution, treatment temperature and treatment time (Wang et al., 2012). These parameters need to be optimized to generate the kapok fiber which can exhibit excellent oil absorbency in oil-in-water emulsions.

1.2 Problem Statement

The kapok fiber developed by the previous researchers showed a good performance in absorbing oil. However, it was only tested in absorbing oil in the absence of water and emulsifier. In study conducted by Wang et al. (2018a), it was reported that the separation efficiency of the sorbent would decrease due to the complex structure of the oil components. Thus, if the sorbent is used to treat the oily wastewater in industry that contains various kinds of emulsifiers, the performance may be affected as well. Hence, the performance of the kapok fiber proposed by Wang et al. (2012) on the industrial oily wastewater must be further studied. The optimized concentration of HCl solution, treatment time and treatment temperature must be determined as well to optimize the oil absorption capacity of modified kapok fiber. Hence, it is proposed that in this study, kapok fibers treated with HCl solutions under different HCl concentrations, treatment temperatures and treatment time will be used as the sorbents to remove oil from oil-in-water emulsions in the synthetic wastewater which is composed of cooking oil, distilled water and food emulsifier, sorbitan tristearate (STS).

1.3 Objectives

i. To optimize the modified conditions for kapok fiber treatment using HCl solution
ii. To evaluate the performance treated kapok fiber with HCl solution on emulsion solution
iii. To study the effect of initial oil concentration on the oil absorption of the modified kapok fiber

1.4 Project Scope

The study mainly focuses on the oil removal efficiency from oil-in-water emulsions by using kapok fiber modified with HCl solution. The operating parameters such as HCl concentration, treatment temperature and treatment time were manipulated to study their effects on the performance of the treated kapok fiber. The oil absorption capacity of the treated kapok fiber was tested on oil-in-water emulsion which was synthesised by cooking oil, distilled water and food emulsifier, STS.

CHAPTER 2

LITERATURE REVIEW

2.1 Emulsion Formation

Emulsion is one of the important components in food products, pharmaceutical products, personal care products and cosmetic products (McClements and Jafari, 2018). It is a mixture composed of two immiscible liquids, for example, oil and water (Entrée, 2010). In this mixture, one liquid (dispersed phase) is dispersed in another liquid (continuous phase) in the form of droplets (Entrée, 2010; LLS Health CDMO, 2019). There are two types of emulsions which are oil-in-water (O/W) emulsions and water-in-oil emulsions (W/O). The O/W emulsions have water as the continuous phase whereas the W/O emulsions have oil as the continuous phase. The sizes of the liquid droplets usually range from 1 μm to 50 μm (Entrée, 2010).

In fact, emulsion is thermodynamically unstable so the physicochemical mechanisms such as phase separation, particle coalescence, flocculation, Ostwald ripening and gravitational separation can break down the emulsions easily after a period of time. Hence, in order to stabilize the emulsions, emulsifiers are added in the emulsion formulation. Emulsifier is an amphiphilic molecule which contains both hydrophobic and hydrophilic parts on the same molecule. There are several examples of emulsifiers which are phospholipids, polysaccharides, proteins and surface-active polymers (McClements and Jafari, 2018). Due to the amphiphilic properties, when the emulsifiers are added to the emulsions, their hydrophobic groups are attracted to the non-polar components (oil) whereas their hydrophilic groups are attracted to the polar components (water) (Cassiday, 2014). Thus, a film is formed between the dispersed droplets and the continuous phase (Team, 2020). This film will eventually act as a physical barrier that creates repulsive force to prevent the droplets from contacting and coalescing (LLS Health CDMO, 2019).

The interfacial tension between the two liquid phases will also be lowered, therefore, stabilizing the dispersed droplets (Cassiday, 2014). In most of the industrial applications, different types of emulsifiers are combined and used in the emulsion formulation to enhance the stability, functional attributes and formation of the emulsions (McClements and Jafari, 2018). Hence, the removal of oil from the emulsions has become more challenging and many researchers have carried out some studies to solve this problem.

2.2 Sorbents for Oil-in-Water Emulsions Separation

2.2.1 Modified Kapok Fiber with HCl Solution

Kapok fiber is a natural fiber produced from the fruits of silk-cotton tree. It consists of cellulose, lignin, polysaccharides and a small amount of waxy coating which causes its hydrophobicity. Due to its low cost, hydrophobicity, reusability, biodegrability and excellent performance in absorbing oil, kapok fiber is widely applied in removing oil from wastewater. It was found that the hollow structure, surface, surface wax and crystallinity of fiber wall matrix of kapok fiber could be changed by treating it with HCl solution. The treated kapok fiber has a higher oil absorption capacity than the raw kapok fiber. Approximately 35 g/g of toluene absorbency can be achieved by kapok fiber, which has been treated with 1% of HCl solution at 80 °C for 1 hour, after 15 minutes. It was reported that the absorption capacity of kapok fiber was not influenced significantly after being reused for 8 times. However, the modified kapok fiber was tested for absorbing oil in the absence of water and emulsifier (Wang et al., 2012).

2.2.2 PU Foam Coated with FA, PDA and DT

A novel oil sorbent was developed by being coated polyurethane (PU) foam with fly ash (FA), polydopamine (PDA) and dodecanethoil (DT). PU foam is a cheap porous material that exhibits superhydrophobicity but it ignites easily causing some parts to turn into ashes so they can no longer function as the sorbent. Therefore, FA which is a waste generated from the burning of coal, fossil fuel, etc. was used to modify PU foam as it is resistant to flame. In addition, FA can create roughness along with PDA, thus, improving the superhydrophobicity of the PU foam. Meanwhile, DT plays the role in improving the absorption band intensity of the foam.

It was reported that the modified foam could achieve above 93.0% of separation efficiency for six different types of oil-in-water emulsions (n-hexane-in-water, toluene-in-water, chloroform-in-water, kerosene-in-water, gasoline-in-water and diesel-in-water emulsions). However, the separation efficiencies of gasoline-in-water and diesel-in-water emulsions were relatively lower than others because of their complicated structures. For toluene-in-water and kerosene-in-water emulsions, almost all of the oil droplets were successfully removed within 2 to 3 minutes. The superhydrophobicity of the modified foam could be maintained after being reused for 15 cycles. It was also found that the modified foam could perform well in highly acidic, alkaline and salty solutions (Wang et al., 2018a).

2.2.3 MF Foam Coated with PDA and DT

In study conducted by Wang et al. (2018b), a similar absorbent was used which was melamine (MF) sponge coated with PDA and DT. The sponge managed to remove 93.8%, 92.7%, 91.5%, 76.6% and 84.2% of oil for n-hexane-in-water, toluene-in-water, chloroform-in-water, gasoline-in-water and diesel-in-water emulsions respectively. It was observed that the solutions turned transparent after addition of the sponge for 3 minutes. The absorption capacity of the sponge was not affected significantly after being reused for at least 60 times (Wang et al., 2018b).

2.2.4 Cotton Fabric Coated with Calcium Stearate and Filled with Kapok Fiber

Meanwhile, Wang and Wang (2018) found a new potential absorbent which was cotton fabric coated with calcium stearate and filled with kapok fiber. It exhibits both oleophilic and hydrophobic properties. It was found that the material could reach more than 98.9% of separation efficiency for toluene-in-water emulsion after 5 minutes when the filling amount of kapok fiber used was more than 0.3 g. Furthermore, more than 97.7% of separation efficiency could be achieved for toluene-in-water emulsion in acidic, alkaline and salty solutions after 5 minutes. After reusing the modified cotton fabric for 12 cycles, the oil absorption capacity decreased by less than 7.3%. However, it was reported that when the amount of fiber filling was less than 0.3 g, there was a small percentage of absorbed oil escaping from the fiber due to its loose structure (Wang and Wang, 2018).

2.2.5 Magnetic Polyurethane Sponge

A magnetic polyurethane (PU) sponge was developed as the sorbent for oil-in-water emulsions separation (Guselnikova et al., 2020). PU sponge was selected as the sorbent because of its high specific surface area, high sorption capacity, low specific weight, low mass production and low cost (Peng et al., 2019; Wang et al., 2014; Wu et al., 2015). However, the hydrophobicity of PU sponge makes it not suitable to adsorb the oil selectively from the emulsions so the PU sponge was modified with 3,5-bis(trifluoromethyl)benzenediazonium tosylate (ADT-$(CF_3)_2$). ADT-$(CF_3)_2$ is a diazonium salt which can provide the superhydrophobicity to the PU sponge. The magnetic nanoparticles, FeNPs-$(CF_3)_2$ which had been modified by ADT-$(CF_3)_2$ was then added to the modified PU sponge. These magnetic nanoparticles contributes to the superhydrophobicity of the PU sponge as well besides improving sorption capacity and thermal stability of the PU sponge.

It was reported that after immersing the magnetic PU sponge in water-in-oil emulsions which were pump oil/water and pluronic/pimp oil/water, above 99% of oil purity was achieved. This indicated that the magnetic PU sponge was superhydrophobic exhibiting good separation efficiency. The superhydrophobic properties of magnetic PU sponge could be maintained at the temperature up to 160 °C as its thermal stability had been improved by the addition of magnetic nanoparticles. Moreover, after reusing the sorbent for 8 times, it was found out that the water contact angle (WCA) decreased very slightly (Guselnikova et al., 2020). However, water-in-oil emulsions were tested and the oil sorption capacity for the emulsions were not observed in this paper.

2.2.6 Activated Carbon

Activated carbon is one of the common adsorbents used to remove the organic compounds such as oil from the water and it has been confirmed to be practicable (Reynolds and Richards, 1996). The United States Environment Protection Agency

(USEPA) also suggested activated carbon as one of the best available technologies (BAT) for the removal of the organic compounds (Nicholas, 2002). However, the cost of activated carbon is high (Okiel et al., 2011). There are two types of activated carbon which are powdered activated carbon (PAC) and granular activated carbon (GAC). In research conducted by Ayotamuno et al. (2006), it was observed that PAC showed a better separation efficiency for petroleum hydrocarbon contaminated ground-water as compared to GAC. Therefore, Okiel et al. (2011) further studied the performance of PAC on the oil-in-water emulsions separation using the wastewater of a petroleum company. It was found out that 0.5 g of PAC could achieve 80.6% of oil removal at initial oil concentration of 1000 mg/L after 2 hours (Okiel et al., 2011).

2.2.7 Bentonite

Bentonite has also been studied for the removal of oil from oil-in-water emulsions due to its high porosity, large surface area, low cost and abundant source (Okiel et al., 2011). Bentonite managed to remove the oil from oily wastewater as much as 50% of its weight in oil. This was approximately 5 to 7 times the oil adsorption capacity of the activated carbon (Alther, 1995; McBride et al., 1977). In addition, when 0.5 g bentonite was used to treat the oily wastewater of a petroleum company, 91.5% of oil removal could be reached after 2 hours at initial oil concentration of 1000 mg/L (Okiel et al., 2011).

2.2.8 Deposited Carbon Black

Deposited carbon black (DC) is one of the components of diesel exhaust. Approximately 73 to 80% of the diesel exhaust is deposited carbon black (Watson et al., 1994; Zaebst et al., 1991). The diesel exhaust is normally produced via the fuel combustion.

Therefore, deposited carbon black is cheap as it can be easily obtained. It has the potential to be used as the adsorbent for oil-in-water emulsions separation as well due to its high porosity and large surface area. It was reported that 0.5 g of DC could give 93.6% of oil removal for the industrial oily wastewater at initial oil concentration of 1000 mg/L after 2 hours (Okiel et al., 2011).

2.2.9 Summary and Comparison

Table 2.1 Summary and Comparison of Sorbents for Oil-in-Water Emulsions Separation

Materials	**Performance**	**Time**	**Cost**	**Reusability**	**References**
Kapok fiber treated with HCl solution	• Achieve 35 g/g toluene absorbency in the absence of water and emulsifier	15 min	Low	Can be reused for 8 cycles	(Wang et al., 2012)
PDA/FA/DT coated foam	• Achieve more than 93.0% of oil removal • Flame-	2-3 min	Low	Can be reused for 15 cycles	(Wang et al., 2018a)

	retardant • Can be used under extremely harsh and turbulent water environment				
MF coated with PDA and DDT	• Achieve 93.8%, 92.7%, 91.5%, 76.6% and 84.2% of oil removal for n-hexane, toluene, chloroform, gasoline and diesel respectively	3 min	Low	Can be reused for at least 60 times	(Wang et al., 2018b)
Cotton fabric filled with kapok fiber	• Achieve above 98.9% of oil removal	5 min	Low	Can be reused for 12 cycles	(Wang and Wang, 2018)

	• Can be used in acidic, alkaline and salty solutions				
Magnetic PU sponge	• Achieve 99% of oil purity for water-in-oil emulsions • Has good thermal stability	5-8 min	Low	Can be reused for 8 times	(Guselnikova et al., 2020)
DC	Achieve 93.6% of oil removal	2 hr	Low	-	(Okiel et al., 2011)
Bentonite	Achieve 91.5% of oil removal	2 hr	Low	-	(Okiel et al., 2011)
PAC	Achieve 80.6% of oil removal	2 hr	High	-	(Okiel et al., 2011)

Based on Table 2.1, kapok fiber treated with HCl solution has great potential to be used as sorbent to treat the industrial oil-in-water emulsions due to their high oil absorption capacity, short time, low cost and long lasting. In addition, it requires less types of materials for the sorbent preparation.

CHAPTER 3

METHODOLOGY

3.1 Materials and Equipment

The variables for the experiment are HCl concentrations, treatment temperature and treatment time.

Table 3.1 shows the list of raw materials required to prepare modified kapok fiber with HCl solution and oil-in-water emulsion. Meanwhile, Table 3.2 shows the list of equipment required to prepare and characterize modified kapok fiber with HCl solution and oil-in-water emulsion.

Table 3.1 List of Raw Materials

Material	Usage
Kapok fibre	Raw material of sorbent
Hydrochloric acid	Chemical used to treat kapok fiber
Distilled water	Sample to dilute hydrochloric acid and form oil-in-water emulsion
Cooking oil (sunflower oil)	Sample to form oil-in-water emulsions
Sorbitan tristearate (STS)	Sample to stabilize oil-in-water emulsions

Table 3.2 List of Equipment

Equipment	Usage
Electric grinder	Crush the raw kapok fiber into smaller sizes
Weighing machine	Measure the weight of kapok fiber, cooking oil and STS
Oven	To dry the treated kapok fiber
UV-Vis spectrophotometer	Determine the concentration of oil-in-water emulsions
Magnetic mixer	To heat the HCl solution and stir the HCl solution and kapok fiber and oil-in-water emulsion
Mechanical stirrer	To stir mixture of cooking oil, distilled water and STS for oil-in-water emulsion preparation
Stopwatch	To record time for kapok fiber treatment and oil removal using kapok fiber
Filter paper	Separate kapok fiber from aqueous medium

3.2 Flow Chart of Methodology

In general, the project is classified into two sections: synthesis of optimum kapok fiber and its application in oil-in-water emulsion, which can be illustrated through the flow chart as shown in Figure 3.1.

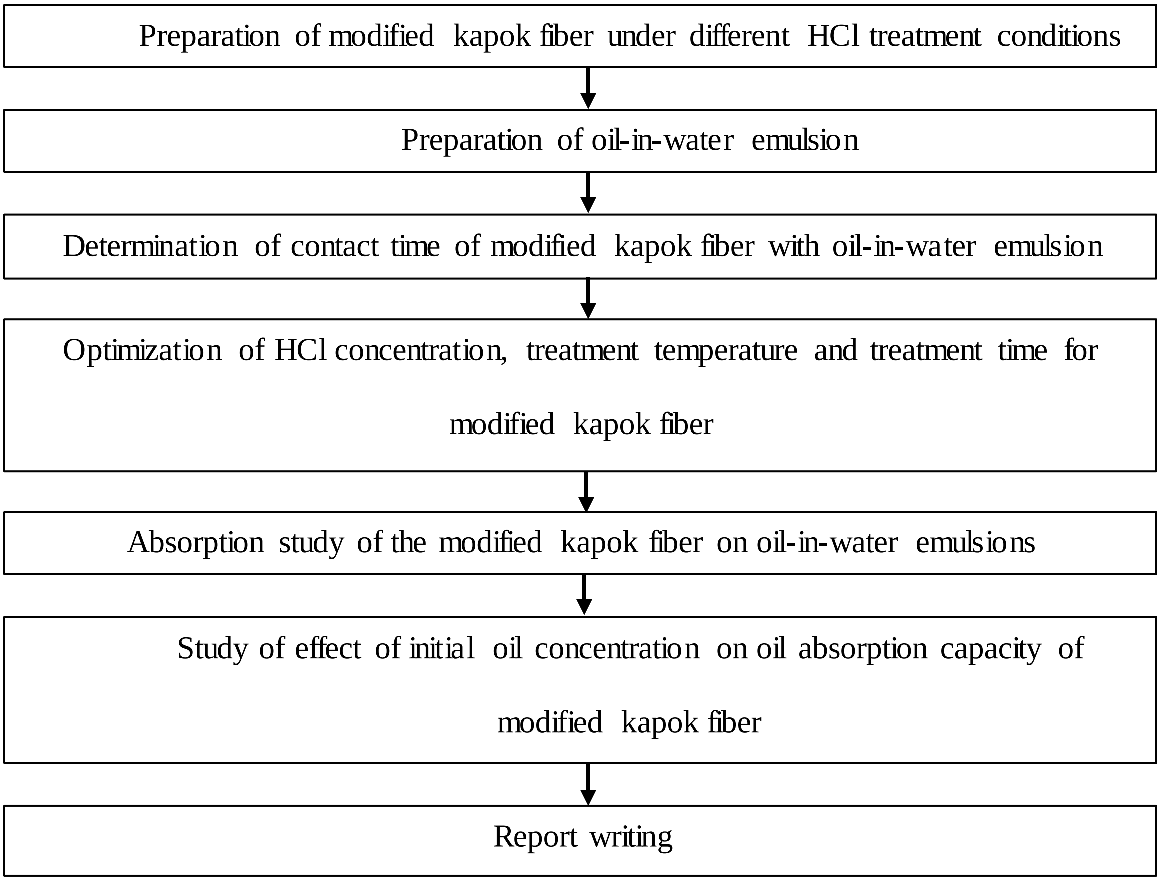

Figure 3.1 Flow Chart of Methodology

3.3 Preparation of Modified Kapok Fiber with HCl Solution

Raw kapok fiber was crushed into smaller sizes by using an electric grinder. 100 mL of HCl solution with the required concentration (0 – 2.0 %) was prepared and heated to the required treatment temperature (50 - 90 °C) using a magnetic mixer. 1 g of kapok fiber was immersed in the heated HCl solution. After the required treatment time (20 – 60 min) reached, the kapok fiber was filtered and rinsed with distilled water for few times. It was then dried at 70 °C for 4 hours (Wang et al., 2012).

3.4 Preparation of Oil-in-Water Emulsion

For 5000 ppm of oil-in-water emulsion preparation, 9 g of cooking oil was weighed and then added with 1 g of STS. This mixture was then placed in a 2 L volumetric flask. Distilled water was added into the volumetric flask until the calibration line was reached. The mixture was then transferred to a large plastic beaker and stirred at 6000 rpm by using a mechanical stirrer for 1 hour. For preparation of oil-in-water emulsion with other concentrations (1000 – 4000 ppm), 5000 ppm of oil-in-water emulsion was diluted with distilled water until the required concentration was reached.

3.5 Absorption of Oil by Kapok Fiber from Oil-in-Water Emulsions

0.4 g of modified kapok fiber was added into 100 mL of oil-in-water emulsion. The mixture was stirred using a magnetic mixer at room temperature and stirring speed of 400 rpm for 4 hours. The UV-Vis spectrophotometer was applied to determine the absorbance of oil-in-water emulsion at 1-hour time interval. After the optimized contact time (2 hours) was determined, the immersion time of modified kapok fiber in the emulsion was shortened to 2 hours. After that, the kapok fiber was filtered and the absorbance of sorbate was measured using the UV-Vis spectrophotometer.

The removal percentage of oil is determined using the equation below,

$$\mathrm{Removal\ percentage} = \frac{A_f - A_i}{A_i} \times 100\ \% \qquad \text{Eq. 1}$$

where A_i is initial highest absorbance of sorbate and A_f is final highest absorbance of sorbate.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Effect of Contact Time

Absorption of oil from oil-in-water emulsion by modified kapok fiber with HCl solution was studied by varying the HCl concentrations (0% - 2.0%) used while maintaining the treatment temperature and treatment time at 70 °C and 50 minutes. For each HCl concentration, the contact time of modified kapok fiber was studied. The trend of this study was illustrated in Figure 4.1. It was observed that for kapok fibers treated with 0% and 0.5% HCl solution, the oil removal percentage increased as the time increases. It indicated that both of the kapok fibers were still unsaturated, thus, drawing oil in continuously. Meanwhile, for kapok fibers treated with 1.0% and 1.5% HCl solution, the oil removal percentage was found to decrease after 3 hours. These findings were in accordance with the fact that the structure of kapok fiber is loose. Therefore, when the kapok fiber is saturated with oil, it tends to release the oil back to the environment easily (Wang and Wang, 2018). Hence, the optimum contact time for kapok fibers treated with 1.0% and 1.5% HCl solution were 3 hours. Similar trend also went with kapok fiber treated with 2.0% HCl solution, however, its optimum contact time (2 hours) was the shortest. This was due to the effect of HCl concentration which will be discussed in subtopic 4.2.

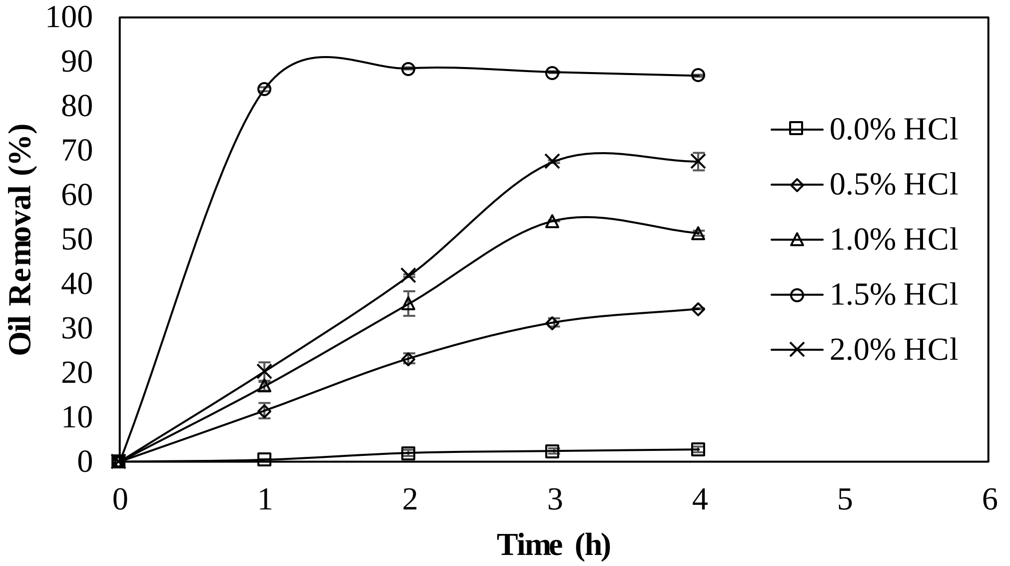

Figure 4.1 Oil Removal Percentage from Oil-in-Water Emulsion by Kapok Fiber Treated at Different HCl Concentrations versus Contact Time

4.2 Effect of HCl Concentration

Effect of HCl concentration was illustrated in Figure 4.1. The oil removal efficiency was found to increase with increasing of HCl concentration from 0% to 1.5%. Kapok fiber contains pectin, natural oils and waxy particles on its external surface, thus, creating a smooth surface. With HCl treatment, these substance can be removed, improving the roughness of kapok fiber surface. Due to the rougher surface and hydrophobicity of kapok fiber, the surface energy decreases. In addition, the crystalline packing order in kapok fiber is enhanced after being treated with HCl solution. This allows the chemical substances to penetrate the kapok fiber more easily. With lower surface energy and easier chemical penetration, the oil absorption capacity increases (Wang et al., 2012). However, when the HCl concentration increased further from 1.5% to 2.0%, the oil removal efficiency dropped. This is due to high HCl concentration can damage the structure of kapok fibre, affecting its performance in absorbing oil (Wang et al., 2012). Hence, the optimum HCl concentration in this study was 1.5%.

4.3 Effect of Treatment Temperature

The relationship between treatment temperature and oil removal percentage was studied by varying the treatment temperatures of kapok fiber with HCl solutions, ranging from 50 to 90 °C. The HCl concentration and treatment time were kept at 1.5% and 50 minutes. The results of this study were illustrated in Figure 4.2. It could be seen that when the treatment temperature increased from 50 to 70 °C, the oil removal percentage increased. This is because at high treatment temperature, the development of rough surface on kapok fiber is boosted. Therefore, it rises the oil affinity of kapok fiber (Wang et al., 2012). Nevertheless, when the temperature increased beyond 70 °C, the oil absorbency decreased. During the HCl treatment under high temperature, the partial hemicellulose and cellulose are eliminated from kapok fiber, thus, shortening the length of the kapok fiber. In addition, the damaged hollow structure of kapok fiber under high temperature also lowers its ability to absorb oil (Wang et al., 2012). Thus, the optimized treatment temperature for kapok fiber was 70 °C.

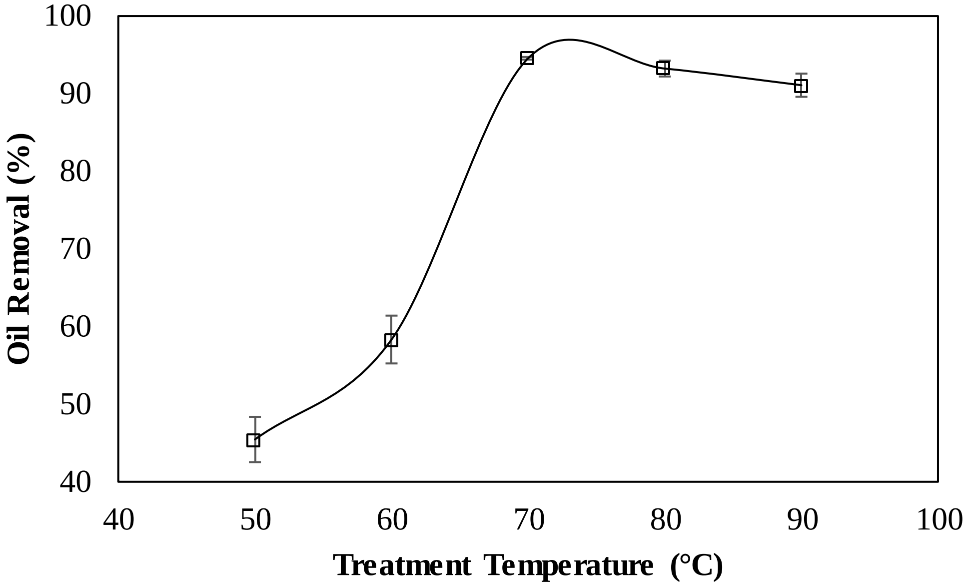

Figure 4.2 Oil Removal Percentage from Oil-in-Water Emulsion versus Treatment Temperature

4.4 Effect of Treatment Time

The effect of treatment time on oil absorption of kapok fiber was investigated by manipulating the treatment time ranging from 20 to 60 minutes and maintaining the HCl concentration and treatment temperature at 1.5% and 70 °C respectively. The relationship between the treatment time and oil absorption percentage was illustrated in Figure 4.3. It was observed that the increment of treatment time increased the oil removal of kapok fiber due to the formation of roughness on the surface of kapok fiber. However, when the treatment time went beyond 50 minutes, the oil removal percentage declined because of the destroyed structure of kapok fiber after being treated with HCl solution for too long time (Wang et al., 2012). Thus, the treatment time for kapok fiber is suggested to be 50 minutes for this study. Under optimized HCl concentration, treatment temperature and treatment time, 94.52% of oil removal efficiency was achieve at initial oil concentration of 5000 ppm.

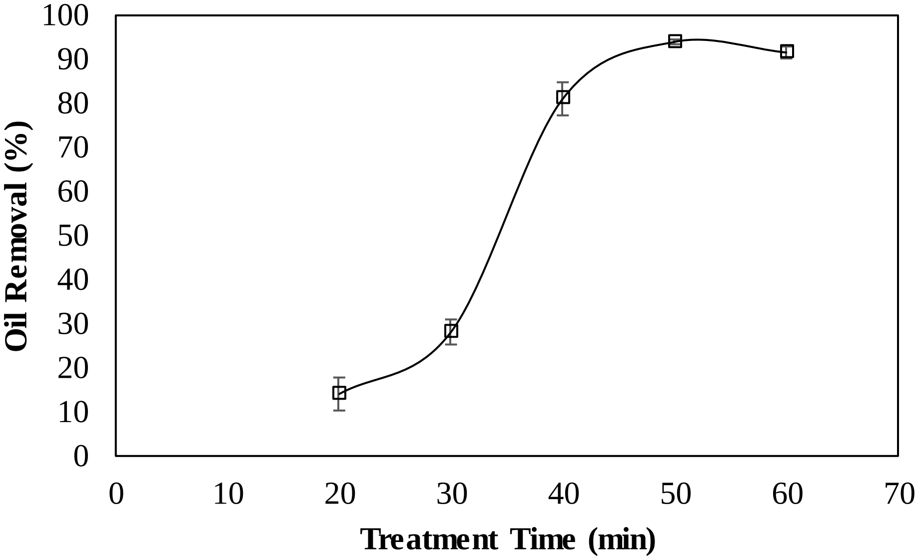

Figure 4.3 Oil Removal Percentage from Oil-in-Water Emulsion versus Treatment Time

4.5 Effect of Initial Oil Concentration of Absorbate

The oil absorption capacity of kapok fiber treated with HCl solution at HCl concentration of 1.5%, treatment temperature of 70 °C and treatment time of 50 minutes was tested with different initial oil concentrations (1000 – 5000 ppm) to study the effect of initial oil concentration of absorbate on the performance of modified kapok fiber. The trend of this study was illustrated in Figure 4.4. It could be clearly seen that the increasing of initial oil concentration slightly decreased the oil removal efficiency of kapok fiber. This was consistent with the research conducted by Okiel et al. (2011) which stated that at higher initial oil concentration, larger amount of oil was absorbed by the sorbent, thus, reducing the available surface area to absorb oil.