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MODIFICATION OF COCONUT COIR AS ADSORBENT IN OIL SPILL REMOVAL

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By

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

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Chairman : Professor Nor Azah Yusof, PhD
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Water pollution resulting from oil spill on water surface tends to have adverse effects on the environment and living organisms. Thus, researchers give a great deal of attention in rapid oil removal by using various types of adsorbent materials. Coconut coir, an agricultural waste, was chemically modified using esterification reaction by fatty acid chloride (oleoyl chloride and octanoyl chloride) and by graft copolymerization by using polymethyl acrylate (PMA) for oil adsorption purposes. The use of 10% (v/v) concentration of oleoyl chloride and octanoyl chloride were conclude to partial hydrophobically modified the coir surface via refluxing in DMAc/LiCl solvent system with present of 1% of NBS catalyst for 4 h at temperature range 70 - 90˚C. Besides, the PMA-g-coir was successfully prepared via refluxing of coir with 0.09 mol of methyl acrylate monomer for 2 h and at 70˚C. The maximum percentage of grafting (Pg) obtained is 136.32% respectively. The modified coir obtained was also characterized by FTIR, CHNSO analysis, FESEM, TGA, and contact angle measurement. From studies, it can be conclude oleoyl chloride is suitable fatty acid type compared to oleic acid. Besides, DMAc/LiCl is the suitable solvent system used in this study. The FTIR spectra showed that the coir was successfully esterified according to the existence of new peak corresponding to C=O carbonyl group of ester bond for long chain acyl group. Besides, the narrower peak of O-H corresponding to hydroxyl group also showed some of hydroxyl group in coir was modified. The high intensity of C-H stretching for CH_2 and CH_3 also indicates an increasing of alkyl group in esterified coir. For PMA-g-coir, the presence of C=O stretching of an ester functional group of polymethyl acrylate (PMA) give evidence of grafting. A new peak was also found which indicate C-H stretching in the PMA molecule. From CHNSO elemental analysis, it can be seen clearly that the percentage of C, H and O increasing upon the modification. This is due to the addition of long chain acyl group for coir-oleoate and coir-octanoate, and also due to polymethyl acrylate bond in PMA-g-coir. Thermal studied give the results that esterified coir had low thermal stability compared to raw coir due to the substitution of some hydroxyl group with acyl group which breaks the hydrogen bond in the polymer backbone. However, PMA-g-coir showed higher thermal

stability compared to raw coir which indicated that grafting process improved the thermal stability of coir.

The ability of coir-oleoate, coir-octanoate and PMA-g-coir to adsorb oil on the surface of water was investigated. Although PMA-g-coir showed good hydrophobic character in instrumental analysis, it showed very poor oil adsorption capacity in all parameter studied. In effect of adsorbent dosage studied, it can be seen that 0.6 g coir-oleoate was able to remove 100% of 10 mL oil with adsorption capacity 12.27 g/g compared to 10 g/g for both raw coir and coir-octanoate, while only 5.5 g/g for PMA-g-coir respectively at the same amount of adsorbent. The adsorption capacity of raw coir, coir-oleoate and coir-octanoate increase as the oil concentration increased until achieved equilibrium. In contact time study, the adsorption process seemed to occur rapidly during first 2 minutes and start to constant which showed the adsorbents displayed a fast adsorption property towards oil. The isotherm study indicated that the oil adsorption fitted well to Langmuir model rather than Freundlich model. Kinetic study showed that the oil adsorption data of raw coir, coir-oleoate, coir-octanoate, and PMA-g-coir fit well with pseudo-second-order kinetic model with correlation coefficient R^2>0.99 and the experimental adsorption capacities is close to the calculated one. Water adsorption study conducted showed that modified coir adsorb water less than the raw coir both in water only and also in water-oil mixture. It showed that the new functional group present give hydrophobic properties to the coir. PMA-g-coir undoubtedly showed high hydrophobic property since it only adsorbs 20% of water in water and water-oil sample. In water-oil sample, all the adsorbents showed decreasing in water adsorption compared to in water sample. The study therefore suggests that esterified coconut coir can serve as a potential biomaterial for the adsorption of spilled oil during operational failures.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untk ijazah Sarjana Sains

PENGUBAHSUAIAN SABUT KELAPA SEBAGAI PENJERAP SEMULAJADI DI DALAM MASALAH TUMPAHAN MINYAK

Oleh

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Pencemaran air yang disebabkan oleh tumpahan minyak di permukaan air cenderung untuk mempunyai kesan buruk terhadap alam sekitar dan organisma hidup. Oleh itu, para penyelidik telah memberi perhatian yang serius dalam penyelidikan berkaitan penyingkiran minyak dengan menggunakan pelbagai jenis bahan penjerap minyak. Sabut kelapa, merupakan sisa pertanian, telah di ubah suai secara kimia menggunakan tindak balas pengesteran asid lemak klorida (oleoil klorida dan oktanoil klorida) dan tindak balas pengkopolimeran cangkuk menggunakan metil akrilat (PMA) untuk tujuan penjerapan minyak. Penggunaan 10% (v/v) kepekatan oleoil klorida dan oktanoil klorida telah berjaya mengubah permukaan sabut menjadi lebih hidrofobik melalui proses refluks di dalam pelarut DMAc/LiCl dan menggunakan 1% (m/v) NBS sebagai pemangkin dengan mengambil masa selama 4 jam pada suhu di antara 70 - 90˚ C. Selain itu, PMA-g-coir telah berjaya disediakan melalui kaedah refluks antara tindak balas sabut dengan 0.09 mol cangkuk metil akrilat selama 2 jam dan pada suhu 70˚C. Peratusan maksimum cantuman (Pg) yang diperolehi adalah 136.32%. Sabut yang telah diubahsuai telah di analisis menggunakan kaedah FTIR, analisis CHNSO, FESEM, TGA, dan pengukuran sudut sentuh. Daripada kajian awal, ia dapat disimpulkan bahawa oleoil klorida merupakan jenis asid lemak yang lebih sesuai berbanding asid oleik. Selain itu, DMAc/LiCl adalah sistem pelarut yang paling sesuai digunakan dalam kajian ini. Berdasarkan keputusan daripada spektrum FTIR, ia menunjukkan bahawa pengesteran sabut telah berjaya dengan kewujudan puncak baru yang mewakili C=O kumpulan karbonil daripada ester untuk kumpulan asid rantai panjang. Selain itu, dengan berkurangnya keluasan spektum kumpulan hidroksil O-H juga menunjukkan beberapa kumpulan hidroksil dalam sabut telah diubah suai. Intensiti yang tinggi pada puncak spektra CH_2 dan CH_3 menunjukkan semakin banyak kumpulan alkil dalam sabut ester. Untuk PMA-g-coir, intensiti yang tinggi bagi spektrum C=O mewakili kumpulan berfungsi ester polymethyl akrilat (PMA) membuktikan percantuman telah berjaya. Puncak baru di 828 cm^{-1} juga menunjukkan kumpulan alkil C-H yang terdapat dalam molekul PMA itu. Dari CHNSO analisis unsur, ia boleh dilihat dengan jelas bahawa peratusan C, H dan O semakin meningkat selepas pengubahsuaian di lakukan. Ini disebabkan oleh penambahan kumpulan asil rantai panjang untuk coir-oleoat dan

coir-oktanoat, dan juga disebabkan oleh ikatan polimethil akrilat PMA-g-coir. Kajian kestabilan terma menunjukkan bahawa sabut ester mempunyai kestabilan haba yang rendah berbanding sabut mentah kerana penggantian beberapa kumpulan hidroksil dengan kumpulan asil yang memecah ikatan hidrogen dalam tulang belakang polimer. Walau bagaimanapun, PMA-g-coir menunjukkan kestabilan haba yang lebih tinggi berbanding dengan sabut mentah yang menunjukkan bahawa proses cantuman meningkatkan kestabilan terma sabut.

Keupayaan coir-oleoat, coir-oktanoat dan PMA-g-coir untuk menjerap minyak di permukaan air telah dikaji. Walaupun PMA-g-coir menunjukkan karakter hidrofobik yang sangat baik dalam analisis instrumen, ia menunjukkan kapasiti penjerapan minyak yang sangat rendah dalam semua parameter yang dikaji. Dalam kajian kesan dos bahan penjerap, ia boleh dilihat bahawa 0.6 g coir-oleoate dapat menyingkirkan 100% daripada 10 mL minyak dengan kapasiti penjerapan 12 g/g berbanding 10 g/g untuk kedua-dua sabut mentah dan coir-oktanoat, manakala hanya 5.5 g/g untuk PMA-g-coir pada jumlah dos yang sama. Kapasiti penjerapan sabut mentah, coir-oleoat dan coir-oktanoat meningkat selari dengan peningkatan kepekatan minyak sehingga keseimbangan tercapai. Untuk PMA-g-coir, keputusan yang diperolehi tidak konsisten dan memberikan kapasiti penjerapan yang sangat rendah. Dalam kajian kesan daripada masa penjerapan, ia menunjukkan proses penjerapan berlaku dengan sangat cepat dalam 2 minit pertama dan mula malar pada minit seterusnya. Ia menunjukkan semua penjerap berupaya menjerap minyak dengan sangat cepat. Kajian isoterma menunjukkan penjerapan minyak mematuhi dengan baik model Langmuir berbanding model Freundlich. Kajian kinetik pula menunjukkan bahawa data penjerapan minyak bagi sabut mentah, coir-oleoate, coir-oktanoate dan PMA-g-coir mematuhi pseudo-kedua untuk model kinetik dengan pekali korelasi $R^2 > 0.99$ dan kapasiti penjerapan bagi eksperimen berhampiran dengan yang dikira. Kajian penjerapan air yang dijalankan menunjukkan bahawa sabut diubahsuai menjerap air kurang daripada sabut mentah di dalam kedua – dua keadaan iaitu di dalam air sahaja dan juga di dalam campuran air-minyak. Ia menunjukkan bahawa kehadiran kumpulan berfungsi baru daripada proses pengubahsuaian memberikan ciri-ciri hidrofobik untuk sabut tersebut. PMA-g-coir menunjukkan sifat hidrofobik yang tinggi kerana ia hanya menjerap 20% air dalam sampel air dan air-minyak. Dalam sampel air-minyak, semua penjerap menunjukkan penurunan dalam penjerapan air berbanding dalam sampel air sahaja. Oleh itu, kajian ini menunjukkan bahawa sabut kelapa ester boleh berfungsi sebagai biomaterial yang berpotensi untuk menjerap minyak yang tumpah semasa kegagalan operasi.

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

Coir-oleoate	Coconut coir modified with oleoyl chloride
Coir-octanoate	Coconut coir modified with octanoyl chloride
DCP	dichlorophenol
DMAc/LiCl	dimethylacetamide and lithium chloride mixture solvent
EV	exfoliated vermiculite
h	hour
MA	methyl acrylate
min	minutes
mL	mililiter
NBS	n-bromosuccinimide
PMA-g-coir	poly(methylacrylate) grafted coconut coir
PP	polypropylene
PS	polystyrene
WPG	weight percent gain
%(m/v)	concentration of a substance in solution expressed as ratio of the mass of the substance to the total volume of the solution multiplied by 100%.
%(v/v)	concentration of a substance in solution expressed as ratio of the volume of the substance to the total volume of the solution multiplied by 100%

CHAPTER 1

INTRODUCTION

1.1 Problem statement

Oil spill has become one of the major problems all around the world starting from thousands of years ago. Recently, a lot of oil spill problems are reported which involved established and developed countries (Lim and Huang, 2006). Oil spill accidents can be due to the human mistakes such as war and illegal dumping or also can be caused by the oil drilling operations, accidents involving oil tankers, runoffs from offshore oil explorations, and spills from tanker loading and unloading operations (Chapman *et al.*, 2007). The spill of oil on the surface of water gives adverse effect not only towards environment and marine ecosystem, but also in human life. Thus, researchers give a great deal of attention in rapid oil removal and many commercial methods have been developed for the same purpose. Numbers of methods are applicable and widely used including boom, skimmer, surfactant, solidifier and adsorbent.

Crude oil and petroleum based oil products are persistently poisons and have a bad impact directly or indirectly towards marine organism as well as human. These injuries may occur at organism, population or community level (Aguilera *et al.*, 2010). Usually, hydrocarbon from oil can enter the marine food chain and then be passed from prey to predator. The toxicity of oil spill is depending on the concentration of the light aromatic components in the oil and the duration of exposure to these components. For example, light crude oil and light refined products such as petrol or kerosene contain relatively high proportions of low molecular weight aromatic compounds that can cause acute toxic effects.

Generally, oil spill can affect animals and plants in two ways whether from the oil itself or from the response or cleanup operations. Spilled oil can harm living things because its chemical constituents are poisonous. This can affect organisms both from internal exposure to oil through ingestion or inhalation and from external exposure through skin and eye irritation. Oil can also smother some small species of fish or invertebrates and coat feathers and fur, reducing birds' and mammals' ability to maintain their body temperatures (http://response.restoration.noaa.gov retrieved 9th April 2014).

Besides that, oil spills can cause serious damage to fishery resources through physical contamination, toxic effects on stock and by disrupting business activities. The nature and extent of the impact on seafood production depends on the characteristics of the spilled oil, the circumstances of the incident and the type of fishing activity or business affected.

1.2 Background of study

One of the most efficient and favourable method in remediating the oil spill is sorption by adsorbents. It is proven to be the best method for oil spills cleaned up because it allows the collection and complete removal of oil from the oil spill area. Besides, the addition of adsorbent will cause the liquid oil change to semi – solid phase which then can be easily remove from the water surface (Lim and Huang, 2006).

Activated carbon has undoubtedly been the most popular and widely used adsorbent in wastewater treatment throughout the world. Charcoal, the forerunner of modern activated carbon has been recognized as the oldest adsorbent known in wastewater treatment due to high adsorption capacity and porous structure. Activated carbon is produced by a process consisting of raw material dehydration and carbonization followed by activation (Bhatnagar and Sillanpää, 2010). However, it also has disadvantage due to the fact that emulsified oil can blind its pore spaces during operation and the high cost activated carbon limited its application in large scale oil spill removal (Alther, 1995). Recently, materials with super-oleophilic properties have attracted considerable interest in the field of oil-water separation. Carbon nanotubes (Gui *et al.*, 2010), mesh films (Feng *et al.*, 2004) and graphene (Bi *et al.*, 2012) have all been used for separating oil from water. However, in spite of abundant uses of these types of adsorbents, these materials have limitations for practical applications such as high costs, complex preparation processes, and difficulties in fabrication.

The usage of non-commercial biomaterials has a great potential as oil adsorbent nowadays because of their high sorption capacity, low cost, biocompatibility, non – toxic, and biodegradability as an alternative to replace activated carbon. The use of cheap and eco-friendly adsorbent which might have good potential in removing an oil spill from surface of water are worth to explore since these materials are easily available. The possibilities for cleaning oil pollution by adsorbents on the basis of fibers, polymer, and wood, however, have not been well investigated. Good remediation of oil- polluted water can be achieved with wood-fibrous adsorbents, and in particular with fibrous and lignin-containing materials due to their high sorption capacity towards oil, oil products, and heavy metals. These fibrous materials are cheap, as well as being waste agricultural product with high sorption ability. Agricultural wastes which have been used for this purpose are sugarcane bagasse, cotton, straw, corn corb, peat moss, kapok fiber, banana trunk, natural wool, and etc.

Chitosan is also widely applied in water and wastewater treatment as effective adsorbent material. It has characteristics of coagulants and flocculants, high cationic charge density, long polymer chains, bridging of aggregates and precipitation that make it an effective coagulant and flocculant for removal of contaminants (Renault *et al.*, 2008). It has been reported from previous study that chitosan has high adsorption affinity towards oil as studied by (Ahmad *et al.*, 2005). Compared to bentonite and activated carbon, chitosan showed the best removal of residual oil from Palm oil mill effluent (POME) with 99% of removal. Sokker *et al.*, (2011) studied the modified chitosan based polyacrylamide (PAM) prepared by radiation induced graft polymerization towards the adsorption of crude oil with removal efficiency of 2.3 g/g.

Rajakovic *et al.*, (2007) reported that the natural organic adsorbent which are natural wool fiber (NWF) and recycled – wool – based nonwoven material (RNWM) showed maximal efficiency and maximal oil adsorptivity compared to inorganic adsorbent which are sepiolite and bentonite. Based on studies by Abdullah *et al.*, (2010) and Lim and Huang, (2006), they reported that kapok (fibrous cottonlike substance that grows around the seeds of the ceiba tree) has higher oil adsorption capacity compared to polypropylene and it remains stable after more than three cycles of used.

Nevertheless, biopolymers themselves have limited application as oil adsorbent due to their hygroscopic property. The hydroxyl groups in each glucose molecules from cellulose and hemicelluloses offer the possibility to react with water molecules and form hydrogen bonds which cause them to sink and decrease it buoyancy. High water uptake is associated with the low hydrophobicity or water repelling ability of the adsorbents that will reduce the effectiveness of adsorbent microporous structure to adsorb oil due to high water uptake (Likon *et al*., 2013). In response, many studies have been conducted on various surface modification to improve the oil removal efficiency and hydrophobicity of natural adsorbent by mean of alkalization (Abdullah *et al.*, 2010), acetylation (Adebajo and Frost, 2004), and esterification (Banerjee *et al.*, 2006). Besides that, introduction of coupling agent, latex coating, silylation and end peroxide treatment on fiber also can enhance the hydrophobicity of the fiber (Sreekala and Thomas, 2003).

The introduction of hydrophobic functional groups into the backbone of lignocellulose structures will significantly decrease their hydrophilicity as reported by previous researcher (Ramadevi *et al.*, 2015). The example of oil sorption mechanism by esterified coir is showed in Figure 1.1. In this present study, two types of chemical modification which are acylation and graft-copolymerization were studied to hydrophobically modified lignocellulose materials. Acylation reaction will introduced long chain hydrocarbon into lignocellulose backbone by esterification reaction whereas graft-copolymerization will introduced long chain polymer which covalently attached to the lignocellulose to form copolymer.

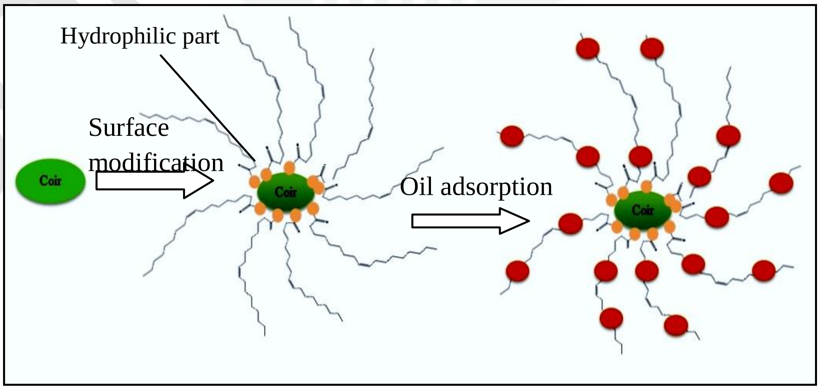

Figure 1.1: Propose oil adsorption process by esterified coir

1.3 Objectives of study

The general objective of this study was to develop an oil adsorbent material from agricultural waste which is coconut coir. The specific objectives of this study were

1) To modify the coconut coir by esterification reaction using fatty acid chloride and graft-copolymerization using methyl acrylate.
2) To characterize the raw and modified coir.
3) To compare the adsorption capacity of raw coir, coir-oleoate, coir-octanoate and PMA-g-coir adsorbents towards oil adsorption.

1.4 Significant of study

The contribution of this study would be of interest to develop oil adsorbent from agricultural waste product with high adsorption capacity, low cost and have biodegradable property. The findings of this study showed that partial esterification of coconut coir give more hydrophobic property as well as increase its oil adsorptivity. Thus, this study will help to understand the modification reaction involved lignocelluloses materials and its effect towards oil adsorption properties.

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