

POLYACRYLAMIDE HYDROGEL GRAFTED ONTO POLYETHYLENE
TEREPHTHALATE TEXTILE VIA ULTRAVIOLET LIGHT-EMITTING DIODE
PHOTOGRAFTING FOR OIL/WATER SEPARATION

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Special dedication to:

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Also to:

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ABSTRACT

This study was invented to provide a cheaper alternative filter material for oil/water separation application. Polyethylene terephthalate (PET) textiles with diameter of 4.5 cm were chemically grafted with a thin layer of polyacrylamide (PAAm) hydrogel via UV LED photopolymerisation system. From the grammages that were tested, 85 gsm PET was the most favourable to be used as a matrix. Based on the weight loss data the alkali treatment was optimised under the following condition *i.e.* 2 hours of treatment with 10 wt % of NaOH at 60 °C. For the grafting process, the effects of UV curing time (5-30 minutes), positioning of filter paper as a spacer (M1 representing the filter paper at the bottom side only and M2 representing the filter papers at both top and bottom sides) as well as technique of grafting (immersion and dipping) were also taken into the account. The obtained samples were characterised using the basic characteristics such as the degree of grafting (DG) and Fourier transformed infrared spectroscopy (FTIR). The DG values for immersed samples were significantly higher (190 % for M1 and 160 % for M2) than dipped samples (90 % for M1 and 60 % for M2). The obtained samples were also characterised in terms of surface morphology by field emission scanning electron microscopy (FESEM), oil fouling, pure water permeability and oil/water permeability test. The results indicated that, the oil/water separation performance of the hydrogel-grafted filter materials (PAAm-g-HPET) were strongly influenced by the DG of grafted PAAm hydrogel. However, the immersion grafting technique was found not suitable to be used for commercialisation purposes because of the low water permeability due to hydrogel grafting inside pores as evidenced by FESEM images. The dipping grafting method with the positioning of M2 was selected to be the best method to deal with filtration in oil/water separation. Different UV curing time influenced the oil fouling behaviour of filter samples. Data concluded that 20 minutes of curing was the optimum time for hydrogel grafting. Wettability data indicated that the filter materials after undergoing alkali treatment as well as after being grafted with PAAm hydrogel changed from hydrophobic to hydrophilic. To some extent, this innovation has shown in the near future as promising device for oil/water separation.

ABSTRAK

Kajian ini telah dijalankan bagi menyediakan alternatif bahan penapis yang lebih murah dalam aplikasi pemisahan air/minyak. Tekstil polietilena tereftalat (PET) dengan diameter 4.5 cm telah dicantumkan secara kimia dengan lapisan hidrogel poliakrilamida (PAAm) yang nipis melalui sistem cahaya UV LED. Antara semua jumlah berat tekstil PET yang telah diuji, tekstil PET dengan jumlah berat 85 gsm adalah yang terbaik untuk digunakan sebagai matrik. Berdasarkan data penurunan berat, keadaan rawatan alkali yang optimum adalah; pada masa 2 jam dengan kepekatan NaOH sebanyak 10 wt% pada suhu 60 °C. Sementara itu, bagi proses pencantuman pula, kesan terhadap masa pematangan UV (5-30 minit), kedudukan kertas turas sebagai pemisah; M1 mewakili kertas turas pada sebelah bawah sahaja dan M2 mewakili kertas turas pada kedua-dua belah bahagian atas dan bawah serta teknik pencantuman (rendaman dan celupan) turut diambil kira. Sampel yang diperoleh kemudiannya dinilai dengan menggunakan ciri-ciri asas seperti darjah cantuman (DG) dan spektroskopi jelmaan Fourier infra-merah (FTIR). Nilai DG bagi sampel rendaman adalah lebih tinggi (190% untuk M1 dan 160% untuk M2) berbanding sampel yang dicelup (90% untuk M1 dan 60% untuk M2). Seterusnya, sampel yang diperoleh ini juga turut dicirikan dari segi morfologi permukaan menggunakan medan pelepasan elektron mikroskop pengimbas (FESEM), ujian kotoran minyak, kebolehtelapan air tulen, kebolehtelapan minyak dan ujian penolakan minyak. Keputusan menunjukkan bahawa prestasi bahan penapis yang dihasilkan melalui pencantuman hidrogel dalam pemisahan minyak/air sangat dipengaruhi oleh darjah cantuman PAAm hidrogel. Walau bagaimanapun, teknik pencantuman rendaman tidak sesuai digunakan pada peringkat komersil kerana kadar kebolehtelapan air rendah disebabkan oleh pencantuman hidrogel dalam pori-pori seperti yang ditunjukkan oleh gambar FESEM. Oleh itu, teknik pencantuman secara celupan dengan kedudukan kertas turas M2 telah dipilih untuk menjadi kaedah terbaik dan sangat sesuai untuk digunakan dalam penapisan bagi pemisah air/minyak. Selain itu, masa pematangan UV yang berbeza memberi impak ke atas sifat kotoran minyak terhadap sampel penapis. Setakat ini, tempoh pematangan selama 20 minit adalah masa yang optimum untuk hidrogel dicantumkan. Data yang diperolehi daripada kebolehbasaan menunjukkan bahan penapis selepas menjalani rawatan alkali serta dicantumkan dengan hidrogel berubah menjadi hidrofilik. Adalah dijangkakan, inovasi ini mempunyai kegunaan yang cerah sebagai bahan penapis yang baik untuk pemisahan minyak/air pada masa hadapan.

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LIST OF SYMBOLS AND ABBREVIATIONS

%	-	Percent
>	-	More than
°C	-	Celsius degree
µm	-	Micrometre
cm	-	Centimetre
-g-	-	Grafted
gsm	-	gram per metre square
mL	-	millimetre
3D	-	Three dimensional
AA	-	Acrylic acid
AAm	-	Acrylamide
AMPS	-	2-acrylamido-2-methyl-1-propansulfonic acid
AAS	-	Atomic absorption spectrometer
BP	-	Benzophenone
C _F	-	Concentration feed
C _P	-	Concentration permeates
CAs	-	Contact Angle
COD	-	Chemical oxygen demand
DG	-	Degree of grafting
FESEM-	-	Field Emission Scanning Electron Microscopy
FO	-	Forward osmosis
FTIR	-	Fourier Transform Infrared Spectroscopy
LED	-	Light-emitting diode
LCST	-	Lower critical solution temperature
MBAAm-	-	<i>N,N'</i> -methylenebisacrylamide
MMC	-	Monolithic macroporous carbon

MW	-	Molecular weight
NaOH	-	Sodium hydroxide
NC	-	Nanocomposites
OMMT-		Organo-montmorillonite
PAAm	-	Polyacrylamide
PDMS	-	Polydimethylsiloxane
PDMAAAm-		Poly(<i>N,N</i> -dimethylaminopropyl acrylamide)
PEG	-	Poly(ethylene glycol)
PET	-	Polyethylene terephthalate
PEGDA-		Poly(ethylene glycol)diacrylate
PES	-	Polyethersulfone
PHEMA-		poly (2-hydroxyethylmethacrylate)
PI	-	Photoinitiator
PP	-	Polypropylene
PTFE	-	Polytetrafluoroethylene
PU	-	Polyurethane
PVA	-	Poly(vinyl alcohol)
PVDF	-	Polyvinylidene fluoride
RO	-	Reverse osmosis
SP	-	Smart Polymer
UV	-	Ultraviolet
W_1	-	Initial weight of PET textiles
W_2	-	After modification weight of PET textiles
W_s	-	Weight of hydrogel in swollen state
W_d	-	Weight of hydrogel in dry state
w/v	-	Weight per volume
wt	-	Weight

CHAPTER 1

INTRODUCTION

1.1 Background

Nowadays, tremendous attention have been paid towards development of functional hydrogels (Kumar *et al.*, 2007; Yang *et al.*, 2011; Wandera *et al.*, 2010; Chirani *et al.*, 2015). Many researchers are interested in exploring the potential application of hydrogel in various fields such as drug delivery, tissues engineering, biomedical, artificial tissue (Ahmed 2013) and recently in bioseparation field e.g. oil/water separation (Xue *et al.*, 2011; Wu *et al.*, 2012; Adrus, 2012; Liang *et al.*, 2012; Xue *et al.*, 2013; Yuan *et al.*, 2015), protein mixture (Gunavadhi *et al.*, 2012; Wang *et al.*, 2013) and many more.

Hydrogels are highly hydrophilic polymeric matrices that built up of three-dimensional network (Adrus, 2012). Hydrogels are also unique because they have self-cleaning (Wang *et al.*, 2011) and oil-fouling resistance (Sagle & Freeman 2009) properties. In addition, due to their hydrophilicity, hydrogels have the ability to retain high water content and minimize the foulant contact such as adsorption of protein (Charles *et al.*, 2009), adhesion of cells or bacteria (Kunz *et al.*, 1999) as well as oil-repellent property (Yoshida and Okano 2010).

Furthermore, research that related to hydrogel is still very active due to the great attention with regard to its outstanding properties. Polyacrylamide (PAAm) hydrogel is one of the frequently used hydrogel. In the literature, PAAm hydrogel

was mainly used in electrophoresis application for protein and DNA separation (Lin *et al.*, 2004). But recently, the research on hydrogels have focused on oil/water separation field due to their oil-repelling characteristics (Kumar *et al.*, 2007 and Stuart *et al.*, 2010). According to Xue *et al.*, (2011) and Feng *et al.*, (2012), PAAm hydrogel-coated mesh shows special wettability with both underwater superoleophobic and low oil-adhesion characteristics in oil/water/solid three phase systems. This is because, it consist of rough nanostructured hydrogel coatings and micro scale porous substrate. Similarly, Gao *et al.*, (2013) also had synthesized such hydrophilic and oleophobic molecules by using TiO_2 and it successfully applied for the separation crude oil/water mixtures.

In order to assess the surface wettability of hydrogels, it is highly recommended to use hydrogels as a grafted layer on the polymer substrate as compared to bulk hydrogels (Adrus and Ulbricht, 2012). Engineering polymer such as PET, polyurethane (PU), polyethersulfone (PES) and polyvinylidene fluoride (PVDF) are commonly employed as polymer substrates. In recent times, PET textiles have been broadly used in various applications by using different types of textile namely woven and nonwoven. Those include medical (Rahman and East, 2006), filter, geotextiles (East and Rahman, 1999) and many more. On top of that, PET is much cheaper, have good mechanical properties that comparable with metals, easy to handle as well as user-friendly amongst other polymeric materials.

In this work, the formation of hydrogel layers onto the polymer substrates could further be enhanced by photopolymerization using UV LED system. To the best of our knowledge, hydrogel grafting for oil/water separation using photopolymerization is limited. Most of the studies were reported to use physical grafting only. So, photografting technique was preferred in this study. In recent years, the UV-initiated grafting or copolymerization is progressively proposed for an effective surface modification as it offers unique ability to tune and to manipulate surface properties without damaging the bulk materials (Praschak *et al.*, 2000; Bahners *et al.*, 2004). Therefore, the aim of this work was to graft PAAm hydrogels onto PET textiles surfaces (which was direct activated with photoinitiator) via UV LED photografting and to characterize the grafted textiles (PAAm-g-HPET) from

degree of grafting (DG), Fourier transformed infrared spectroscopy (FTIR), field emission scanning electron microscopy (FESEM), surface wettability by contact angle, pure water permeability test and oil/water separation measurement. The hydrogel grafted PET textiles were envisioned to have oil-repelling properties due to the synergistic of combining hydrophilic hydrogel onto PET textiles surfaces. Thus, this innovation would have bright vision in the near future as effective materials for oil/water separation.

1.2 Problem Statement

To date, oily wastewater, polluted ocean water and frequent oil spill accident have becoming a main worldwide problem. According to the previous studies (Benfer *et al.*, 2001; Inagaki *et al.*, 2002; Huang & Lim, 2006), various approaches have been used to overcome these problems such as removal oil by kapok fibre, adsorption of oil by using activated carbon, separation by gravity and separation by ceramic membrane. Although those materials exhibited high adsorption capacity up to 99%, but there are limitations occur for a large scale fabrication of such adsorber materials and for practical application. This ascribed to the high prices of the materials, complex fabrication procedures, low stability and flexibility, difficult practical condition, lead to the oil fouling as well as poor selectivity and recyclability (Guvendiren *et al.*, 2009). In response to the problems stated above, it was therefore, worthwhile to investigate the simple and necessary way to overcome the limitation of previous innovation.

Previously, there were several studies reported that hydrogels including PAAm were usually coated onto stainless steels mesh and successfully employed for oil/water separation (Xue *et al.*, 2011). However, stainless steel mesh did not have a functional group to be grafted with hydrogel. As a result, it directed the hydrogels only attached by physical coating. This event would lead to the short lifetime and low stability. The use of engineering PET as a substrate could be good alternative as PET has also outstanding mechanical properties similar to metal. Two years later, a superhydrophobic and superoleophilic material was developed by Xue *et al.*, (2013).

Sol-gel was coated onto PET textiles. Since the obtained material is designed to be superhydrophobic and superoleophilic, the probability for pores of the textiles to be clogged with oil is very high. As a result, post-treatment is needed which directed in higher cost. Moreover, in 2014, Yuan and co-workers have developed polysulfone membrane clicked with poly (ethylene glycol) (PEG) of high density and uniformity for oil/water emulsion purification. Although the membrane shows superior oil/water emulsion separation performance, but due to its complicated fabrication, this study is not feasible to be scale up.

Although PET was widely used in numerous applications such as used as a filter device, bottles and apparels, it has limitations with regard to their surface properties. This is because PET is less hydrophilic in nature. Therefore pre-treatment is a must process to modify its surfaces. Here, the non-woven PET textiles were grafted with a thin layer of PAAm hydrogel via photografting approached for oil/water separation. PET textiles were chosen as substrate for this research because it was much cheaper, easy to handle and environmentally friendly as compared to other polymeric materials, stainless steels and ceramics.

Additionally, the curing of hydrogels was mostly focused on conventional ultraviolet-A (UVA) system. However, as the conventional UVA lamp has high energy consumption and takes time to warming up (as the emitting lamps for UVA curing are typically from mercury sources), thus UV LED was used instead. The development of UV LED light source for hydrogels curing is promising technology that can be used to replace conventional UVA system (Ayub *et al.*, 2017) . To the best of our knowledge, UV LED has not yet been reported as a source to irradiate or cure the hydrogels.

1.3 Objectives of the Research

The ultimate aim of this study was to produce highly efficient filters for oil/water separation where they were cheaper, easier to fabricate as well as environmentally friendly. The sub objectives could be further divided into:

- i. To study the effect of alkali treatment on the hydrophilicity of PET textiles with various grammage (17-120 gsm), different concentrations of NaOH (4 and 10 wt%), times of treatment (2, 4 and 8 hours) and temperatures of treatment (30 and 60 °C).
- ii. To synthesize the PAAm hydrogel grafted onto PET textiles surfaces (PAAm-g-HPET) with different times of irradiation (5-30 minutes) via UV LED photopolymerization, method of grafting (immersion and dipping) and also different positioning of filter paper (M1 and M2)
- iii. To characterize the PAAm-g-HPET samples via DG, FTIR, oil fouling test, water permeability, FESEM, surface wettability and the performance of PAAm-g-HPET samples towards oil/water separation.

1.4 Scope of the Research

In general, this project was divided into three tasks. The first task started with alkali treatment of PET textiles. The purpose of this process was to improve i) the hydrophilicity of PET as it is hydrophobic in nature, ii) performance of PET textiles and iii) to eliminate the dirt as well as other contamination. The concentration of NaOH, time of treatment and temperature were varied in order to determine the optimum conditions for alkali treatment towards the PET textiles.

Meanwhile, the second task of this research involved the study regarding the photografting process of PAAm-g-HPET via UV LED system. Primarily, both surfaces of pure and alkali-treated PET textiles were activated using type II photoinitiator (benzophenone, BP). At first, PAAm hydrogel was grafted onto PET

textiles without using any spacers. Later, the same process was deliberated using filter paper as a spacer with different positioning. Moreover, the photografting was carried out either via immersion or dipping method. These two methods were studied to investigate the effect of grafting onto the PET textiles as well as the performance of the samples towards oil/water separation. The samples were exposed to different times of curing in order to find the best UV time for grafting. Then, the grafted PET textile samples were soaked in the distilled water to wash out the residual chemicals and unreacted monomer before being dried in the oven.

Finally, all the designated samples were characterized using the basic polymer characterization process such as DG and FTIR. In addition, FESEM, oil fouling test, pure water permeability, and surface wettability using contact angle as well as oil/water separation measurement were also conducted to study the ability of PAAm-g-HPET samples to separate oil and water.

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