



UNIVERSITI PUTRA MALAYSIA

***SYNTHESIS AND CHARACTERIZATION OF POLYETHER SULFONE/
POLYETHYLENE GLYCOL / MONTMORILLONITE NANO MIXEDMATRIX
MEMBRANE FOR REMOVAL OF PHOSPHATE FROM WATER***

ALI BADERLDIN ABDELMILK ALANSARI

FS 2017 27



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MATRIX MEMBRANE FOR REMOVAL OF PHOSPHATE FROM WATER**

By

ALI BADERLDIN ABDELMILK ALANSARI

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

May 2017

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the Degree of Master of Science

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May 2017

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Eutrophication is one of the most problematic water pollution, caused by the high level of nutrients in water, mainly phosphate, which is due to the fertilizers runoff from the agricultural areas, as well as the untreated disposal of domestic and industrial wastewater into the environments. Besides, consuming high concentration of phosphate from drinking water can lead to various types of health diseases. Considering the adverse effects of phosphate pollution to human and environment, scientists and researchers had done numerous studies using different methods to remove phosphate from water, such as adsorption, extraction, flotation, precipitation, ion exchange, electro dialysis, membrane filtration and electrocoagulation.

Phosphate removal using membranes based on polyether sulfone (PES), with different ratios of organically modified montmorillonite (OMMT) nanoparticles and polyethylene glycol (PEG), prepared by phase inversion method was investigated. The hydrophilicity of prepared PES membranes was studied by water content and porosity measurements. FTIR and FESEM-EDX was used to study the chemical composition as well as to ensure the phosphate absorption by the membranes. Thermal properties of the membranes were investigated by TGA and DSC. While the morphology of the membrane investigated by FESEM. Performance of prepared membranes was determined in terms of the water flux and removal by changing conditions of pressure range 1 to 3 bar and pH form 2 to 10 as well as OMMT nanoparticles concentration 1 to 7 wt.%. It was observed that increase in pressure and OMMT nanoparticles loading is inversely proportional to the removal, and directly proportional to the water flux. Eleven different membranes, containing different ratios of PES, OMMT nanoparticles and PEG were sampled in order to prepare optimum membrane. At 1 bar pressure and 50 ppm phosphate concentration the neat PES membrane successfully achieved rejection of phosphate up to 97.6 % and water flux up to 2.26 L/m². h whereas the optimum membrane achieved rejection of phosphate up to 94.70%, water flux up to

49.2 L/m². h, and the optimum pH was pH 4. The experimental results revealed that the PES/OMMT/PEG could have a promising performance for the phosphate removal in wastewater treatment technology.



Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Master Sains

**SINTESIS DAN PENCIRIAN MEMBRAN CAMPURAN NANO
POLIETERSULFON/POLIETILENA GLIKOL/MONTMORILONIT
DIUBAH SUAI SECARA ORGANIK BAGI PENGGUNAAN BERPOTENSI
DALAM PENYINGKIRAN FOSFAT DARI AIR**

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Eutrofikasi adalah salah satu punca pencemaran air yang paling bermasalah, oleh kerana kandungan nutrient yang tinggi, terutamanya fosfat, dimana ia disebabkan oleh tumpahan baja daripada kawasan pertanian, dan juga pembuangan sisa air tidak terawat daripada pihak industri dan tempatan ke alam sekitar. Tambahan lagi, pengambilan fosfat berkepekatan tinggi daripada air minuman boleh menjurus kepada pelbagai jenis penyakit pada kesihatan. Memandangkan kesan buruk pencemaran fosfat terhadap manusia dan alam sekitar, ahli-ahli sains dan para penyelidik telah menjalankan banyak kajian menggunakan kaedah yang berbeza untuk menyingkirkan fosfat dari air, seperti penjerapan, pengekstrakan, pengapungan, pemendakkan, pertukaran ion, dialisis elektro, penapisan membran dan pembekuan elektro.

Penyingkiran fosfat menggunakan membran berasaskan Polietersulfon (PES), dengan nisbah yang berbeza daripada partikel nano montmorillonite terubasuai dengan bahan organik (OMMT) dan Polietilena glikol (PEG), yang disediakan dengan kaedah fasa penyongsangan telah dikaji. Kehidrofilikan membran PES yang disediakan telah dikaji menggunakan ukuran keliangan dan kandungan air. FTIR dan FESEM-EDX telah digunakan untuk mengkaji komposisi kimia dan juga untuk memastikan penyerapan fosfat oleh membran tersebut. Sifat haba membran telah dikaji menggunakan TGA dan DSC. Sementara itu morfologi membran telah dikaji menggunakan FESEM. Prestasi membran yang disediakan telah ditentukan dari segi penolakan dan fluks air dengan menukar tekanan dan keadaan pH serta kandungan partikel nano OMMT. Ia dapat diperhatikan bahawa peningkatan tekanan dan kandungan partikel nano OMMT adalah berkadar songsang dengan penolakan, dan berkadar terus dengan fluks air. Sebelas membran yang berbeza, yang mengandungi nisbah PES yang berbeza, partikel nano OMMT dan PEG telah disampel untuk menyediakan membran optimal. Pada tekanan 1 bar dan kepekatan fosfat 50 ppm,

membran PES kosong berjaya mencapai penyingkiran fosfat sehingga 97.6% dan fluks air sehingga 2.26 L/ m².h, manakala membran optimal mencapai penyingkiran fosfat sehingga 94.70%, fluks air sehingga 49.2 L/m².h, dan pH optimal adalah pH 4. Keputusan eksperimen menunjukkan bahawa PES/OMMT/PEG mampu menjanjikan prestasi yang baik untuk penyingkiran fosfat dalam teknologi rawatan air sisa.



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I certify that a Thesis Examination Committee has met on 2 May 2017 to conduct the final examination of Ali Baderldin Abdelmilk Alansari on his thesis entitled "Synthesis and Characterization of Polyether Sulfone/Polyethylene Glycol/Montmorillonite Nano Mixed-Matrix Membrane for Removal of Phosphate from Water" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

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LIST OF ABBREVIATIONS

ABPBI	Poly (2, 5-benzimidazole)
AM	anion exchange membranes
ASP	Activated Sludge Process
BPA	bisphenol A
CA	cellulose acetate
CM	standard cation exchange membranes
COD	Chemical Oxygen Demand
Da	Dalton
DETA	Diethylenetriamine
DMF	N, N-dimethyl formamide
DNP	dinitrophenol
DNSA	dinitrosalicylic acid
DSC	Calorimetry Analysis
EBPR	Enhanced Biological Phosphorus Removal
EC	Electro-coagulation
EDX	dispersive X-ray analysis
EPS	Extracellular Polymeric Substances
FESEM	Field Emission Scanning Electron Microscopy
FTIR	Fourier Transform Infrared Spectroscopy
HNT	halloysite nanotube
HSA	human serum albumin
MMMs	mixed matrix membranes
MMT	Na-montmorillonite

MVA	monovalent selective anion exchange membranes
NA	Nafion
NMP	N-methyl-2-pyrrolidone
NP	nitro phenol
OLR	organic loading rates
OMMT	organically modified montmorillonite
OSP	oyster shell powder
PA	polyamide
PAN	polyacrylonitrile
PBI	Polybenzimidazole
PC	polycarbonate
PE	Polyethylene
PEEK	Polyetheretherketone
PEG	polyethylene glycol
PEI	polyetherimide
PES	Polyether sulfone
PLSN	Polyether sulfone layered silicate nanocomposites
PMMA	polymethyl methacrylate
PP	Polypropylene
PPO	polydimethyl phenylene oxide
PPSu	polyphenylsulfone
PSf	Polysulfone
PTFE	Poly(tetrafluorethylene)
PVA	Poly vinyl alcohol
PVDF	polyvinylidene fluoride

PVP	polyvinylpyrrolidone
RO	Reverse Osmosis
SED	selectrodialysis
TFNC	Thin-film nanofiltration composite
TGA	Thermogravimetric Analysis
UF	ultrafiltration
ZCB	Zirconium-Modified Chitosan



CHAPTER 1

INTRODUCTION

1.1 Background of research

Increased in human population has triggered food demand globally, and this influences productivity in agro-allied industries for food supply in meeting the demand of the populace. Hence, untreated industrial waste is released to the environment as a result of urbanization, industrialization, and agricultural activities which enhance environmental pollution and disrupt ecosystem. Polluted water when consumed may cause hormonal imbalance which depends greatly on physiochemical characteristics and quantity of pollutants discharged. Two types of water pollution were identified, the point and non-point source pollution. When comparing both types of pollution, the point source pollution is known to have fixed sources of pollutants which are emitted in large amount, while the non-point pollution consists of mobile sources of contaminants which are discharged in low quantity. The pollution from the point sources is often detected and treated easily. Meanwhile, the emission of the pollutants from the non-point sources is difficult to be traced and controlled such as discharge of untreated municipal waste water from factories and the domestic wastes like human and animal excretion, food bodies and household garbage are sources of phosphates respectively (Sharpley *et al.*, 1999).

Phosphate is among lethal pollutants which affects surface and groundwater, the phosphate pollutants spread easily within ecosystem.

Phosphorus is found as phosphate (PO_4^{3-}) in nature and present as orthophosphate, polyphosphate and organic phosphate in water. Orthophosphates are usually applied to agricultural or residential land as fertilizer, which will then be carried into surface waters with storm runoff and melting snow. In addition, phosphates can also be found in various consumer products such as water softeners, baking powders, pharmaceuticals, cured meats, toothpastes, soft drinks, processed cheeses and evaporated milk. In addition, phosphate is also found abundantly in the wastewater from laundering agents for examples shampoo and detergents (Köhler, 2006).

The three main sources of phosphorus are livestock waste, human waste and agricultural fertilizers. Phosphates can be effectively removed by the restriction of phosphates in household detergents, the installation of sewage collection and the treatment of waste water in sewage plants (Köhler, 2006). Phosphorus is typically the constraining supplements in the environment. The phosphorus cycle as shown in Figure 1-1.

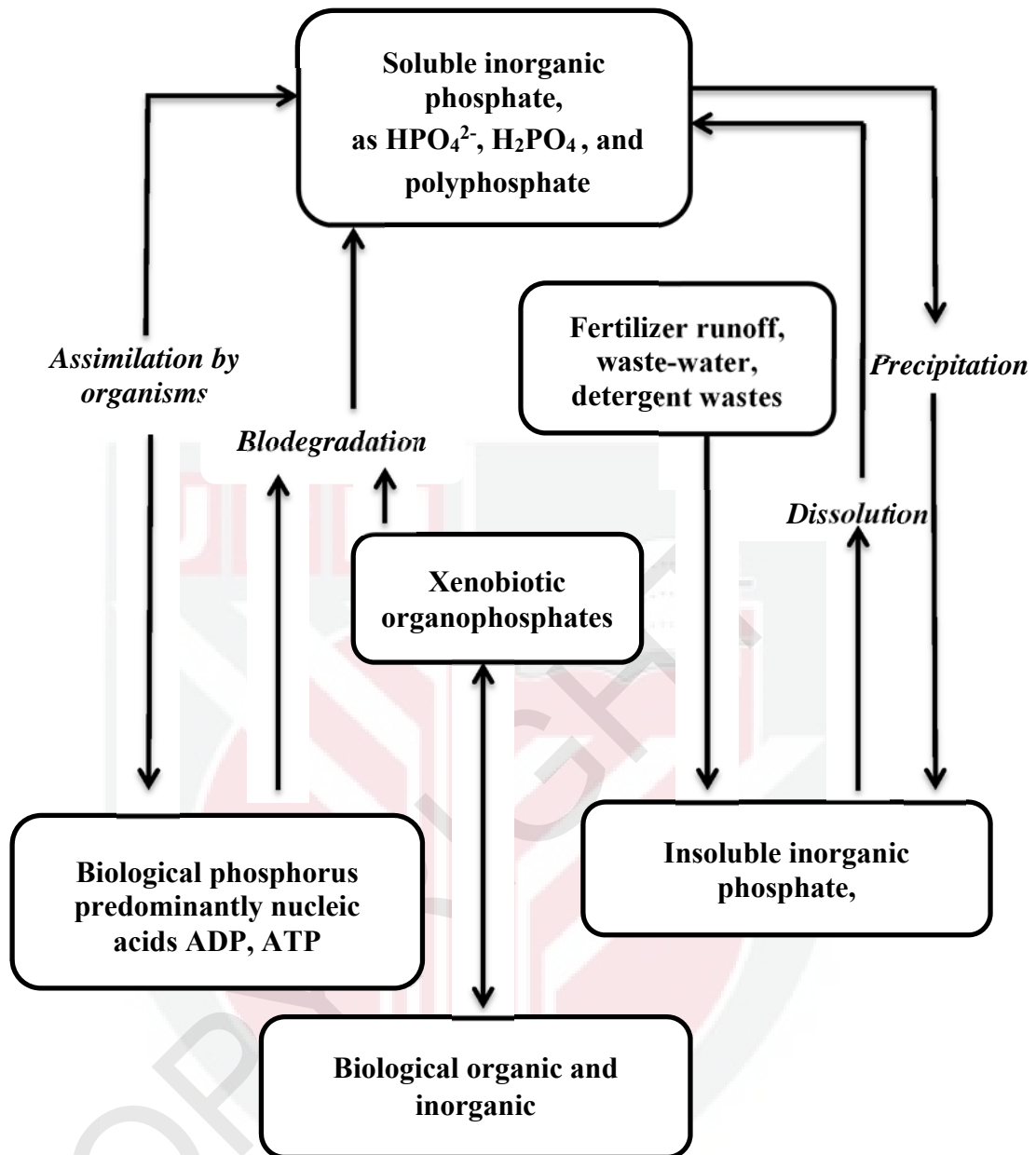


Figure 1.1 : The phosphorus cycle

The phosphorus cycle is endogenic, as there are no common stable gaseous forms of phosphorus in nature. In the geosphere, phosphorus exists in insoluble inorganic form such as hydroxyapatite (calcium salts) or iron phosphates. Following that, the soluble phosphorus from phosphate minerals and fertilizers is absorbed by the plant roots and incorporated into nucleic acid which is an essential component of genetic materials. Subsequently, the phosphorus is returned to soil by mineralization of biomass through microbial decay process and atmospheric precipitation of soluble inorganic phosphate.

Malaysian researchers were concern on issues arise from the phosphate and nitrate pollution as a results of agricultural activities, ten years ago (2006) there was a study conducted to determine the concentration of both nitrate and phosphate in rivers and wells of nine (9) states in Peninsular of Malaysia (Ikhtiar et al., 2007).

The experimental data established that the states other than Kedah, Selangor and Terengganu have river waters which are slightly contaminated phosphate. Based on Department of Environment in national water quality standard in Malaysia, the level measurement to discharge phosphate is 0.2 mg/L.

1.2 Problem statement

Water is an invaluable natural asset crucial to guarantee the satisfactory advancement of human life. However, some number of factors can lead to readily lack of accessible safe drinking water in various regions globally (van Rooijen *et al.*, 2010; Postel *et al.*, 1996). These factors can mostly be placed into one of two main groups – factors related to changes in the earth’s climate and factors related to pollution. Moreover, worldwide 80% of the used water is neither collected nor treated (Corcoran *et al.*, 2010).

It has been proposed that the changes in the earth’s climate (due to mostly combustion of fossil fuels) have a negative impact on water resources around the world (Lockwood *et al.*, 2010; Gambhir *et al.*, 2010). These changes have led to a change in chemistry water and increasing in drought periods, water temperatures and floods, an approximately 29% decrease in annual maximum daily stream-flow has been reported to be due to increased temperatures and increased evaporation (Smith *et al.*, 2011; Rieman *et al.*, 2007; Regonda *et al.*, 2005). Ground water recharge rates and depths have also been greatly affected. Floods have been shown to have negative influences on the contamination of surface and ground water and water shortage (Legates *et al.*, 2005; Lettenmaier *et al.*, 1999).

Poor water quality is a close crisis notwithstanding the general water deficiency in numerous parts in the world. Almost 2.2 million people are dying, mostly children under the age of five due to scarcity of clean water for drinking (Corcoran *et al.*, 2010).

Although the reasons for why there is water shortages in various regions of the world vary, the one thing that a number of towns and/or cities located in such regions have in common is the lack of adequate processes for generating sufficient quantities of safe drinking water. Meeting of the growing demands for safe drinking water imposes a huge requirement to develop effective and economic technologies for water treatment (Butt *et al.*, 2005).

The potential sources identified for reuse are industrial effluents, domestic waste water and rain water, numerous applications for domestic waste water reuse are toilet flushing, car washing, green landscapes irrigation, firefighting and cooling operations carried out by water (Asano *et al.*, 1996).

There is a large amount of phosphate, which is extracted from the phosphate compounds from industrial chemicals, fertilizer, and food additives. Phosphorus is also a constituent to highly degree poisonous compounds, such as military poison nerve gas and insecticides (Manahan, 2000). Phosphorus is key to the development of natural life forms, including both their metabolic and photosynthetic processes. Phosphates is essential nutrients for plant growth, hence it is widely used in the manufacture of fertilizers. The high concentration of phosphate in wastewater effluents and from agricultural runoff will result in the increase of nutrients levels in water. This will stimulate extensive phytoplankton growth and causes algal blooms, which then disrupts the normal aquatic ecosystem functioning. The huge population of algae will use up all the dissolved oxygen in the water, disturb any flora and fauna to live. Besides, the massive lumps of algae also block sunlight from penetrate through the water for the photosynthesis of aquatic plants. This is called the eutrophication phenomenon. Eutrophication destroy water quality such as high turbidity, low oxygen concentration, unpleasant odour and bad flavour (Sharpley *et al.*, 1999).

A wide range of water treatment methods have been used for wastewater treatment Including phosphate removal. They can be divided into mechanical, thermal, biological, chemical and physical processes. A selection of a certain wastewater treatment technique depends on many aspects, such as type of wastewater, cost, available space, lifetime, and the need for chemical additive(s) (Sarkar *et al.*, 2006). Examples of mechanical wastewater treatments are the use of settlers, filters and sieves. Settlers for example are used to separate components on the basis of a difference in density between the components and water (e.g. sedimentation of sand, degumming of oil). This technique is sometimes supported by adding chemical compounds (coagulants, flocculants) to increase the rate of settling. Examples of thermal treatments are evaporation and distillation techniques. A drawback of these techniques is their very high energy consumption, which is required to evaporate the water. Biological treatment is carried out by using biological aerated filters, sequencing batch reactors and rotating biological contractors. The main drawbacks of these conventional processes are: treatment process time, extensive testing before treatment, monitoring throughout treatment, difficulties in controlling the processes and high operating and capital costs.

Physical water treatment processes, which essentially consist mostly of membrane based processes, have received significantly increased interest over the last few decades.

Membrane technology differentiate mixtures of membranes labour-like filter. Hence, materials filtered were not physically, chemically or biologically affected (Humpert *et al.*, 2016). Globally, the domain of application of membrane technology became more

and more wide, this increase can be assigned to stringency of water quality that inefficiently met by conventional treatment methods which can intensify retention of coagulates and micro-organisms removal. Membrane processes has been used in drinking water treatment. Membranes are available in different forms for wastewater treatment which include biological oxidation as a hybrid system called Membrane Bioreactor (MBR) (Vincenzo, 2012). Some of the advantages of membrane is that, low-pressure membrane filtration processes highly effective in removal of suspended matters and smaller footprints such as microfiltration (MF) and ultrafiltration (UF), these are widely used in portable water purification of suspended solids and pathogens (Guo *et al.*, 2014).

The pressure difference between feed side and permeate side, produce driving force for the separation process which called transmembrane pressure (Wijmans & Baker, 1995). Membrane selectivity and capacity are essential in ensuring effectiveness of membrane. Selectivity of membrane clarifies its ability in separation of elements and compounds of a mixture also differentiate phases. Effectiveness of a membrane can be described by calculating permeate flow under specific operational conditions. Permeability is an important factor of a membrane which can be determined by calculating flow of permeate, it is described as quotient of flow and transmembrane pressure the quotient from flow and the accompanying transmembrane pressure. The main drawback of polymeric membrane is fouling. To mitigate the fouling of membrane and to make the membrane anti-fouling or fouling resistant, various inorganic metal/metal oxide or carbon-based nanoparticles have been used in polymeric membranes because of the properties such as hydrophilicity, anti-fouling, self-cleaning, photocatalytic, photodegradation, etc. Different membranes can be tailored with some or all of these properties but the overall effect should be prevention in fouling. Inorganic materials like alumina, zirconia, silica, etc. mostly used as fillers, improved the performance of membrane process by enhancing the permeate flux, increasing the salt removal and improving the thermal, chemical and mechanical stability. However, due to large size of these particles, their use was limited to MF and UF, which requires membranes of pore size 0.1 micron and 0.01 micron, respectively. With the development in the field of novel materials with at least one dimension in nano-range, the scope of application of inorganic materials was extended to NF as well as RO. Nanoparticles used for membrane modification are mostly metal and metal oxide. (Jhaveri *et al.*, 2015).

1.3 Objectives

The main objective of this research is to employ PES/PEG/OMMT matrix membrane for the removal of phosphate from water.

The specific objectives include:

- 1- To prepare and characterize Neat PES membrane
- 2- To modify neat PES membrane with OMMT and PEG
- 3- To study the removal of phosphate from the wastewater using the PES, PES-OMMT, PES-PEG and PES-PEG-OMMT nanocomposites membranes.

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