# APPLICATION OF POLYMETHACRYLATE-BASED TANGENTIAL FLOW FILTRATION SYSTEM FOR WASTE WATER TREATMENT

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# DECLARATION

I hereby declare that the content of this thesis is my own except for quotations, equations, summaries and references, which have been duly acknowledged. The developed technology is currently pending for patent approval, thus the thesis should be kept confidential and not made publicly available without the prior consent of the inventor and UMS.

7th March 2019

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# ABSTRACT

The ultimate goal of a wastewater filtration process is to remove contaminants, thus leaving only clean water as the product. However, conventional wastewater filtration process requires improvements in terms of its overall cost and effectiveness. The most commonly used filtration system is the dead end filtration system which is prone to clogging. Tangential flow filtration (TFF), a type of filtration system where the feed flows tangentially across the filter, offers longer effective filtration life span compared to dead end filtration system. This is highly attributed to the limited filter cake build-up in TFF. However, a TFF system requires a good filter membrane to function and, for that purpose, polymethacrylate monolithic (PM) membrane was chosen in this research due to its dynamic nature of controllable pore size and reactive epoxy groups for easy functionalization. Several TFF prototypes were tested whereby the final prototype had a separate compartment to encase the PM membrane thus allowing easy access and maintenance of the filter membrane. The final prototype was designed and fabricated using a 3D printer and Computer Numeric Control (CNC) machine. A number of parameters (thickness of monolith, percentage of porogen, initiator content and initial polymerization temperature) were tested for their abilities to control the pore size of the PM membrane. Percentage of porogen was opted as the pore-determining parameter due to its practicality and cost effectiveness. The combined TFF system and PM membrane was then used to filter wastewater samples (turbid water containing PM powder, lake water and river water) followed by water quality test. Among the 60%, 65% and 70% porogen-based monolithic membranes tested, the 60% porogen content monolithic membrane gave the optimum filtration performance due to its smaller pore size than the 65% and 70% porogen monolith. The turbidity level of river water sample was reduced from 17.41 Nephelometric Turbidity Unit (NTU) to 0 NTU and lake water from 9.02 NTU to 0.35 NTU. Water samples filtered using monolithic membrane of 60% porogen yielded no bacterial growth in nutrient agar even after 24 hours of incubation. However, no significant reduction or changes in the pH level of water samples before and after filtration. For total dissolved solid (TDS) analysis, a slight reduction of 68 parts per million (ppm) to 63 ppm was observed for lake water after filtration and a reduction of 36 ppm to 26 ppm was observed for river water sample. The monolithic membrane of 60% porogen content reduced the colour of lake water sample from 221 Hazen to 36 Hazen and 205 Hazen to 38 Hazen for river water sample. The combined TFF system along with the monolithic membrane was also tested for prolonged usage, and the data suggested that it is reliable for long term usage. The developed filtration system provides insight and alternative to the conventional wastewater treatment process, hence could be improved to be applied in remote areas where access to treated water is not available.

# ***ABSTRAK***


## ***PEMBANGUNAN SISTEM PENAPISAN ALIRAN TANGEN BERASASKAN POLIMETAKRILAT UNTUK RAWATAN AIR SISA.***

*Matlamat utama proses penapisan air sisa adalah untuk mengasingkan bahan cemar, sekali gus hanya meninggalkan air bersih sebagai produk. Walau bagaimanapun, proses penapisan air sisa konvensional masih memerlukan penambahbaikan dari segi kos dan keberkesanan secara menyeluruh. Sistem penapisan yang paling biasa digunakan ialah sistem penapisan buntu yang senang tersumbat. Penapisan aliran tangen (PAT), sejenis sistem penapisan di mana aliran masuk mengalir secara tangen pada seluruh penapis, menawarkan penapisan lebih berkesan jika dibandingkan dengan sistem penapisan buntu, kerana kotoran yang cenderung melekat pada penapis terus dihanyutkan semasa proses penapisan. Walau bagaimanapun, sistem PAT memerlukan membran penapis yang baik untuk berfungsi dan, dalam hal ini, membran monolit polimetakrilat (MP) telah dipilih untuk kajian ini kerana sifat dinamik saiz liang boleh dikawal dan kumpulan epoksi reaktif untuk pemfungsian mudah. Beberapa prototaip TFF telah diuji di mana prototaip terakhir mempunyai ruangan khas untuk menempatkan membran MP dengan tujuan membolehkan akses mudah dan penyelenggaraan membran penapis. Prototaip akhir direka dan dicetak menggunakan mesin pencetak 3D dan Kawalan Berangka Berkomputer (CNC). Beberapa parameter (ketebalan monolit, peratusan porogen, kandungan pemula dan suhu pempolimeran awal) telah diuji untuk keberkesanan mengawal saiz liang membran MP. Peratusan porogen telah dipilih sebagai parameter penentuan saiz liang kerana lebih praktikal dan jimat. Sistem gabungan PAT dan membran MP kemudiannya digunakan untuk menapis sampel air sisa (air keruh mengandungi serbuk MP, air tasik dan air sungai) diikuti dengan ujian kualiti air. Di antara 60%, 65% dan 70% kandungan porogen membran monolitik yang diuji, membran monolitik dengan kandungan 60% porogen memberikan hasil yang optimum kerana ianya mempunyai saiz liang paling kecil berbanding membran monolitik dengan kandungan porogen 65% dan 70%. Ia mampu mengurangkan kekeruhan sampel air sungai dari 17.41 Unit Kekeruhan Nephelometric (NTU) kepada 0 NTU dan air tasik dari 9.02 NTU hingga 0.35 NTU.*

*Sampel air yang ditapis oleh sistem PAT dengan 60% kandungan porogen membran monolitik menunjukkan tiada pertumbuhan bakteria dalam nutrien agar walaupun selepas masa inkubasi selama 24 jam. Walau bagaimanapun, tiada pengurangan atau perubahan drastik dalam pH sampel air sebelum dan selepas penapisan. Bagi jumlah pepejal terlarut (TDS), analisa menunjukkan pengurangan daripada 68.00 bahagian dalam sejuta (ppm) kepada 63.00 ppm untuk air tasik selepas penapisan dan pengurangan 36.67 ppm kepada 26.00 ppm untuk sampel air sungai. Membran monolitik 60% juga dapat mengurangkan warna sampel air tasik dengan ketara daripada 221 Hazen kepada 36 Hazen dan 205 Hazen kepada 38 Hazen untuk sampel air sungai. Sistem gabungan TFF bersama-sama dengan membran monolitik juga diuji untuk kegunaan berpanjangan dan data mencadangkan ia boleh digunakan untuk kegunaan jangka masa panjang. Sistem penapisan yang dibangunkan memberikan pandangan baru dan alternatif kepada proses rawatan air sisa konvensional dan boleh ditambahbaik untuk digunakan di kawasan terpencil dimana tiada akses kepada air bersih yang telah dirawat.*

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# LIST OF ABBREVIATIONS

| | | |
|---|---|---|
| **TFF** | - | Tangential Flow Filtration |
| **DEF** | - | Dead End Filtration |
| **EPA** | - | Environmental Protection Agency |
| **U.S.** | - | United States |
| **BOD** | - | Biological Oxygen Demand |
| **COD** | - | Chemical Oxygen Demand |
| **ppm** | - | Parts per Million |
| **E. Coli** | - | Escherichia coli |
| **DBPs** | - | Disinfection by-products |
| **GI** | - | Gastrointestinal |
| **CNC** | - | Computer Numeric Control |
| **GMA** | - | Glycidyl methacrylate |
| **EDMA** | - | Ethylene glycol dimethacrylate |
| **AIBN** | - | Azobisisobutyronitrile |
| **mm** | - | Milimeter |
| **n.d.** | - | No date |
| **Pt-co Units** | - | Platinum Cobalt Colour |
| **JKR** | - | Jabatan Kerja Raya |
| **PSI** | - | Per square inch |
| **mL** | - | Mililiter |
| **nm** | - | Nanometer |
| **WEPA** | - | Water Environment Partnership in Asia |
| **FTU** | - | Formazin Turbidity Unit |
| **ppm** | - | Parts per million |
| **UV** | - | Ultraviolet |
| **ACU** | - | Apparent Colour Unit |
| **TCU** | - | True Colour Unit |
| **°C** | - | Degree celcius |
| **TOC** | - | Total Organic Carbon |
| **DDT** | - | Dichlorodiphenyltrichloroethane |

| | | |
|---|---|---|
| **MCPA** | - | 2-methyl-4-chlorophenoxyacetic acid |
| **L** | - | Litre |
| **MOH** | - | Ministry of Health |
| **m** | - | metre |
| **CFF** | - | Cross Flow Filtration |
| **WHO** | - | World Health Organization |
| **mg/L** | - | Milligram per litre |
| **SEM** | - | Scanning electron microscope |
| **kV** | - | Kilovolt |
| **UMS** | | Universiti Malaysia Sabah |
| **PAT** | | Penapisan Aliran Tangen |
| **MP** | | Monolit Polimetakrilat |
| **rpm** | | Revolutions per minute |

# CHAPTER 1

# INTRODUCTION

## 1.1 Background of Study

Water is a finite and an important resource on earth. An essence of life, all living things on earth need water to survive and no other living things other than human uses water for so many applications. Wastewater contains different types of contaminant depending on its sources (Sophonsiri & Morgenroth, 2004). Wastewater from agricultural runoff usually contains high levels of nitrogen and phosphorus which play a major role in eutrophication (Elser, Marzolf, & Goldman, 1990; Zimmo, Van Der Steen, & Gijzen, 2004), while water from domestic wastes contains mostly organic matter. The uncontrolled disposal of domestic and industrial wastewaters into the environment causes severe pollution problems such as eutrophication or oxygen depletion in lakes and rivers, which makes wastewater treatment paramount (Cai, Park, & Li, 2013). Globally, the World Health Organization (WHO) estimates that by 2025, about 50% of the world's population will be living in water scarce-areas of which demands exceed the available supply, while currently, even with access to water, 2 billion people use drinking water source that is contaminated with fecal matter which can transmit illnesses such as dysentery, diarrhea, cholera, polio and typhoid (WHO, 2018).

Conventional wastewater treatment comprises several steps to remove different sizes and types of contaminants. However, current wastewater treatment processes still encounter overwhelming challenges. For example, the virus removal efficiency in water treatment is highly dependent on several parameters such as salt, turbidity, concentration, pH and contact time (Riley, Gerba, & Elimelech, 2011). Recently, methacrylate monolith has been successfully used as a tool for waterborne virus removal from wastewater (Rački et al., 2015). A monolith is a single piece of a highly porous material consisting of interconnected pores (Williams,2001).Polymethacrylate monolith is a highly potential material used either in separation or concentration process due to its pH resistance, ease of synthesis and flexibility in tailoring the pore size according to the target solutes (Podgornik, Smrekar, Krajnc, & Štrancar, 2013). Apart from virus removal, another challenge faced by the conventional wastewater treatment process relates to the chemical used in the disinfection process, chlorine, which can react with naturally occurring materials in drinking waters to form by-products that are detrimental to human health (Riley et al., 2011). These by-products are called disinfection by-products (DBPs), and there are more than 600 types that have been identified having a variety of detrimental effects on human's health (Richardson *et al*., 2007).

One of the most important processes in wastewater treatment is filtration. In traditional dead-end filtration, the feed flows directly into the filter, resulting in clogged filter pores over a short period of time. In tangential flow filtration (TFF), the feed flows tangentially across the filter surface enabling prolonged filtration as the filter cake is continuously being washed out during the filtration process (Van Reis & Zydney, 2001). TFF has been widely used in biotechnology to recover proteins and metabolic products from fermentation as well as to concentrate bacterial sample for further analysis (Naja, Volesky, & Schnell, 2006; Van Reis & Zydney, 2001). The TFF system used in wastewater treatment generally produce two outputs simultaneously in a single flowthrough, one being the more concentrated wastewater (retentate) and the other being clean water (permeate).

## 1.2 Problem Statement

Water crisis is a recurring problem faced by many in the developing and arid countries. With the exponential growth of human population, the demand for water is also increasing exponentially hence the reason why water reclamation is important. Filtration is one of the main processes in water reclamation and there are many types of filters existing in the market today each designed to cater for different needs. In recent years, polymethacrylate monolith is used in separation processes due to its controllable pore size and reactive epoxy groups that can be functionalized,(Barroso, Hussain, Roque, & Aguiar-Ricardo, 2013).

Currently available water treatment technology relies heavily on chlorine for microbial disinfection. This process has been known to produce disinfection byproducts that pose risks to human health (Richardson et al., 2007). People who live in rural areas where there are no access to treated water often rely on natural water sources such as river water, rain, ponds and underground water that are not treated where boiling is the only method of disinfection. The water quality at such places is usually affected by weather condition such as heavy downpour that usually brings a massive amount of suspended solids into the water bodies. Although boiling makes water biologically safe, the high level of suspended solid (especially nano particles) makes water undrinkable and unsafe for daily use. Therefore, a simple and affordable yet effective water filtration system needed to be developed.

The pore size of a polymethacrylate monolith is affected by several parameters especially the amount of reagents used for the polymerization process. Apart from that, the amount of heat produced during the polymerization process and the shape of the polymethacrylate monolith greatly affects the pore size formation within the monolith. Manipulation and understanding of these parameters are crucial in fine tuning the pore size of a polymethacrylate monolith into a desired size. In order to fabricate a polymethacrylate monolith into a desired shape, a polymerization mold that has good heat conductivity and water resistance is required, since the mold would be submerged in a water bath. Another challenge is to remove the resulting polymethacryate monolith from the mold, as it has the tendency to stick on the mold itself.

Most commonly used filtration system employs the dead end filtration system which is susceptible to clogging after a brief usage, as the feed flows directly onto the filter membrane itself. A better alternative would be the tangential flow filtration system, where the feeds flows tangentially across the filter membrane thus enabling longer effective filtration process compared to dead end filters as the filter cake is continuously washed away. A simple TFF system that could house the polymethacrylate monolith membrane needs to be developed to integrate both TFF and polymethacrylate monolith membrane into a fully functional filter. It needs to be waterproof and is able to withstand high pressure to prevent leakage during usage.

## 1.3 Hypothesis of Study

The hypotheses of this research were:

1. The polymethacrylate monolith pore size could be adjusted to retain different types of suspended contaminants in wastewater and allow effective high throughput water filtration to be conducted at a fairly low pressure.
2. The fabricated polymethacrylate-based TFF system was able to effectively house the monolithic membrane.
3. The developed TFF system could be used to filter wastewater at varying degrees of efficiency according to the pore size of the monolithic membranes.

## 1.4 Objectives

The objectives of this research were:

1. To characterise the morphology of polymethacrylate monoliths prepared at varied experimental conditions (thickness of monolith, percentage of porogen, amount of initiator and initial polymerization temperature).

2. To design and fabricate a polymethacrylate-based TFF system for wastewater filtration.

3. To test the efficiency of the TFF system for the removal of microbes and suspended particles from wastewater.

## 1.5 Significance of Research

The development of a simple TFF system combining polymethacrylate monolithic membrane to filter wastewater provides an alternative to current technology in wastewater treatment. It has the potential to eliminate the dependency on chlorine for water disinfection which is known to produce disinfection byproducts detrimental to human's health. The developed TFF system combining polymethacrylate monolithic membrane is small in size, portable and can be operated at low water pressure hence without pump. The current prototype can cater for an individual or small family needs in rural areas that lack treated water supply. The proposed system could potentially be up-scaled to process hundreds litres of untreated water. This is due to the fact that polymethacrylate monoliths have been produced successfully in larger size than the current monolith (Chan, Adam, Obeng, & Ongkudon, 2018) .

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