ELECTROSPUN NANOFIBER WATER FILTRATION MEDIA FOR REMOVING SUSPENDED SOLID

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SUPERVISOR'S DECLARATION

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ABSTRACT

Water pollution is a serious problem in Malaysia and impacts negatively on the sustainability of water resources. It reduces total water availability considerably as the cost of treating polluted waters is too high and in some instances, polluted waters are not treatable for consumption. There are several causes of water pollution such as disposal of rubbish, industrial waste, oil pollution, radioactive waste, underground storage leakages and marine that can disseminate the quantity suspended solid in water. Nowadays, this untreated water that consist of suspended solid (SS), heavy metals and chemical waste need to give an attention from responsible parties or the industries themselves.

Therefore, the main objective of the experiment which is to produce high filtration efficiency with additional nanofiber with varied electrospinning time. The project was carried out in order to compare the incorporated electrsopun nanofiber with existing water filter in terms of Suspended Solid (SS). The incorporated filters were then used to filter the collected industrial waste water samples according to European Standard EN 872, Water Quality: Determination of Suspended Solids

To reach the main objective of this experiment, there are several testing approaches in order to evaluate the efficiency of filtration. First of all, the TSS retentions was manually calculated by differences weight of filter samples after and before in weight percentage. Next, was by using calorimeter device to measure the concentration of suspended solid in the filtered samples. Then, the average diameter is analyzed using Image J after SEM process completely conducted. As a result, the presence of nylon 6 is elctrospun nanofibers significantly improved the capability of filters compared to conventional filter paper.

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ABSTRAK

Pencemeran air merupakan salah satu masalah yang serius di Malaysia, yang membawa kepada kesan negatif terhadap kelestarian sumber air. Masalah ini akan mengurangkan jumlah air sedia ada, kerana kos untuk merawat air terlalu tinggi disebabkan keadaan tertentu. Terdapat beberapa punva pencemaran air berlaku antaranya seperti pelupusan sampah, sisa industri, pencemaran minyak, sisa radioaktif, ketirisan simpanan bawah tanah dan laut yang boleh menyebarkan kuantiti pepejal terampai di dalam air. Pada masa kini, air yang tidak dirawat ini terdiri daripada pepejal terampai (SS), logam berat dan sisa kimia perlu memberi pembelaan daripada pihak yang bertanggungjawab atau industri itu sendiri.

Oleh itu, objektif utama eksperimen ini adalah untuk menghasilkan kecekapan penapisan tinggi dengan nanofiber tambahan dengan masa electrospinning yang berbeza. Projek ini telah dijalankan untuk membandingkan penapisan industri yang sedia ada dengan memperkenalkan penapis yang diaplikasikan menggunakan electrospun nanofiber sebagai bahan tambahan kepada sistem penapisan tersebut. Model penapis tersebut telah dihasilkan melalui proses eletrospinning dengan masa yang berbeza-beza. Proses penapisan pepejal terampai telah dijalankan berdasarkan European Standard EN 872, Water Quality: Determination of Suspended Solids.

Justeru, untuk mencapai objektif kajian ini, beberapa ujian telah dijalankan bagi menentukan panapis air yang lebih efisyen. Pertama adalah dengan mengira secara manual prebezaan antara sampel penapis selepas dan sebelum dalam peratusan berat. Seterusnya adalah dengan munggunakan colorimeter untuk menyukat kadar pepejal terampai yang masih ada dalam sampel yang telah ditapis. Kemudian, diameter purata dianalisis menggunakan J Image selepas proses SEM sepenuhnya dijalankan. Dengan ini, kehadiran nylon 6 nanofibers elctrospun ketara meningkatkan keupayaan penapis berbanding kertas penapis konvensional.

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LIST OF ABBEREVIATIONS

TNR = Total Non-Filterable Residue

VNR = Volatile Non-Filterable

TS = Total Suspended Solid

SS = Suspended Solid

SEM = Scanning Electron Microscope

COD = Chemical Oxygen Demand

OC = Organic content

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LIST OF SYMBOL

T = Transmittance

A = Absorbance

I_t = Transmitted Light

I_0 = Initial light intensity

ε = Molar absorptivity

C = Molar concentration

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CHAPTER 1

INTRODUCTION

1.1 BACKGROUND

The term of water crisis is frequently used by the United Nations and other world association for depiction of the present's status of world's water resources relative human demand. Basically, water is one of the main basic needs of humankind. However, nowadays water is increasingly contaminated with variety of toxic chemicals and waste. Water pollution or contamination is a worldwide issue which requires continuous evaluation and revision of water resource policy at all levels. For instance, based on surveyed in the latest national report on water quality in the United States, 44 percent of assessed stream miles, 64 percent of evaluated lake section , and 30 percent of assessed bays and estuarine square miles were categorized as polluted (Agency & Size 2004).

The water pollution is a major issue in Malaysia and effects contrarily on the sustainability of water resources. It reduces total water accessibility considerably as the expense of treating dirtied waters is too high and in a few cases, polluted waters are not treatable for consumption. In fact, the climate and this is influencing water assets. There are few causes of water pollution such as disposal of rubbish, industrial waste, oil pollution, radioactive waste, underground storage leakages and marine. In other way, industry is a huge source of water pollution, it produces pollutants that are extremely harmful to people and the environment. As we know, many industrial facilities use freshwater to carry away waste from the plant into river, lakes and oceans. The pollution released from industry to the atmosphere is ruining the quality of the environment and intensifying the water pollution problem continuously such as shown in Figure 1.1.

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Figure 1.1 : Water pollution by industrial waste

The specific contaminants leading to pollution in water include a wide spectrum of chemicals, pathogens, and physical changes such as elevated temperature and discoloration. Total suspended solid are solid materials, including organic and inorganic, that are suspended in the water. Most suspended solids are made up of inorganic materials; however bacteria and algae can also contribute to the total solids concentration. These solids incorporate anything that floating and gliding in the water from sediment, silt and sand to algae and plankton. In fact, green growth algae such as phytoplankton, are regular occurrences, especially in the ocean. Inorganic materials can easily become suspended due to runoff, erosion and resuspension from occasional water flow. However, when suspended solids exceed expected concentrations, they will give negative impact to the body water.

From my research, there is various ways to remove the suspended solids from water such as through sedimentation or filtration. The term filtration can be characterized in its easiest form as the process of removing solid particles from a fluid (liquid or gas) by driving the fluid through a porous medium through which the solid particles cannot pass (Allhands 2014). Innovation in filtration technologies is one of the advanced approaches for cleaner and better environment. In addition, necessity in filtering technology in an energy and efficient

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cost has led to gaining attention in nanostructured membrane especially for production of nanofibrous via electrospinning.

Electrospinning is the most suitable technique for production of nanofiber. As shown in Figure 1.2, this technique is depends on three fundamental parts: a high voltage supply, a capillary tube containing polymer solution/melt attached to a needle of small diameter, and a metallic collector. To create an electrically charged jet of polymer solution/melt out of the needle, a high voltage is applied between two electrodes connected to the spinning solution/melt and to the collector (normally grounded)(Homaeigohar & Elbahri 2014) .

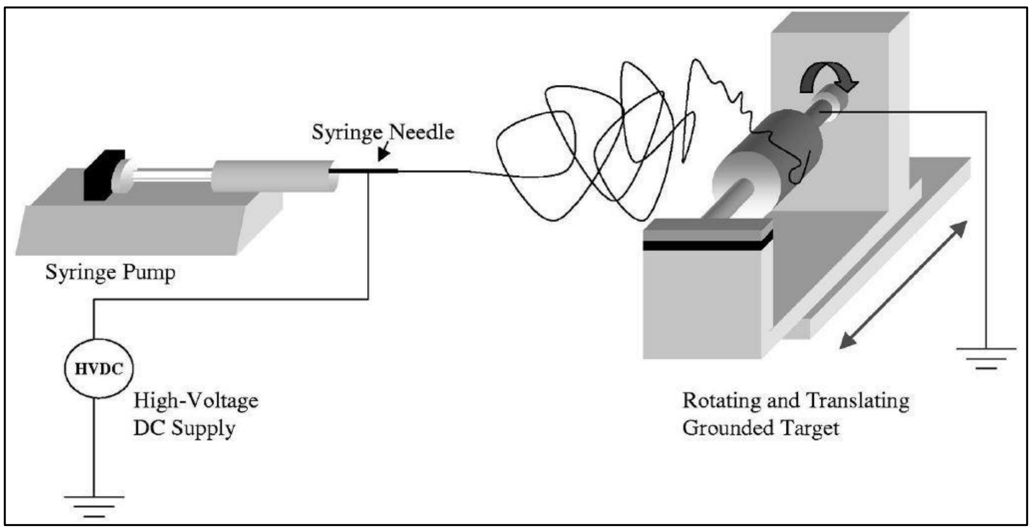

Figure 1.2: Schematic Diagram of Electrospinning (S. Homaeigohar and Elbahri 2014).

Specifically electrospun layer usually possess higher porosity (normally around 80%, however there is no maximum limit), lower base weight, larger effective surface area (up to 40 m2/g depending on the fiber diameter) and ceaselessly interconnected pores, when compared to conventional polymer and ceramic membranes (Wang et al. 2012). The unique structure in nanofibrous membranes are not only give benefits to the quality of water but also a chemical separation in liquid. Furthermore, not only the small pore size, but also flexible in surface functionalities, have large available surface area, and design of the nanofibrous membranes improve their adsorptive nature and selectivity (Homaeigohar 2011)

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1.2 PROBLEM STATEMENT

Filtration is one of the special applications of electrospinning technique that performs the dust filtering, gas and particle separations in the ambient air and aqueous media. Moreover, in order to produce fresh water that being treated by suspended solids, such as rust from piping and vessels, formation sand, and scale particles, or dissolved solids (various chemical ions). For most uses or disposal methods, these solids may need to be removed. Total Suspended Solids (TSS) is a key parameter to evaluate the water quality conditions, which is affects the light attenuation and thereafter the ocean primary production of plankton (Chen et al. 2015)

In this study, suspended solid can be separated from water stream by few methods such as by using gravity settling, hydroclone desanders, filters and centrifuges. Hence, for filtration technique, the volume of solids that can be taken care by sedimentation and desanders cannot be handling by filtering process. The electrospinning process forms nanofibers of long lengths and with diameters typically in the range of 10–500 nm(Shin et al. 2005). The three sorts of filters commonly used are media, cartridge or diatomaceous-earth filters.

For instance, one of unique properties of electrospun nanofibers is there large surface area mass ratio which makes them valuable in different applications, especially for filtration process. The high surface area, small diameter and low basis weight make for the very efficient catching of particles for better filtration capability (Nilsen 2011).

1.3 OBJECTIVES

Based on the problem statements, the current study has been performed with the following objectives:

i. To produce high efficient filtration system for capturing suspended solid in industrial waste water.
ii. To develop a new high efficient water filtration media using polymeric nanofiber.

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1.4 SCOPE OF PROJECT

The scope of this project is:

i. Produce polymeric nanofibers using electrospinning technique.
ii. Fabricate a new filtration media by incorporating layer of elctrospun nanofibers
iii. Measure suspended solid content of a water sample using spectrophotometer
iv. Morphological study of an electospun nanofiber filters membrane using scanning electron microscope (SEM).

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CHAPTER 2

LITERATURE REVIEW

2.1 ELECTROSPINNING

Electrospinning is the most commercially industry process for the production of nanofiber and rising interest is driving research and development in this field. For instance, electrospinning is a process that has been known for centuries and its quite similar to electrospraying, which is discovered by Lord Rayleigh in the late 1800's (Nilsen 2011) . In fact, advance progress has been conducted in terms of the electrospinning process and in the production of nanofibers with superior chemical and physical properties. According to (Kang & Kang 2016) electrospinning is a nano-scale fiber production method with different polymer materials. This technique of electrospinning allows simple fiber diameters control by changing the physical conditions such as applied voltage and polymer solution viscosity during the electrospun process. Basically, there are three components that is normally use for electrospinning process which is a supplier of high voltage, needle connected to a syringe and a metal collector. The voltage supplier introduces a high electrical potential between the needle and the grounded metal collector. Initially, the polymer solution forms a hemisphere at the tip of the due to surface tension. As electrical potential is applied, the hemispherical surface of the polymer solution elongates to form a Taylor cone. Thus, when the electrical potential is increases, the surface tension will overcome and cause the formation of polymer jet towards metal collector. (Cheng et al. 2016).

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2.2 APPLICATION

Electrospinning of nanofibers with the diameter that fall into nanometer has rising consideration from many researchers in recent years due to ability to produce scaffolds with nanoscale properties. In addition, the production of nanofiber, which are used for several value added applications such as medical textile, personal care, filtration, barrier, composite, insulation and energy storage. Besides that, for wide range of applications electrospinning process is widely used for example for medical to consumer products and industrial to high-tech applications for aerospace, energy storage, fuel cells, information technology, and drug delivery (Hasan et al. 2014) . According to (Agarwal et al. 2008) the possibility of large scale productions combined with the simplicity of the process makes this technique very attractive for many different applications. The different functionalities are attributed from different mechanical and chemical properties.

2.3 PARAMETERS

Working parameters are very imperative to understand because the conversion of polymer solutions onto nanofiber .Thus, there are three types of parameters that very important for the process of electrospinning such as solution parameters, process parameters and ambient parameters. The one of important role in the fiber formation during electrospinning process is the concentration of polymer solutions. As the concentration is very low, polymeric micro (Nano)-particles will be obtained. At this time, electrospray occurs instead of electrospinning owing to the low viscosity and high surface tensions of the solution (Li & Wang 2013).

Within electrospinning process, applied voltage is the one of the tribunal factor. So there are three factors that include for this part such as voltage, flowrate, collector and distance between the collector and the tip of the syringe. In addition, when the applied voltage is increases the polymer jet with greater electrostatic repulsion would discharge and, causing it to undergo higher levels of drawing stress (Wong 2010). Moreover, as a recommended a lower flow rate during process of electrospinning is needed as the polymer solution will get enough

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time for polarization. Other than that, during the electrospinning process, collectors usually acted as the conductive substrate to collect the charged fibers. Generally, Al (aluminium) foil is used as a collector but it is difficult to transfer the collected nanofibers to other substrates for various applications. For the last, ambient parameter also give impact to the fiber diameters and morphologies such as humidity and temperature. From the past research by (Mit-uppatham et al. 2004) had proven that the fiber diameter thickness is influence by raising in temperature by conducting experiment with polyamide – 6 fibers as shown in Figure 2.1

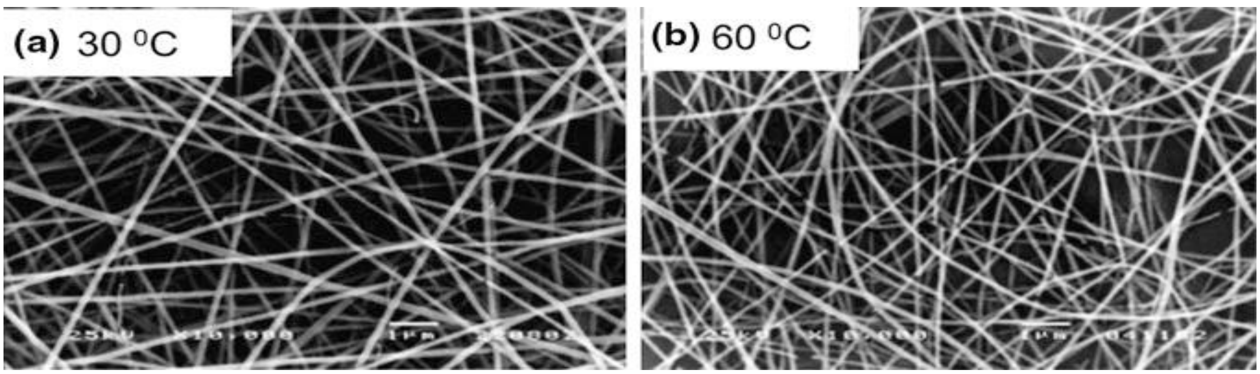

Figure 2.1 : SEM images of the electrospun PA-6-32 fibers under different temperatures. The diameters of A and B are 98 and90 nm, respectively. (Mit-uppatham, Nithitanakul, and Supaphol 2004)

2.4 ELECTROSPUN FILTRATION

Pollution from environmental discharges for example industry waste can produce high levels of suspended solid that can contaminate sediments within the surface water systems subsequently. The main purpose of the study was to assess the filtration effectiveness of three sorts of geotextile nanofiber with various water driven permeability and to compare them and sand filtration. Based on this previous study, Catherine N. Mulligan had carried out experiment of filtration in terms of removal suspended solid on the surface of water. The research is conducted by using water samples at different points close toward to the shore along St. Lawrance, Montreal to determine the range of actual turbidity values of the river. Three different types of nanofiber filter has been used which were non-woven geotextiles, gray non-woven filter and the third one was a white woven fabric filter. After that, by using

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spectrometer each portion was combined and the concentration of heavy metals in each fraction was determined. Sample test were washed with distilled water to set them up for the following step. The results of the total metal and the sequential analysis are shown in Table 2.1.

Table 2.1 : Characterization of Sediment

Element	Content (mg/kg)	CCME ISQG[a] for fresh water sediment	Selective sequential extraction, results shown as % of metal associated with each fraction compared to the total metal content					
			Soluble (%)	Exchang.[c] (%)	Carbonate (%)	Oxide (%)	Organic (%)	Residual (%)
Cu	60	35.7	21.4	5.4	5.4	10.7	32.1	25.1
Zn	130	123	1.7	2.4	14.7	35.3	16.8	29.2
Ni	90	NA[b]	<0.4	38.9	61.1	<0.6	<0.6	<0.7
Cr	200	37.3	<0.3	4.3	0.5	25.6	22.3	47.3
Pb	80	35	<1.2	28.6	14.3	28.6	<1.7	28.6
Total	560							

[a] ISQG – interim sediment quality guideline.
[b] NA – not available.
[c] exchang. – denotes exchangeable.

Filtration test was conducted by in a plastic Nalgae Company with 51cm height and 34cm an inner diameter with 46.5L maximum capacity. The filtration column it made of plexiglass and had 7.9cm in diameter and 43.5m in height such as shown in Figure 2.2. The efficiency of three different types of filters that stated before is evaluated one at time. Furthermore, as the ability of the filter to remove suspended solid on the surface of the water these three elements are intensely related to the efficiency of filtration system in terms of the reduction of turbidity. Table 2.2 shows the filter characterization.

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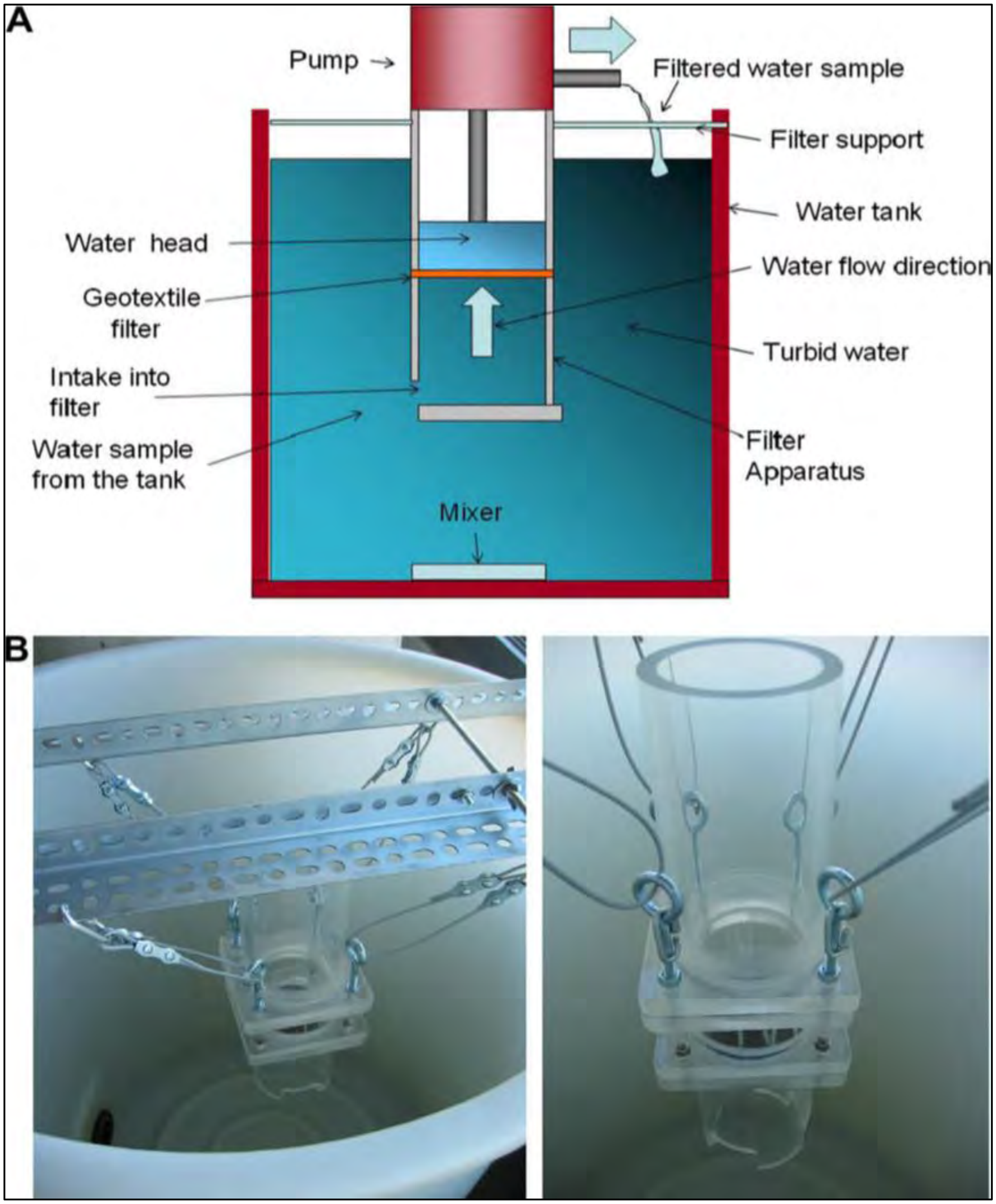

Figure 2.2 : Schematic (A) and filtration column (B) (Mulligan et al., 2009)

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Table 2.2 : Filter characterizations

Filter	Thickness (cm)	Apparent opening size (AOS) (mm)	Mass ($g\ m^{-2}$)	Permittivity (s^{-1})	Water flow rate ($cm^3\ s^{-1}$)
1	0.14	0.12	196	0.88	6.00
2	0.04	<0.035	160	0.66	1.28
3	0.05	<0.035	245	0.29	0.63

From table above, by divided the permeability by the thickness of the filters the permittivity of the thin filters was calculated and tabulated. Then, in order to determine the apparent opening size (AOS) ASTM method D4751-99a was used in this experiment. ASTM D422-63 was used as the standard technique to decide the molecule measure distribution for the sand. As a result, according to ASTM classification 38.8% medium sand 60.4% fine sand and 0.8% fines were calculated.

Filtration procedure after 10 minutes filter no 2 and 3 were saturated before filtration process. For 20L containers filled with water from three different parts of Lake Saint Louis were added with 70g, 50g and 10g of sediment were added. After the sediment is completely dissolve the container is shaken well for 15 min in order to let the larger suspended solid settle under the influence of gravity and to obtain obtain turbidities close to 120, 70, and 20 NTU for each filters. Thus, after 17 litres of water poured into the filtration tank, fine particles that suspended in the water sample were remove by filtering water through the filter media. Flow rate for filter 1 was adjusted to 10L min^{-1}. However, since flow rate for filter 1 and 2 was not possible to use higher flow rate due to backflow from the permeability these filters was fixed at 1L min^{-1}.

From this experiment, turbidity, pH, chemical oxygen demand (COD), suspended solid (SS), and loss on ignition (OC) were measured before and after filtration process. Dissolved and particulates COD was measured in order to get the total COD. Flow rate, pH, and turbidity of the water in the tank and directly after passing through the filter were measured over time

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during the experiment. Flowrate, pH, and turbidity of the water in the tank which is passing through the filter were measured over time during the experiment. Then, the amount of metals retained on the filters before and after the filtration as check for the heavy metal and suspended solid balance by using XRF (Niton). The temperature is constant as a fixed variable during the experiments since the temperature control in the laboratory (22 ± 2^0C).

For preliminary evaluation, 6 hours were taken for filter 1 and only 15 minutes for filters 2 and 3 as experiment durations. For filters 2 and 3 with low pore size were blocked in a short period of time, but filter 1 could be used in the system for 6 hours without blockage due to potential filter in SS, OC, turbidity and COD removal. In the second run of experiment, on order to reduce the turbidity and suspended solid filters 1 and 2 were used in the filter column from three levels of turbidity with conditions same as the first experiment. Lastly, for the third round run of experiment medium sand size was selected for the comparison of filter media. The sand filter with 4cm height were prepared and the sand was washed three times with tap water, air dried and then 350 g of sand was weighed and poured into the filtration column. A cotton woven mesh with suitable size was used to retain the sand and not interfere with the filtration process.

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