ELECTROSPUN NANOFIBER WATER FILTRATION MEDIA FOR REMOVING SUSPENDED SOLID

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SUPERVISOR'S DECLARATION

I have checked this report and the report can now be submitted to JK-PSM to be delivered back to supervisor and to the second examiner.

Signature : .....

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Date : .....

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.

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.

ACKNOWLEDGEMENT

In the name of Allah, the most Gracious and the most Merciful

All praises to Allah for His blessings and guidance in giving me the strength to complete this project. I would like to express my deepest appreciation to my supervisor Dr. Nurfaizey bin Abdul Hamid for giving me this opportunity to do final year project with him. He never hesitated to give me advice and guidance whenever I confronted problems. I am thankful for his patience and tolerance while supervising me in this project.

Meanwhile, I would like to express heartfelt thanks to my course mates and roommates who had been really helpful in the process of finishing my project. With their constructive comments and ideas, enable me to thrive towards my project's goals and objectives. Not to forget the PSM committee for giving me this invaluable chance to involve in PSM. Lastly, special gratitude goes to my mother, and family members who always indirectly lavish.

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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

LIST OF SYMBOL

T = Transmittance

A = Absorbance

I_t = Transmitted Light

I_0 = Initial light intensity

ε = Molar absorptivity

C = Molar concentration

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.

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

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)

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.

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).

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).

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

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

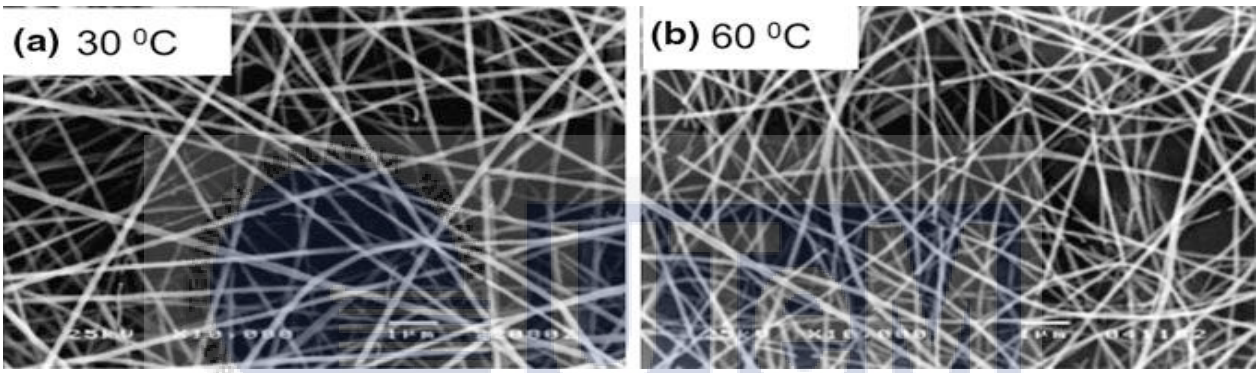


Figure 2.1 : SEM images of the electrospun PA-6-32 fibers under different temperatures. The diameters of A and B are 98 and90 nm, respectively. (Mit-uppatham, Nithitanakul, and Supaphol 2004)

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

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.

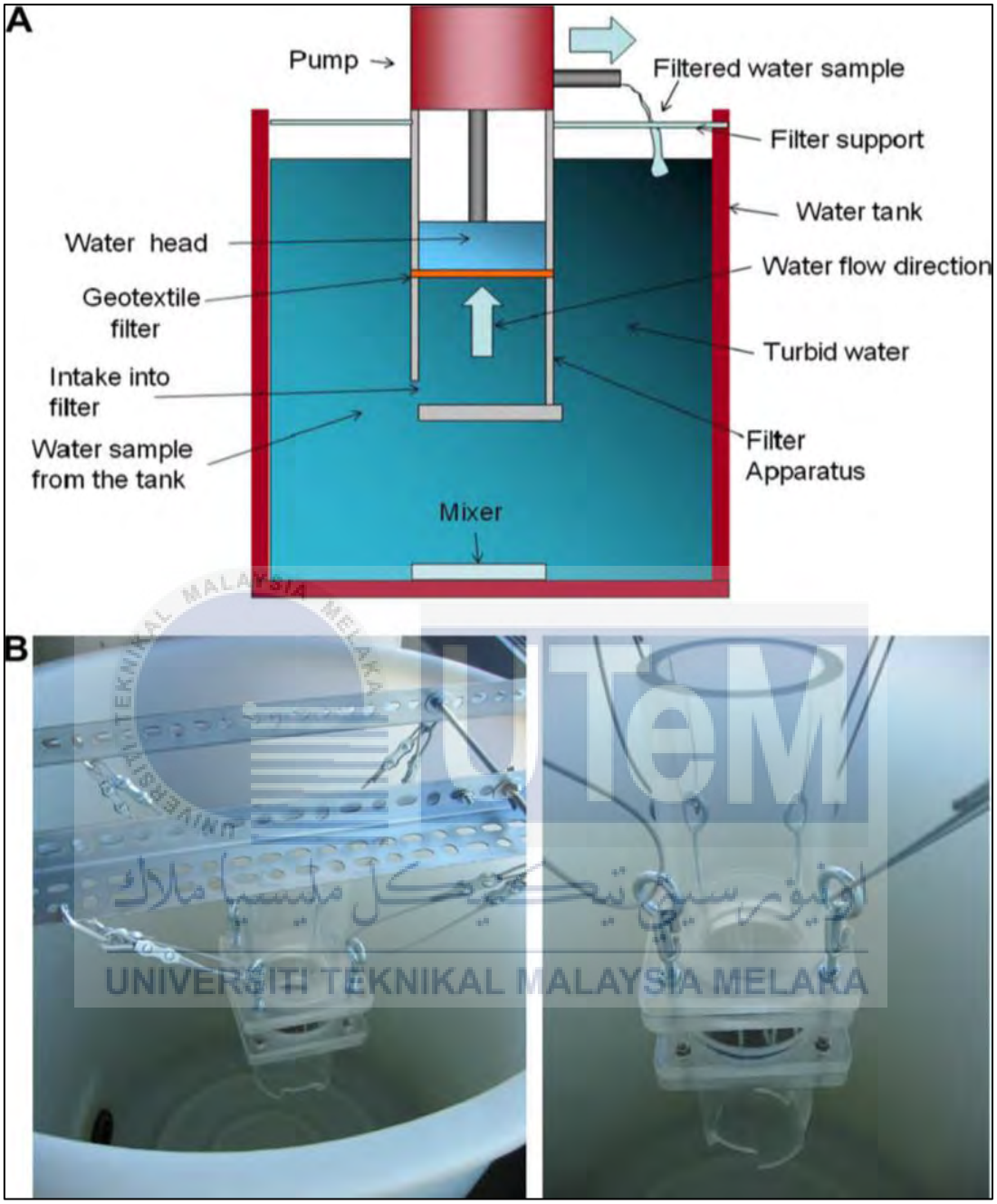


Figure 2.2 : Schematic (A) and filtration column (B) (Mulligan et al., 2009)

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

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.

Table 2.3 Filtration results (Mulligan et al., 2009).

Removal %	SS	OC	COD	TU_d	TU_F	Total heavy metal removal rate (mg m^{-2} d^{-1})[a]	SS removal rate (g m^{-2} filter d^{-1})
Filter 1							
Site 1	98.8	93.7	71.2	98.2	98.3	136	242
Site 2	93.8	87.5	65.5	93.4	93.5	387	691
Site 3	98.9	96.7	65.5	97.6	97.9	240	432
Filter 2							
Site 1	41.7	50.0	53.3	6.5	83.9	3480	6220
Site 2	62.5	33.3	45.4	38.3	82.2	426	760
Site 3	37.2	25.0	46.5	2.7	85.1	3530	6300
Sand							
Site 1	73.9	40.0	25.5	63.0	71.4	2130	3800
Site 2	88.2	90.0	63.2	84.8	86.6	1550	2760
Site 3	66.9	43.8	24.0	55.7	69.7	2320	4150

Site 1: Initial turbidity: 70 NTU.
Site 2: Initial turbidity: 20 NTU.
Site 3: Initial turbidity: 120 NTU.
TU_d is water turbidity filtration the tank, and TU_F denotes water turbidity that passed through the filter.
[a] Total heavy metal denotes the sum of Pb, Cu, Cr, Ni and Zn contents in the solids.

Based on experimental results shown in Table 2.3, sand filtration was found to be effective method among other filters in terms of removing suspended solid from water. However, when compared to filter 1, it clearly shown that filters 1 better in many ways. This is because it was able to do the filtration process more than 40 hours without clogging, whereas the sand bed showed considerable head loss after 3 hours of operation with high turbidity water. In fact, even the sand filter was more efficient than filter 1 in the short run; it would require backwashing to enable it to attain the greater than 90% removal of SS that was seen for filter 1.

2.5 SUMMARY

In view of this late reviews that carried out by Catherine N. Mulligan, the formation of electrospun of nanofiber with incorporated with filtration substrate for example fiber glass filter paper was scarce. There are a few elements that could be enhance by using the nanofiber filtration which is high penetrability, better suspended solid removal, and have the large surface area would be strong enough to sustain the water driven which applied to the filter.

CHAPTER 3

METHODOLOGY

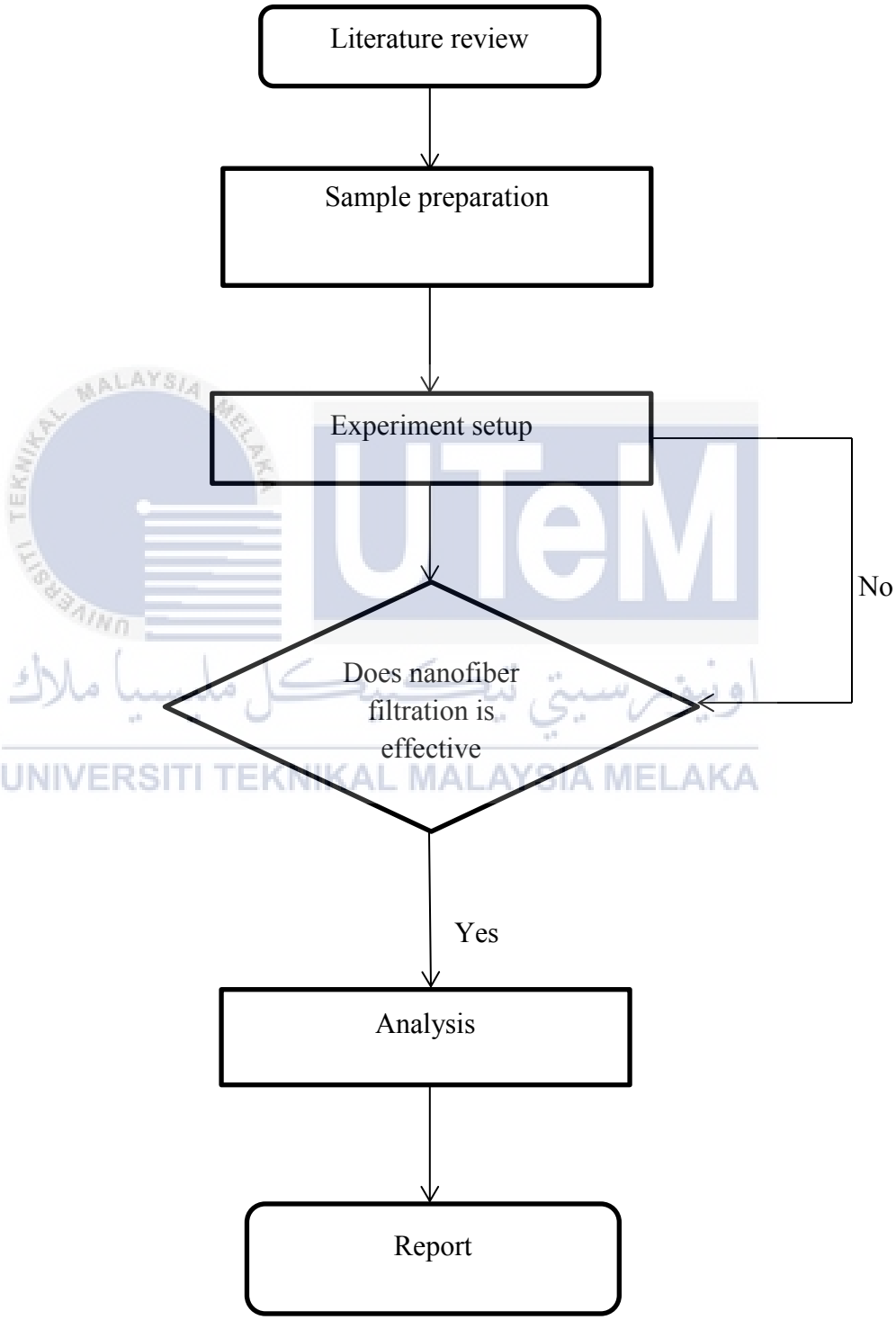
3.1 OVERVIEW OF EXPERIMENT

In this study, an experiment of electrospun of nanofiber incorporated with fiber glass paper as a substrate. For this research, nylon 6 is 6 used as the molten solution that to be electrospun. A water filtration process is set up by using several different types of apparatus such as aspirator, filtering flask, desiccator, drying oven, filter holder with 47mm diameter, graduated cylinder of 100ml, watch glass, furnace muffle and analytical balance. Scanning Electron Microscope (SEM) imaging was utilized to describe the sample's morphology.

3.1.1 EXPERIMENTAL WORK FLOW CHART

For the beginning part of this experiment, literature review of elctrospun of nanofiber to determine the theoretical framework of this study in detail. Then, after literature review was done the experiment process is began with preparation of material. The next step was a water filtration and electrospinning setup with the given apparatus. Then, after all the apparatus is completely done the electrospinning process conducted at advance material lab. The concentration of suspended solid that is trapped at the fiber glass filter is analyzed by using colorimeter. In order to get Total Non-Filterable Residue (TNR) and Volatile Non-Filterable Residue (VNR) the weight of nanofiber coated at the fiber glass filter is measured.

FLOWCHART



3.2 GANTT CHART
PSM 1

Gantt Chart for PSM 1	2016													
Project Activity	Week													
	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Project Briefing	Supervisor Briefing							Mid Term Break						
Identify Objective and Scope of Journal														
Identify Method														
Journal Research														
Summary of Journal														
Find the Experiment Apparatus														
Material Finalization														
Experiment Setup														
Result and Discussion														
Progress Report Submission														
Report writing														
Submission FYP 1														
Presentation FYP 1														

PSM 2

Gantt Chart for PSM 2	2017													
Project Activity	Week													
	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Project Planning	Supervisor Briefing							Mid Term Break						
Material Selection														
Polymer Preparation														
Electrospinning														
Sample Collection														
SEM Analysis														
Concentration Analysis														
Results and Discussion														
Progress Report Submission														
Report writing														
Submission FYP 2														
Presentation FYP 2														

3.3 ELECTROSPINNING SETUP

The electrospinning experiment will be held at the Advance Material Laboratory, Universiti Teknikal Melaka. The machine Electrospinz model ES1 produced by Electrsospinz Limited, NZ will be used to conduct this experiment. The polymer solution will flows from glass header through the silicon tube towards the tip and the spinning has been horizontal fixed in front of collector. The polymeric solution was electrospun to the grounded metal collector.

3.3.1 ELECTROSPINNING PROCESS

For this research, to produce nylon electrospun fibers Electrospinz Model ES1a laboratory scale electrospinning machine is used as shown in Figure 3.1. First of all, when the apparatus is ready to set up, the polymer solution that has been prepared is poured into the main container. After the fiber glass paper is put on the collector, 14 kV voltages input is afflicted to the polymeric solution. The collector plate is grounded as to generate voltage gradient in order to induce polymer traversing. Therefore, when the high electric force is subjected to the surface or inside the polymeric solution, a force is induced due to mutual charge repulsion that is directly opposite to the surface tension of the polymer solution. Thus, when the electric field reaches a critical value, the polymer solution will produce an electrically charged jet at the end of spinneret. Due to surface tension the solution is held at the tip of syringe. A charged jet of the solution is ejected from the tip of the 'Taylor Cone' protruding from the liquid drop of the polymer. Lastly, as the electric overcame the surface tension, the polymer will be projected towards the oppositely charged collector and formations of solid nanofibers will appear on the collector plate.

Figure 3.1 : Electrospinz Model ES1a laboratory scale electrospinning machine.

Figure 3.2 : A voltage input regulato

3.4 MATERIAL

Recently, the uses of organic material is have been widespread to be used in industries due to it benefits such as low density, easy to transport and install, resistance to corrosion and solvent. Therefore, for this project nylon 6 or called or well known as polymide 6 used as the molten solution that to be electrospun. For instance, polyamide, polyster and vinyl are the some examples of important group of organic semi-crystalline polymers. Nowadays, the polyamide (nylon) as a group of semi crystalline polymers gained attraction due to the polar structures and thermal stability. As to generate an effective filtration system, nylon 6 is the most feasible polymer which to be incorporated onto the filter media as nylon 6 fibers are tough, possessing high elasticity and also high tensile strength. Other than that, nylon 6 can absorb a lot amount of water, which is by immersion or simple exposure to high humidity. These properties of nylon 6 make it a very appropriate polymer for filtration as it can endure the hydraulic pressure imposed by the industrial waste water samples. Nylon 6 is mixed with formic acid by the mass fraction of 30 wt%. Figure 3.3 below shows the formation of Nylon 6.

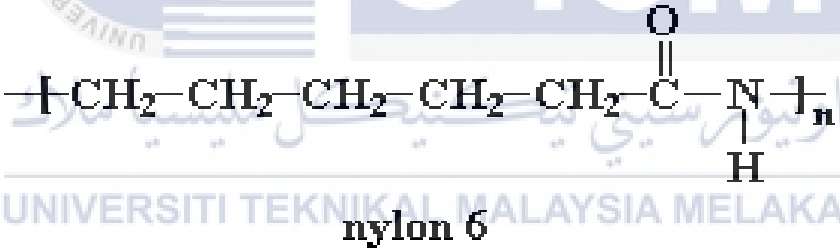


Figure 3.3 : Formation of nylon 6

3.4.1 POLYMER PREPRATION

Preparation of nylon 6 is one of the important parts in this experiment. As stated before, material used is nylon 6 (Sigma- Aldrich 181110) in formic acid (Merck 1002642500) solution. Firstly, nylon 6 is dissolving overnight at room temperature by using magnetic stirrer. After that, the weight of nylon 6 and the formic acid is measured using four significant figures to get the exact mass friction. The amount of polymer solution needed is 150 ml, thus

the weight of nylon polymer is set up for 45 ml and 105 ml for the formic acid. The merging, concentrating and dissolving process are conducted in fume hood laboratory.

3.4.2 SUBSTRATE PREPARATION

Electrospun of nanofiber is usually covered or coated onto a substrate to ensure the durability of the layered composite and the adhesion between the nanofiber and the underlying substrate. Thus, for this experiment Whatman glass microfiber filter Grade GF/C (Sigma – Aldrich WHA1822) is used as a primary filter as shown in Figure 3.4. The advantages of using this borosilicate glass fiber filter is the media can sustain the medium to fast hydraulic flow in order to achieve the flow speed which is up to 100sec/100ml. The diameter of this filter paper is 47mm, thickness of 0.26mm and pore size 1.2µm. The filter paper need to be adjusting into circular shape of 47mm diameter in order to fit in the substrate to the filter holder.

Figure 3.4 : Whattman glass microfiber filter Grade GF/C (Sigma –Aldrich WHA1822)

3.5 WATER SAMPLES PREPARATION

The industrial waste water sample was taken from a treatment plant in UTeM‟s main campus. The water sample was taken from the pre-treatment pond inside the facility as shown in Figure 3.5. For information, the water samples taken from the is second last stage of filtration process before the water is safe to be used.

Figure 3.5 : Water samples preparation form treatment plant at UTEM Main Campus

3.5 WATER FILTRATION SETUP

The water filtration apparatus was provided from Hach Company. The main purpose of this company is to ensure water quality for people around the world. So they provide the apparatus and material for water treatment such as spectrophotometer, turbidity meter, colorimeters, digital reactors, titration system and etc.

3.5.1 WATER FILTRATION APPARATUS

This water filtration testing set up consist of analytical balance, cylinder graduated 100ml, desiccator, drying oven, filter flask, filter holder, filter 47mm, furnace, rubber policeman, tongs, tweezers and watch glass. The diameter of filter is 47mm in order to fit in the substrate to the holder. The filter holder is assembly in the filtering flask such as shown in Figure 3.6 and Figure 3.7 and the tweezer is used to put a fiber filter disc in the filter holder.

Figure 3.6 : Filtering flask

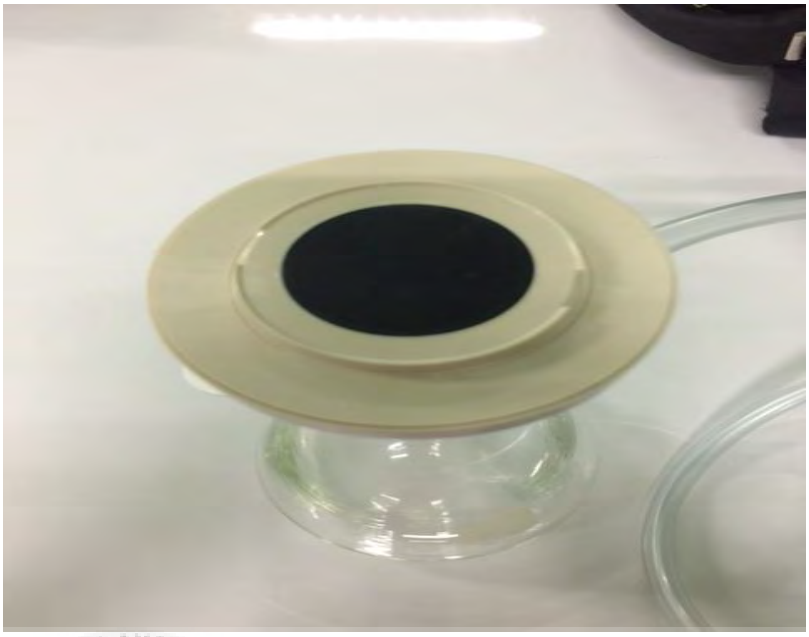


Figure 3.7 : Filter holder

Figure 3.8 : Aspirator

The aspirator as shown in Figure 3.8 was used to apply vacuum to the flask until all of the water is pulled through the filter as a recommended pressure. As an indicator to the pressure applied, the white ball is observed during this experiment. So if the ball moves fast, the pressure applied is higher.

3.6 TEST PROCEDURE

First of all, in order to increase in reliability and validity of this experiment blind test or well known as double-blind experiment is need to be done by using deionized water as a water sample. The main purpose of blind test is to ensure that experiment is not biased. Moreover, the blind test is minimum standard for any test involving subject, opinion, and failure to achieve the better results of experiment. Thus, after all the apparatus is completely set up, put the fiber filter disc by using tweezer in the filter holder such as shown in Figure 3.9 Then, the filter holder is put in the filtering flask that is assembling together. Then, use a graduated cylinder to add 100ml of deionized water to the filtering flask. Furthermore, Figure 3.10 shown if too much remaining material stays on sides or bottom lip of the filter holder, use a rubber policeman to remove the solid then use a small amount of water to pull solid down to the filter such as shown. Besides, apply vacuum to the flask until all of the water is pulled through the filter.

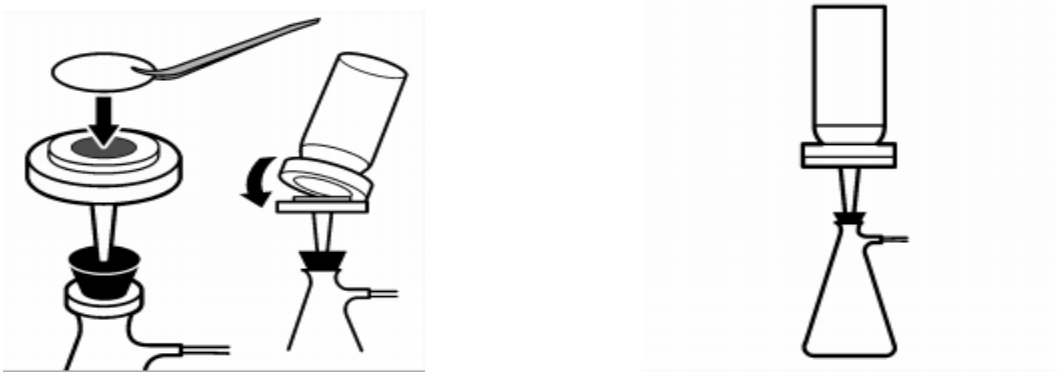


Figure 3.9 : (A) The fiber filter disc is put on the filter holder (B) Filtering holder assembly with filtering flask

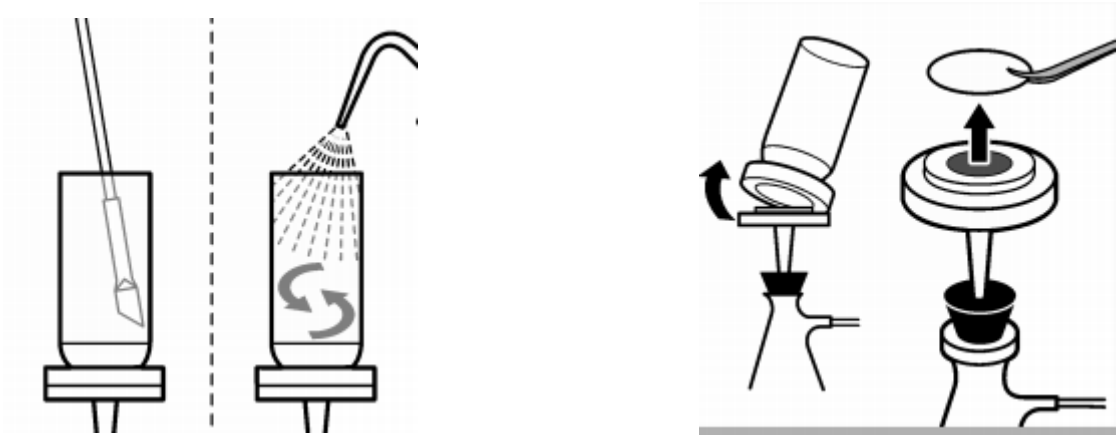


Figure 3.10 : (A) The remaining material is pull down on the fiber filter disc. (B) The filter is put away after filtering process is done

After all the water is completely through the filter, release the vacuum system carefully and remove the fiber filter disc from the filter holder. At that point, use a tweezer to put the fiber filter in a watch glass. The watch glass with the fiber filter disc is put into the preheated drying over at 103-105^{0} C (217-221 ^{0}F) for 1 hour to keep the filter dry. Then, use metal tongs to remove the watch glass with the fiber filter disc from a drying oven and put in desiccator and cover the desiccator immediately. In this experiment, if the volatile non-filterable solid is measure the fiber filter disc is put in a muffle furnace to remove all of the volatile material. Desiccator with desiccants such as shown in Figure 3.11 is used in order to get accurate weight of fiber filter disc. This is because, desiccant are drying agents that are used to ensure that any water vapor present is absorbed before reaching the reagents. Hence, to prevent the high pressure from the hot air inside, make sure the temperature of the watch glass is decrease a little before get seal. Then, let the fiber filter disc and watch glass decrease to room temperature.

Figure 3.11 : Desiccator

Lastly, after the watch glass has reach room temperature remove the watch glass and put the fiber filter to analytical balance. Then the fiber filter disc is weight to the nearest 0.001 g and the record the data as B which is represent a weight (mg) of empty filter disc. Thus, repeat the process of filtering by using representative water sample and record the following data as A which is represent weight (mg)[1] of fiber filter disc with suspended solid. Calculate the test result in order to get the Total Non Filterable Residue (TNR)

For calculation, after the two different weight of fiber filter disc is determined that is A and B, in order to get the Total Non Filterable Residue (TNR) the equations it will generate as the following expression:

$$A - B \div L$$

A = Weight (mg)[1] of fiber filter disc with solids

B = Weight (mg) of empty fiber filter disc

By using this equation, the weight difference between the empty disc and the disc with materials shows (TNR). On other hand, to measure the Volatile Non Filterable Solids (VNR) the equations it will generate as the following expression:

$$A - C \div L$$

A = Weight (mg)[1] of fiber filter disc with solids

C = Weight (mg) of fiber filter disc with solids

The weight difference between the disc and the disc with remaining material shows (VNR).

3.7 MEASUREMENT APPARATUS

3.7.1 COLORIMETER

Calorimeters is widely used to monitor the growth of bacteria and practical application such as testing water quality by screening suspended solid or chemical due to reliable and accurate results when used for the assessment of color. Basically, colorimeter is a light sensitive device for determining the transmittance absorbance of light passing through liquid sample. It allows the absorbance of a solution at a particular frequency (color) of visual light. Since it is relative to the absorbance, this device is possible to find out the intensity or concentration of the color that develops upon introducing a specific reagent into a solution. Figure 3.12 shown a colorimeter that be used in this experiment.

Figure 3.12 : Colorimeter

There are three main components of colorimeter which are light source, a cuvette containing sample solution and a photocell for detecting the light passed through the solution. The output of colorimeter is display by digital meter in terms of transmittance or absorbance.

The working principle of this device is based on Beer Lambert's law which the absorption light transmitted through the medium is directly proportional to the medium concentration. For this process, a beam of light with a specific wavelength is passed through a solution via series of lenses, which navigate the colored light to the measuring device. The amount of light projected through the sample is known as transmittance. Transmittance can be expressed as the ration on intensity between transmitted light I_t and initial intensity of light source I_o.

$$T = \frac{\mathrm{It}}{\mathrm{Io}}$$

In addition, a colorimeter contains a voltage regulator for protecting the devices form fluctuations in mains voltage. Hence the voltage variations created by the colorimeter allows the transmittances data to be analyzed. In fact, there are three elements that affect the transmittance of a sample liquid including: ε, molar absorptivity of the solution, b, cuvette width and C, molar concentration.

$$log\frac{1}{T} = \varepsilon bC$$

Other than that, if the concentration of the solution is greater, more light will be absorbed as shown in the equation below with A as a absorbance.

$$A = \log\frac{1}{T}$$

Combining these two equations it will generate as the following expression:

$$A = \varepsilon bC\ (Beer's\ Law)$$

At the end of this process by having these three elements, the amount os suspended solid can be determine in the term of milligram per liter (mg/L).

3.7.2 SCANNING ELECTRON MICROSCOPE (SEM)

Scanning electron microscope (SEM) such as shown in Figure 3.13 is a device that scans a sample with a focused electron beam over a surface to create an image with information about the samples topography and composition. Thus, for this experiment (SEM) is used based on the capability of this devices to identify the sample morphology, fiber diameter (micro and nano scale) and suspended solid trapped by nanofiber filters. For normal operation of SEM which is in high vacuum mode it has an ultimate resolution approximately three nanometers. In fact, the SEM is fitted with an Energy Dispersive Spectroscopy (EDS) analyzer for determining elemental composition of samples. In addition in order to analyses the detail structural morphology, Image J software is used to determine the fiber diameter and pore size of fabricated nanofiber. Hence, by using this software, better selection of fabricated nanofiber filter can be done. Figure 3.14 shows illustrations of working process of SEM.

Figure 3.13 : Scanning Electron Microscope (SEM)

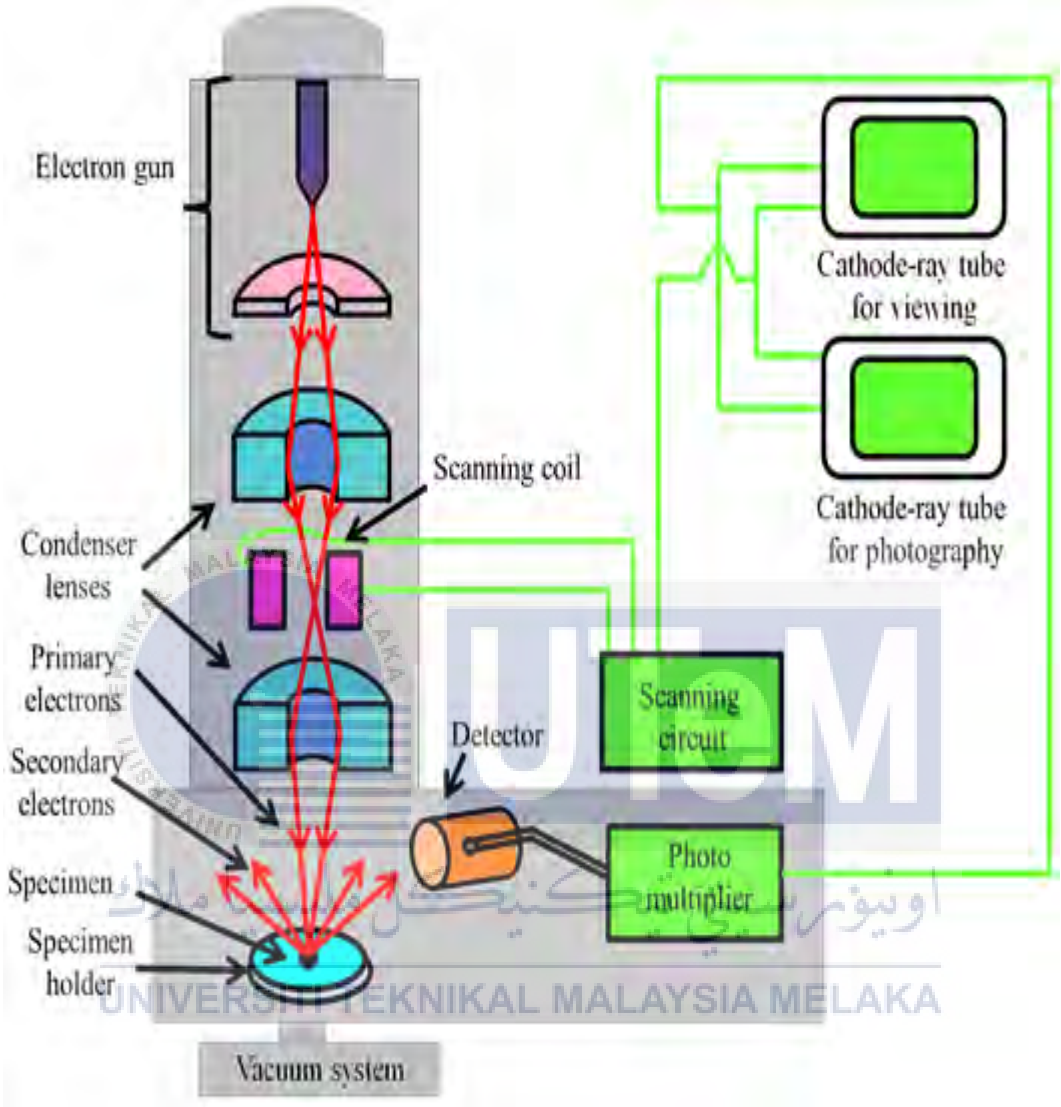


Figure 3.14 : Illustration of working process of SEM

Before the SEM process begin, coating of samples is required in the field of electron microscopy to enable or improve the imaging of samples. Creating a conductive layer of metal on the sample inhibits charging, reduces thermal damage and improves the secondary electron signal required for topographic examination in the SEM. Fine carbon layers, being transparent to the electron beam but conductive, are needed for x-ray microanalysis, to support films on grids and back up replicas to be imaged in the SEM. The coating technique used depends on the resolution and application. Figure 3.15 shows the sputtering process which is required before scanning process. The process time is depends on what material need to be sputter.

Figure 3.15 : Sputtering process

CHAPTER 4

RESULTS AND DISCUSSION

4.1 ELECTROSPINNING OF NYLON 6, 20wt%

Electrospinning process was carried out using a laboratory scale electrospinning machine (Model ES1a, Electrospinz, NZ). A constant applied voltage of 14 kV was used throughout the electrospinning process. The distance between the spinneret and the collector is constant which is 10cm. Polymer solution was prepared by dissolving Nylon 6 pellets (Sigma-Aldrich 181110) in formic acid (Merck 1002642500) to a final concentration of 20 wt.%. In this research, the varied parameter was the time of the filter paper is being electrospun which is 20,40,60,80,100, and 120 second through electrospinning process such as shown in Figure 4.1. The sample is divided by three and average weight is taken to estimate the actual value as tabulated at Table 4.1. This parameter is very influential in order to get the desired nanofiber morphology.

Figure 4.1 : Filter paper that have been layered by nanofiber through electrospinning process

Table 4.1: The weight of each sample with different time of electrospinning

Sample / **Time(s)**		**0**	**20**	**40**	**60**	**80**	**100**	**120**
1	Weight (g)	0.0990	0.0992	0.1006	0.1010	0.1014	0.1018	0.1020
2	Weight (g)	0.0990	0.0995	0.1002	0.1012	0.1015	0.1016	0.1018
3	Weight (g)	0.0990	0.0993	0.1003	0.1010	0.1013	0.1019	0.1021
4	Average weight (g)	0.0990	0.0993	0.1003	0.1011	0.1014	0.1018	0.1020

Thus, for further analysis, the average weight of samples is taken as weight after electrospinning. So, in order to get the weight of electrospun, the differences between weights after and weight original filter paper were calculated and tabulated as shown in Table 4.2. At the beginning of the process, the original filter paper Whatmann was used, which does not have nanofiber layered on it.

Table 4.2: The weight of electrospun were calculated

Model	F1	F2	F3	F4	F5	F6	F7
Time(s)	0	20	40	60	80	100	120
Weight before (g)	0.0990	0.0990	0.0990	0.0990	0.0990	0.0990	0.0990
Weight after average (g)	0.0990	0.0993	0.1003	0.1011	0.1014	0.1018	0.1020
Weight of nanofiber (g)	0	0.0003	0.0013	0.0021	0.0024	0.0028	0.0030

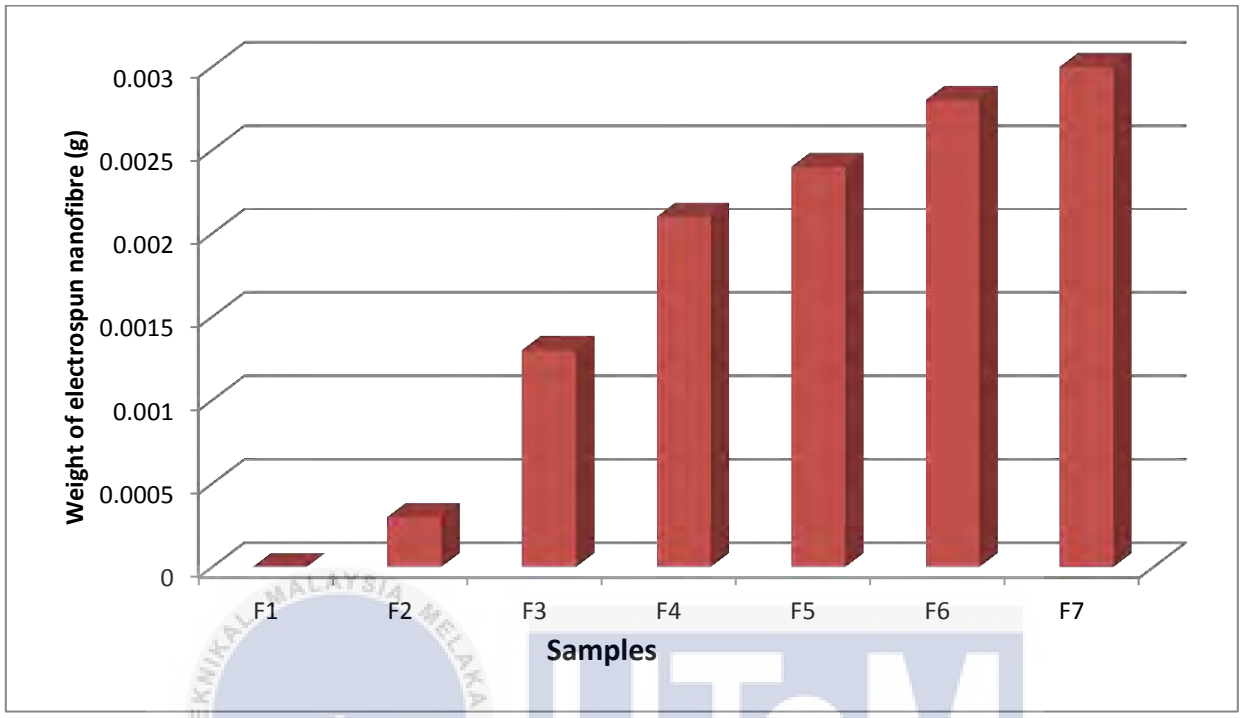


Figure 4.2 : Graph of collected nanofiber.

From the Figure 4.2 the histogram shows the graph between samples(time of electrospinning process) against the weight of electrospun nanofiber which is collected to the filter paper. As mentioned before in Chapter 4.1 for each sample there is the differences between the times of electrospinning process. Thus for the beginning of the process the collected nanofiber on filter paper went up from 0 to 0.0003. After that, for 40, 60, 80, 100 and 120 seconds shows steadily increases which is from 0.0013g to 0.0030g. As a conclusion, the weight of collected nanofiber is influence by the time of electrospinning process.

4.2 FILTRATION

Firstly, as refer to Figure 4.3, to ensure the filtration process is done without clogging; the pressure inlet was set up as 10kpa. In some other reasons, there is the way to make sure the processes give uniform hydraulic pressure for each sample. After the 100ml is put into the container, as observation, at the beginning of the filtration process the water drop was fast flow that shown the permeability is high before the suspended solid (SS) started to flow through filter media.

Figure 4.3 : Pressure Inlet

Figure 4.4 : The water samples is going through filter paper

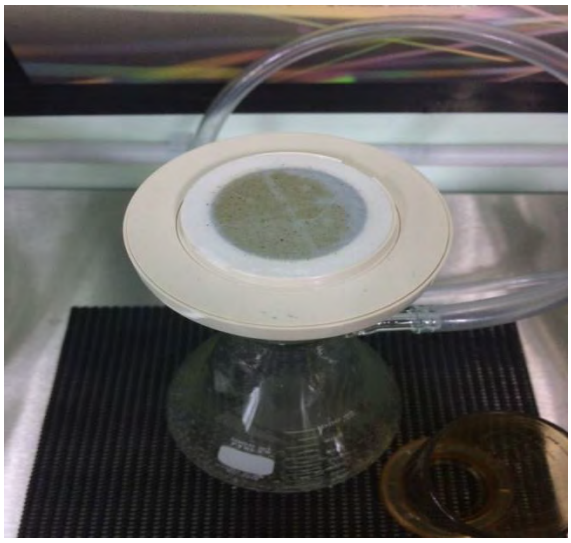


Figure 4.5 : The filter paper after filtration process.

The result in the Table 4.3 shown uniform increases the weight of nanofiber layer on filter paper. This proved, the effect of time increased in electrospinning process. Based on Table 4.4 below, for BS EN 872 method the value of total suspended solid (TSS) were calculated from dry weight of the filters before and after filtration. Hence, the difference between the values divided by the amount of water sample for each test gave the amount of total suspended solid (TSS) that trapped by the filter in weight percentage (wt. %). Thus, for the first test by using original filter with pore size of 1.2 µm and diameter 47mm the TSS value was 4 mg/L. However, at the end of results, the total suspended solid (TSS) retained were increased same as the time of electrospinning process increased. So the values of TSS were further calculated by using sample F2, F3, F4, F5, and F6. Based on the value pattern, this shown the presence of nylon 6 is elctrospun nanofibers significantly improved the capability of filters. In addition, steady increases of TSS suggest that there were positive trend between filter capability and the amount of applied nanofibers by using electrospinning process. The Figure 4.5 and 4.4 above showed the filter paper samples after filtration process.

Table 4.3: The weight of each sample after filtration process

Sample / **Time(s)**		**0**	**20**	**40**	**60**	**80**	**100**	**120**
1	Weight (g)	0.0994	0.1001	0.1012	0.1022	0.1032	0.1038	0.1042
2	Weight (g)	0.0993	0.0999	0.1013	0.1026	0.1031	0.1038	0.1041
3	Weight (g)	0.0994	0.1000	0.1015	0.1026	0.1031	0.1037	0.1045
4	Average Weight (g)	0.0994	0.1000	0.1013	0.1025	0.1032	0.1038	0.1043

Table 4.4: Total Suspended Solid (TSS) retentions using BS EN 872 method

Model	F1	F2	F3	F4	F5	F6	F7
Time(s)	0	20	40	60	80	100	120
Voltage)KV)	14	14	14	14	14	14	14
Weight before (g)	0.0990	0.0993	0.1003	0.1011	0.1014	0.1018	0.1020
Weight after average (g)	0.0994	0.1000	0.1013	0.1025	0.1032	0.1038	0.1043
Total Suspended Solid retained (mg/L)	4	7	10	14	18	20	23

For instance, in order to get value of total suspended solid (TSS) in milligram per liter (mg/L) the formula of TSS is used, which is the differences of weight after and weight before filtration process divided by (0.1L = 100ml) that refer to volume of water samples for each test.

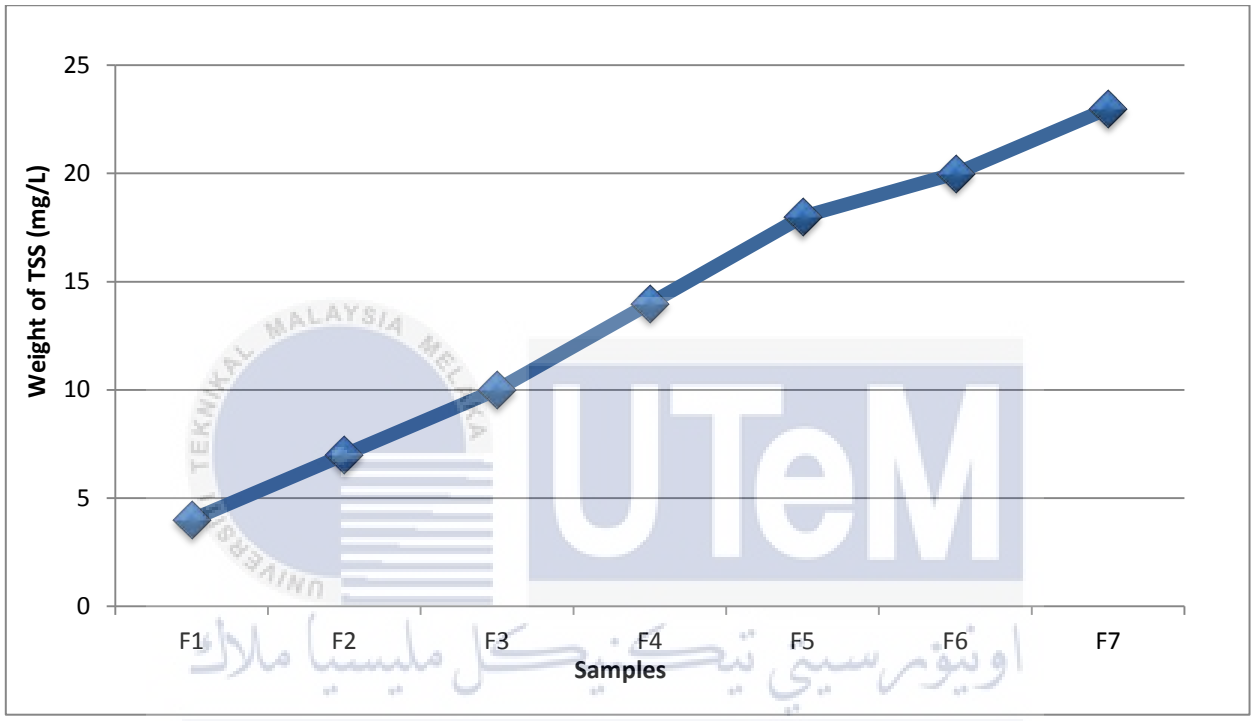


Figure 4.6 : Weight of retained SS

From Figure 4.6, the result has been proved that the samples with nanofiber layered captured greater amount of suspended solid compare to original filter paper. So, the addition of nylon 6 significantly improved the quality of filtration process of waste water. Hence, by far the most efficient filter paper is sample F7 filter that attained high suspended solid which is 23 mg/L. Hence, there is hypothesis can be made by this results which is the higher time of electrospinning process (addition of nanofiber) the higher amount of suspended solid can attained by filtration media. Thus, the graph above shows the time of electrospinning is directly proportional to the weight of TSS

4.3 TOTAL SUSPENDED SOLID (CALORIMETER)

Calorimeter is generally a device that specifies sense of measuring samples concentration and makes it possible to ascertain the concentration of the waste water suspended solid, since it is proportional to the absorbance. For comparison purpose, waste water taken form water treatment plant at UTEM Main Campus is being tested at the beginning of the process. Then, by using the calorimeter waste water for each test have been tested on this device. TSS value of water samples before and after filtration by using calorimeter was tabulated on Table 4.5. The varied parameter was the electrospinning time (addition of nanofiber) and the TSS before filtration was constant for this project, because same waste water is used during whole filtration process.

Table 4.5: Total suspended solid (TSS) values before and after filtration using calorimeter

Sample	F1	F2	F3	F4	F5	F6	F7
Electrospinning time (s)	0	20	40	60	80	100	120
TSS before (mg/L)	8	8	8	8	8	8	8
TSS after (mg/L)	4	2	1	0	0	0	0

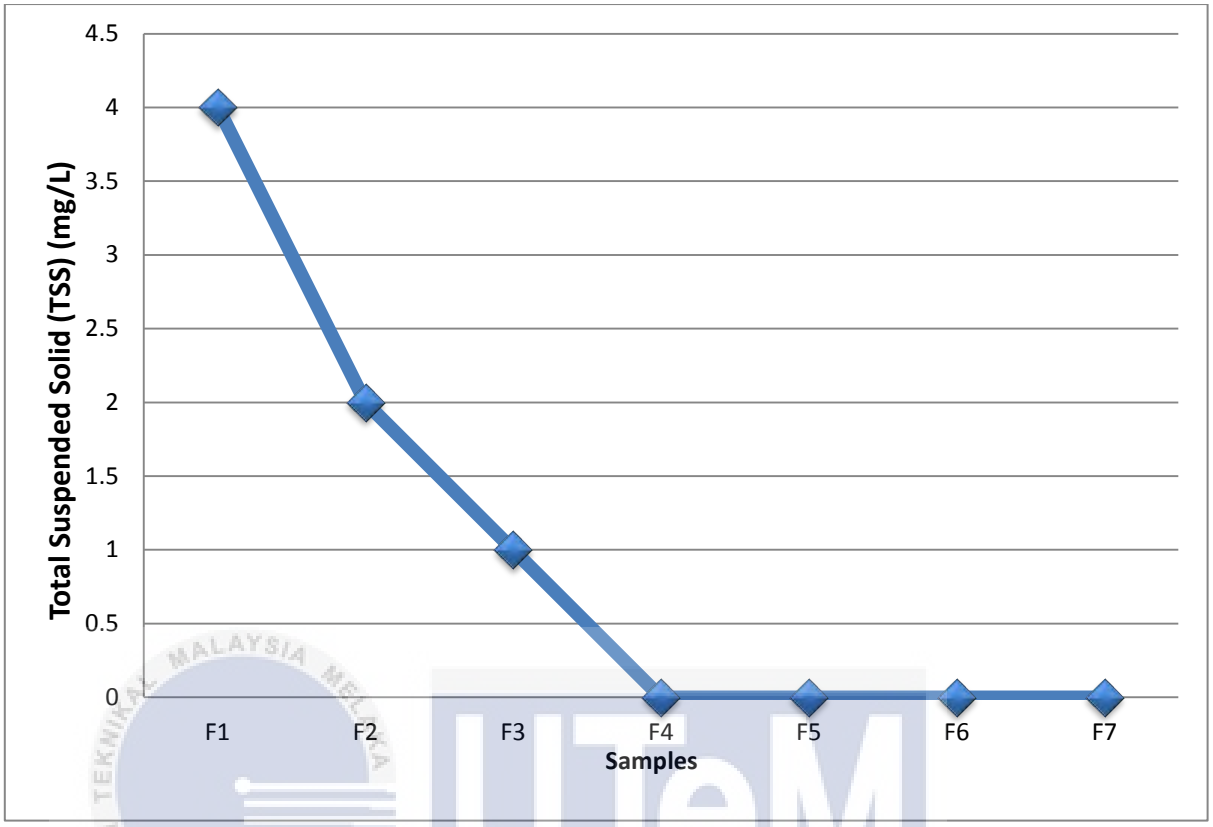


Figure 4.7 : Graph of TSS value after filtration by using calorimeter

From the Figure 4.7 above, it significantly shows that samples F2, F3, F4, F5, F6 and F7 is attained more SS compare to original filter paper because of the presence of nanofiber. However due to limitation of this device the actual TSS value were only recorded as Not Detected (ND). As a results, both method proved that media filter with nanofiber layered performed better filtration compared to the original filter and attained greater of total suspended solid (TSS).

4.4 SCANNING ELECTRON MICRSOSCOPY (SEM) ANALYSIS

For this step, after scanning process have been done, nanofiber diameter dispensation of the SEM images for each samples within electrospinning time which was 20, 40, 60, 80, 100 and 120 seconds. Then, for further analysis Image J software is used in order to get the nanofiber diameter. Next, after ten different reading of diameter is for each sample was taken, the average nanofiber diameter is summarized in Table 4.6. Therefore, the calculations of range of diameter R were calculated by the differences of maximum diameter X_{max} and minimum diameter $X_{min.}$

Table 4.6: Table of average nanofiber diameter

Samples	F2	F3	F4	F5	F6	F7
No of measurement	10	10	10	10	10	10
Min. Diameter, X_{min} (nm)	101.28	103.342	88.124	140.823	93.58	103.522
Max. Value, X_{max} (nm)	114.06	123.647	111.52	388.127	158.862	132.326
Average Value (nm)	108.222	113.689	97.665	269.138	118.246	120.43
Range, R (nm)	12.78	20.305	23.396	247.302	65.282	28.804
Average Nanofibre Diameter	108.22	113.689	97.665	269.138	118.246	120.43

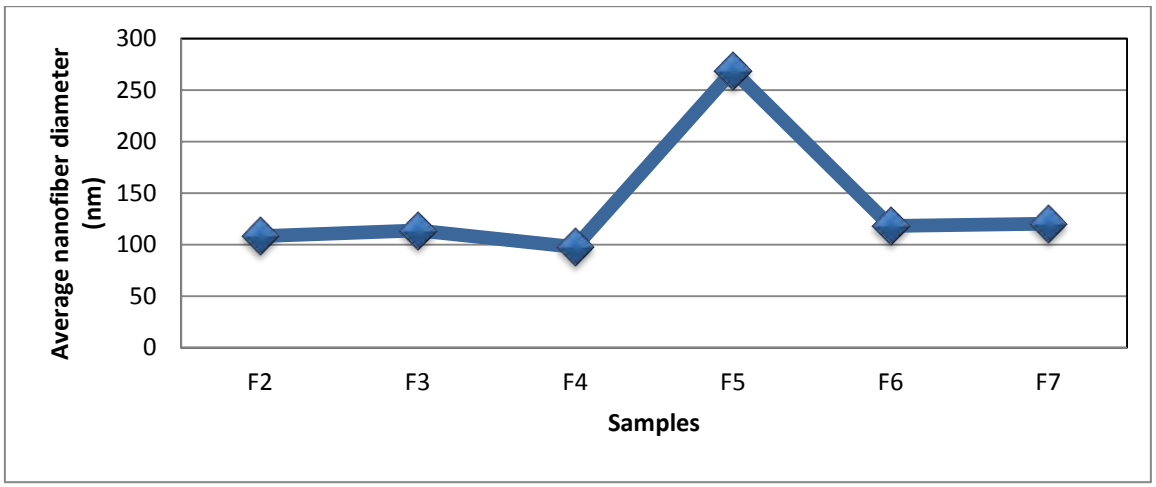
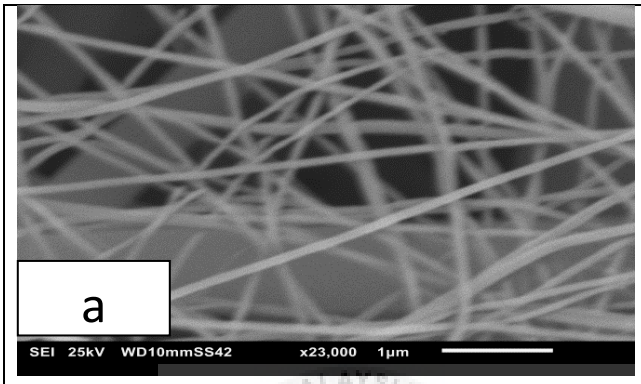
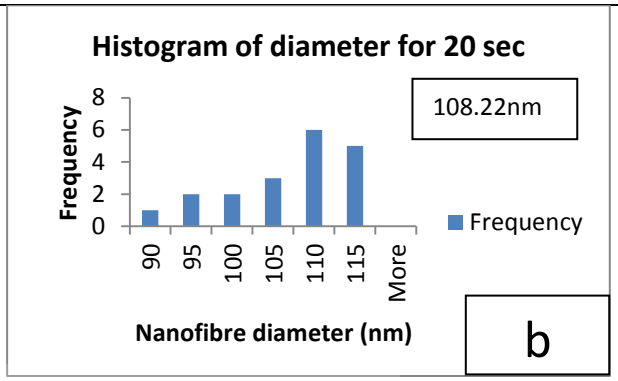
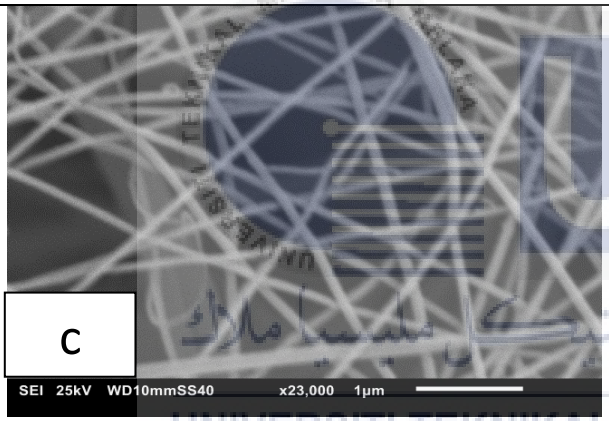
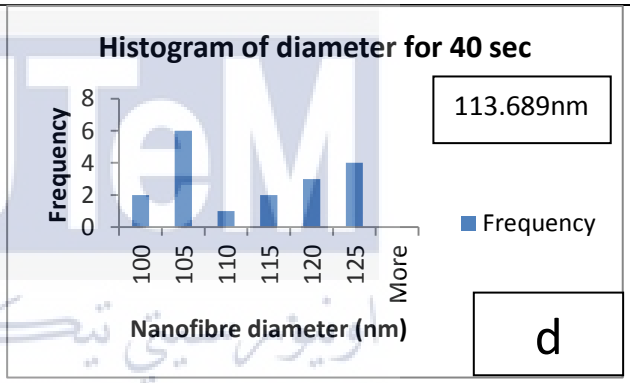


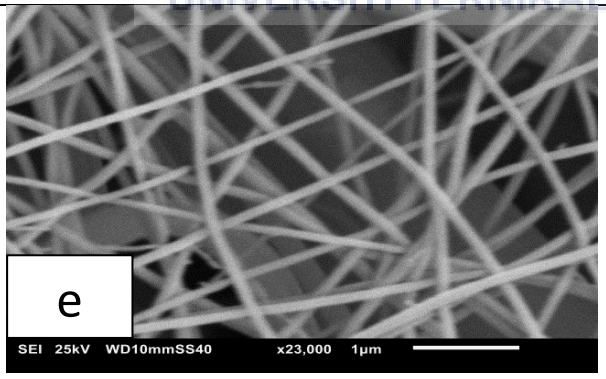
Figure 4.8 : Graph of average diameter

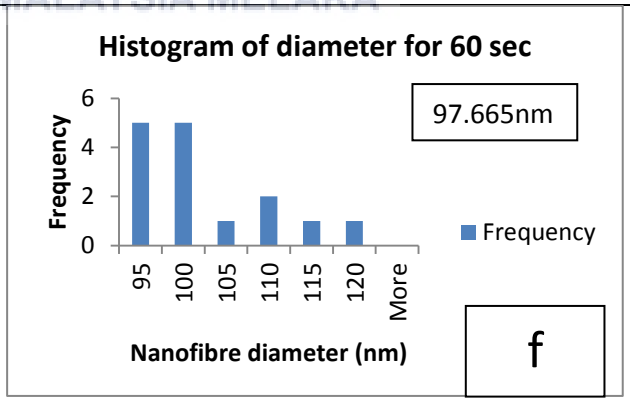












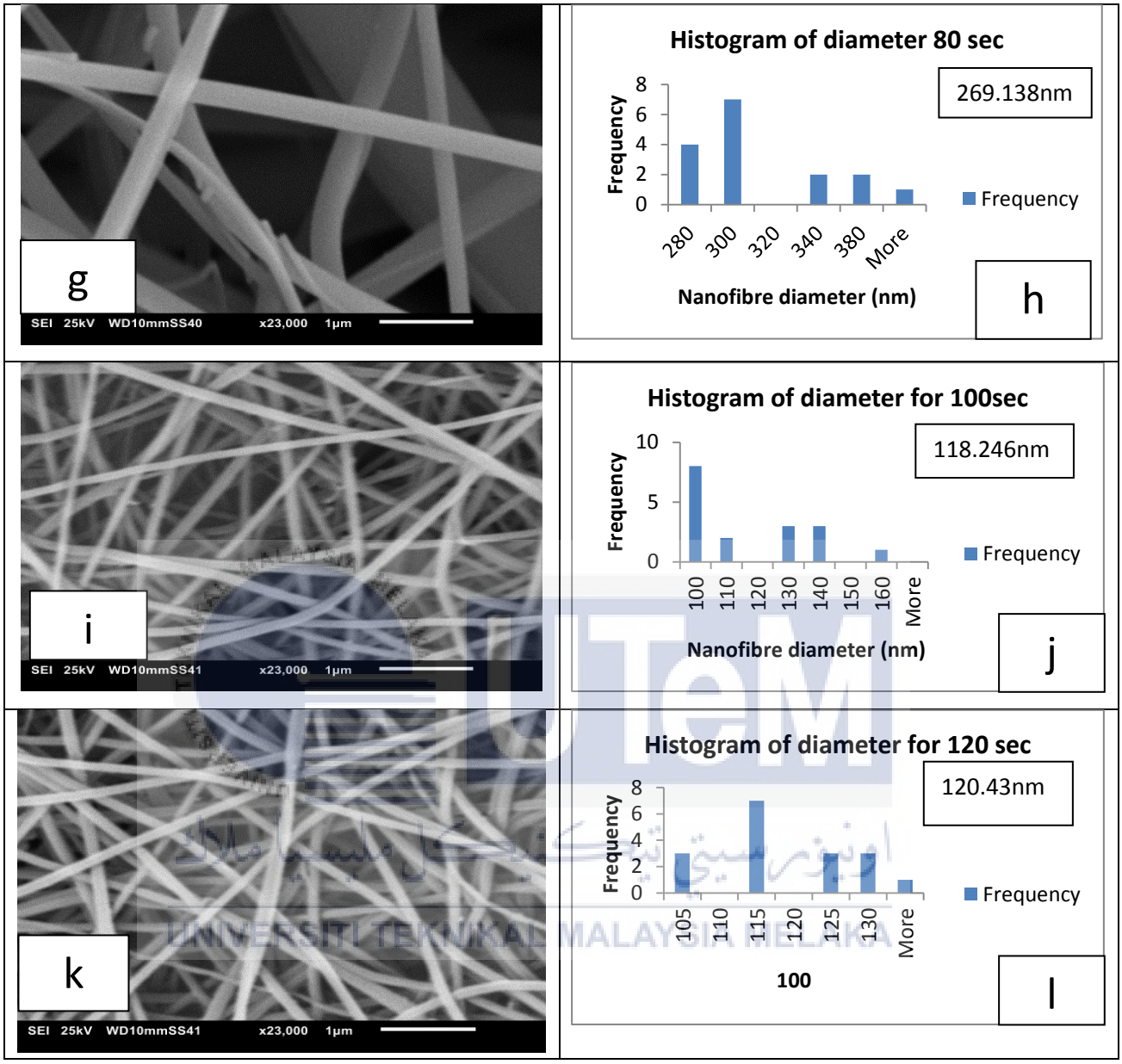


Figure 4.9 : The SEM images of each time samples with histogram of nanofiber diameter against frequency

Figure 4.9 shows the SEM images at 23 000x magnification which the nanofiber diameters were manually analyzed using Image J software. By proceeding the next step, the result from the Image J software were extended analyzed by using Microsoft Excel in order to get the range of frequency against nanofiber diameter. The histogram given in 4.6 (b), (d), (f), (h), (j) and (l) shows approximately normal distribution of nanofiber diameter. The diameter

lying on each sample was in range 95 to 120nm that proved electrospinning process capable to producing fibers in nano range.

From Figure 4.6(a) it can be seen that nanofiber coated are less than other samples. So, the major distribution of the nanofiber diameter was at 110nm – 115nm regions. The average nanofiber diameter recorded was 108.22 nm. Next, for Figure 4.6 (l) the histogram shown the major distribution was in diameter 115nm regions. The average nanofiber recorded was 120.43nm which was the highest one compare to other samples. From these two results there was a difference between the average diameters that shows the diameter was raised from 108.22nm to 120.43nm respectively. However, there is some error occur in 60 and 80 seconds samples. For the 60 second sample the average nanofiber diameter recorded was 97.665nm which less than the 20 and 40 seconds sample. This error occurred might be due humidity that can affect the results of nanofiber diameter. The effect of humidity on fiber diameter is dependent on the interaction between the solution and the surrounding water vapor. According (Golin 2014) to demonstrated this effect on a core-shell nanofibers composed of a poly(caprolactone) shell and a poly(ethylene glycol) core. For some polymers, a higher relative humidity has been shown to result in reduced fiber diameter instead.

The average diameter of nanofibers were obtained manually by selected 10 randomly nanofibers on the SEM images. However, this process is very repetitive, time consuming task and can lead to human error because of enervation. So, for the 80 seconds filter paper the SEM process was captured the nanofiber morphology which not in correct position due to limitations of this devices. The SEM combines high contrast imaging, broad depth of field and high spatial resolution to examine and characterize features at the nanofiber scale. Refer to Figure 4.10 below there is one example of the better scanning by SEM which is clear shown the differences between micro glass fiber and additional layered nanofiber. As a conclusion, the average diameter of nanofiber is influenced by electrospinning time.

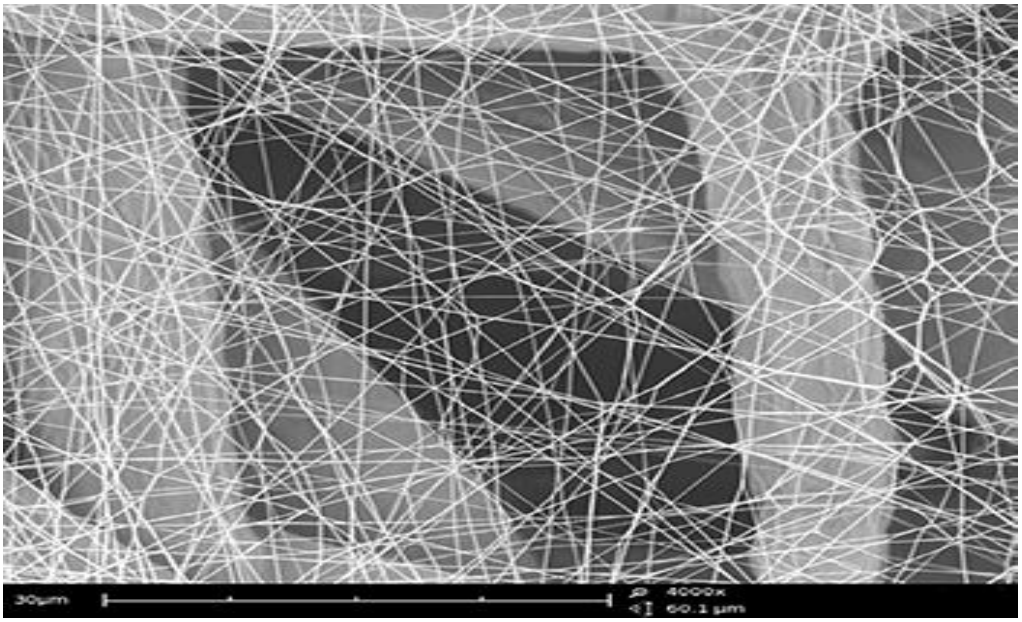


Figure 4.10 : SEM images of nanofiber layered on micro glass fiber (source http://www.nanoscience.com, 2017)

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

There are two main objectives in this project which is to produce high efficient filtration system for capturing suspended solid in industrial waste water and to develop a new high efficient water filtration media using polymeric nanofiber. The filtration effectiveness is determined by approaches some testing including suspended solid retention, turbidity reduction of the industrial waste water sample. The production of nanofiber is conducted by using electrospinning process which used the addition of nylon 6 polymer. After that, in order to get the average diameter of each filter paper samples, Scanning Electron Microscopy (SEM) was conducted at SEM laboratory at Fasa B, UTeM.

As preparation before filtration process was conducted, the water samples was taken from water treatment plant in main campus UTeM. The water samples used for this project need to be uniform for the whole experiment in order to get the desired result. The water filtration apparatus was provided from Hach Company including apparatus and material for water treatment such as calorimeter, turbidity meter, analytical balance, filter flask, filter holder, filter 47mm, tongs, tweezers and watch glass. The filtration processes were done according to European Standard EN 872, Water Quality: Determination of Suspended Solids.

The results in Chapter 4 indicate the success of this project in several different approaches. The nanofibers suggested to be layered on original filter paper appear to attained higher amount of suspended solid in terms of weight and orientation (diameter). The time of electrospinning for this experiment was different for each sample. As a result, after the

filtration process was successfully done, the weight of SS was manually calculated. For further analysis, the waste water after filtration was collected to analysed using calorimeter. The colorimeter SS concentration readings accentuate the efficiency of the whole filtration system. The concentrations of the filtered samples show that the nanofiber incorporated filter paper managed to perform better filtrations than the original filter paper.

5.2 RECOMMENDATION

There are some areas of improvements needs to be executed in this experiment. The electrospinning machine was providing by advance material laboratory in Industry Campus, UTeM can be improvised in many ways. Thus, one of the suggestions is to made collecting nanofiber that can made aligned nanofiber rotating collector of electrospinning which highly recommended for collecting aligned nanofiber sheet. This is the simplest & effective method to make aligned nanofibers. By using different length or diameter ratio, with different rotating speed, an even fiber sheet or aligned fibers can be made.

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