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Multifunctional Electrospun Nanomembranes in Wet Filtration: A Comprehensive Review

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Abstract: Nanomembranes have great potential to play a vital role in filtration and separation technology. Electrospun nanofibers possess unique attributes like exceptional filtration effectiveness, minute pore dimensions, excellent permeability, and affordability. Hence, they are the favored choice for numerous applications involving nanomembranes and filtration. Polymeric nanofibrous membranes are currently used commercially for air filtration; however, their uses in water filtration have not yet been fully explored. Electrospun nanofibrous membranes (ENMs) are the common name for electrospun fibrous membranes, which are flexible, have a large surface area, and have a porous structure that allows for significantly higher sites for separation processes. By providing a method that is lighter, more affordable, and less energy-intensive than that of traditional membranes, ENMs represent a breakthrough in the treatment of water and wastewater. Compared to traditional membranes, ENMs have high porosity, typically in the region of 80%, as opposed to 5-35%, for conventional membranes. This review highlights the application of electrospun nanofiber membrane (ENM) in wastewater treatment and surface modification of nanomembrane to address the fouling issue of membranes. In addition to the removal of suspended particles, heavy metals, and particulate matter, nanofibrous membranes can be used for the removal of pathogens and chemicals. The separation of oil from water is an exigent issue due to the higher amount of oily industrial wastewater and polluted water of the ocean. This review consists of the importance of nanotechnology in wet filtration and the application of nanomaterials (electrospun nanofibers) in wet filtration applications.

Keywords: Electrospun Nanomembranes, Water and Oil Separation, Surface Modifications, Clean Water.
Polymer-based nanofiber membrane which is produced using electrospinning can be used in filtration process for wastewater treatment.

1 Introduction

The World Health Organization and the United Nations have been using the term "water crisis" for some time to represent the status of water resources in the world. Water pollution and scarcity of usable water present a substantial aspect of the water crisis. Currently, around 10 to 11 % of the world's population has inadequate access to drinking water. The water crisis could affect around 4 billion people by 2050. Water constitutes approximately 70% of the earth's surface, 97% of this water is salty water and cannot be used without any treatment. Additionally, around 3% are trapped underground, leaving less than 1% of the world's water supply for humans. Water resources are under more strain than ever because of how quickly the human population is growing all over the world. Our seas, lakes, and rivers are, in a sense, being squeezed by human activity. As a result, the quality of their water has been drastically degraded. Poor water quality means that water has been polluted and this pollution of water has been more excruciating, especially after the 19th-century industrial revolution. Water pollution is generally caused by biological, toxic, organic, and radioactive waste, chemical waste, microorganisms, and nutrients present in water, which causes detrimental effects on humans and animals.

People have been dealing with several problems due to the shortage of clean water, fresh drinking water around the world, especially in developing and underdeveloped countries, including 1.2 billion people have insufficient access to clean drinking water, 2.6 billion people have poor or no sanitation, and millions of people die each year from infectious diseases spread through contaminated water or human excreta [1-2]. As indicated by certain research, the global population is projected to reach around 9 billion by 2050, and approximately 75% of this population might experience a

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scarcity of fresh water by 2075 [5]. The health and safety of all nations depend on the availability of clean and potable water supplies. When toxic matter enters rivers, lakes, streams, and oceans, they generally get dissolved in water or stay suspended in water, thereby causing pollution, and degrading the quality of water to a great extent. Pollutants present in water can easily seep down and affect the quality of groundwater deposits. This water ultimately used in households is highly contaminated and carries microorganisms, which cause diseases. The water used in agriculture containing fertilizers and pesticides ultimately drains into rivers and oceans and causes pollution. With the rise in human population, urbanization, and continuous environmental degradation due to human activities, the shortage of potable water supply will be more adverse and will certainly constitute a major concern in the years to come. The water crisis will grow consistently and there must be some planning at the international level to address this alarming issue.

It is high time to address this problem, and we certainly need excessive research at the international level to explore new methods of water filtration and purification with minimum energy usage and low cost at the same time minimizing the use of chemicals and their impact on the environment. Viruses of animal and human origin can easily spread in the environment and infect people through water. Intestinal parasitic infections and diarrheal caused by waterborne bacteria have been a health problem due to poor digestion of food by people sickened by the poor quality of water [1,3]. The fastest increase in the world's population, urbanization, industrialization, and drastic changes in our lifestyles are the basic drives for energy and water demand along with the uncontrolled amount of wastewater generation due to human activities [4]. As indicated by certain research, the global population is projected to reach around 9 billion by 2050, and approximately 75% of this population might experience a scarcity of fresh water by 2075 [5]. New technologies and methods for wastewater treatment have become indispensable. Advanced technology has led to the focus of researchers' attention on advanced nanomaterials, such as nanofiber membranes for the filtration of wastewater. Considering the rising demand for freshwater supply in both industrialized and developing countries, and keeping in view the current water supply scenario, there is an obvious need for the latest and unprecedented technologies to solve the water crisis.

Numerous techniques, including distillation, chemical disinfection, sand filtration, reverse osmosis, and membrane filtration, have been and still are in use to clean water. One of these more recent technological advancements is membrane technology that offers various advantages such as the potential for scaling, minimal energy usage, absence of chemicals, and operation at lower temperatures. A membrane serves as a selectively permeable medium for filtration, allowing specific compounds and molecules to pass while preventing the passage of others. Electrospinning is a novel and adaptable method for producing nanofibers. Electrospun nanofibers are an ideal choice for filtration applications, due to their additional characteristics of minimum fouling, selectivity, high filtration efficiency, high permeability, small pore size, and low cost. Consequently, the demand for technological innovation to allow desalination and water treatments cannot be overstated [5].

2 Nanomembranes in Water Treatment

Nanotechnology is the art and science of maneuvering matter at the nanoscale, atomic scale, or molecular level, presenting the potential of engineered nanomaterials for filtration applications. Due to its critical role in developing novel materials with the smallest possible dimensions and containing cutting-edge properties for use in both industrial and residential applications, nanotechnology has been expanding at a rapid pace in recent years. Nanotechnology is the creation of materials, components, devices, and/or systems at the near-atomic or nanometer levels (one or more dimensions are between 1 and 100 nm). Nanotechnology involves fabrication, measuring, imaging, modeling, and manipulating matter at the Nano level. The purpose of nanotechnology is to control an atom, molecule, and particle to drastically improve the chemical, biological, physical, and physiochemical properties of novel materials and devices. It is impacting a broad range of highly multidisciplinary fields, such as colloidal science, physics, chemistry, materials science, medicine, engineering, and biology.

Nanomaterials are closely integrated with our society and this integration will continue for many decades. Nanotechnology has had a profound influence on various fields of science and engineering, including electronics, polymer engineering, and materials science. It has been ascertained that the influence of nanotechnology in developing techniques for water treatment has been gaining more attention due to promising aspects of nanotechnology. With the fast depletion of freshwater resources, it is expected that engineering nanomaterials will play a significant role in seawater desalination and wastewater treatment. Nanomaterials are smaller than 100 nm in at least one dimension. At this scale, materials often possess astonishing and size-dependent properties that are different from their bulk counterparts, many of which have been explored for applications in water and wastewater treatment. Nanotechnology can provide solutions to environmental problems. Most of the nanomaterials are being examined for their applications in the treatment of groundwater, surface water, and drinking water contaminated by toxic metal ions, organic and inorganic pollutants, and microorganisms [6]. Metal oxide nanoparticles are being developed for environmental monitoring, remediation, and pollution prevention. Metal oxide nanoparticles generally used in water treatment are

TiO_2, Fe_3O_4, Fe_2O_3, MnO_2, CeO_2, MgO and Al_2O_3. Due to the large surface area, high aspect ratio, shape, and dimension, extensive attention has been focused on using metal oxide nanoparticles in catalyst, adsorption, and membrane separation. TiO_2 has been widely used in the treatment of contaminated water. When TiO_2 nanoparticles are irradiated with UV-visible light, these nanoparticles exhibit bactericidal activity. Chang et al. [7] demonstrated that the irradiation of TiO_2 with a wavelength of approximately 380 nm led to bactericidal action. Researchers used iron oxide nanomaterials to remove toxic ions and organic contaminants from water. Surface-modified iron oxide has been used in water treatment, including remediation and disinfection. Bulk MnO_2 and its composites can be used to remove Cd (II), copper (II), lead (II), uranium (VI), As (III), As(V), Se (IV), and organic waste from water adsorption and subsequent catalyst combustion at relatively low temperature [8]. Recently, many functionalized Al_2O_3 membranes have been developed for use in water filtration.

Nanomaterials deal with processes and systems that are built on nanoscale dimensions. Nanomaterials and nanostructures having a dimension in the nanoscale (1 to 100 nm) often exhibit exotic and unbelievable properties, such as silicon being an insulating material but at the nanoscale, it becomes highly conducting. This is due to the structure, large surface area to volume ratio, and quantum effects of nanomaterials. Nanotechnology can contribute to long-term water quality and availability by employing advanced filtration techniques such as nanofibrous materials. Graphene repels water naturally. However, if narrow pores are made in it, they allow water the permeation. This idea spurred researchers to investigate nanographene as a potential candidate in water treatment. nanographene sheets (perforated with holes) allow water molecules to pass through and block the passage of contaminants present in water. Graphene possesses outstanding properties such as lightweight high surface area. Graphene can be used as an energy-efficient and environmentally friendly filter media in wastewater treatment and desalination. It has been found out that very thin graphene oxide membranes are impermeable to all gases and vapors, besides water, and further investigation disclosed that an accurate mesh can be fabricated that will allow the separation of atomic species thereby enabling super-efficient filtering. The newly emerged 2D graphene has been a potential candidate as a filter media due to its chemical and thermal stability, good flexibility, and solution processability [9]. Moreover, graphene or graphene oxide (GO) can form ordered films with 2D nanochannels between two graphene sheets using a facile filtration-assisted assembly process [10]. Some researchers fabricated wet graphene membranes employing wrinkled hydrazine-reduced GO and investigated its potential application in nanofiltration for nanoparticles and dyes [10]. On the contrary, some other researchers fabricated micrometer-thick GO membranes and determined that these membranes were impermeable to vapors, liquids, and gases but solely allowed unrestricted evaporation of H_2O; after heat treatment and dried graphene membrane became impermeable to any substance including water vapor [10].

The emergence of carbon nanotubes in 1992 further facilitates ideal nanofiltration, since carbon nanotubes possess exceptional properties such as lightweight, stability, high surface area, flexibility, and processability. Additionally, carbon nanotubes provide unique 1D nanochannels for water transport [10]. Carbon nanotubes have attracted extensive attention for the synthesis of novel membranes with fascinating features for water purification. Experimental studies revealed that permeation of water through a carbon nanotube is fast. Majumder et al. [11] determined experimentally that mass transport through multiwalled carbon nanotubes with a pore diameter of around 7nm was 4 orders of magnitude larger than conventional hydrodynamic flow. Many researchers have reported the use of vertically aligned carbon nanotubes for separation and filtration applications. Well-aligned carbon nanotubes can serve as interconnecting pores for water purification, desalination, and decontamination applications [12]. The hollow structure of carbon nanotubes can provide efficient transport of water molecules and small diameters can constitute energy barriers at the channel entries, rejecting ions and permitting water through the hollow structure [13].

Polymeric nanofibrous membranes have gained tremendous attention in recent years in filtration applications due to their outstanding properties. The polymers generally used for fabricating nanoporous membranes are polyacrylonitrile, cellulose nitrate, aromatic polyamide, aliphatic polyamide, cellulose acetate, aromatic polyamide, polycarbonate, polytetrafluoroethylene, polyvinylidene fluoride, polydimethylsiloxane, polypropylene, polyvinylidene difluoride etc. Nanotechnology is being used in water treatment. The electrospun nanomembranes are under laboratory stages for water treatment. In water treatment, several types of membranes such as microfiltration (MF), ultrafiltration (UF), reverse osmosis (RO), and nanofiltration (NF) are used. Considering the scope of this article, only the electrospun nanomembranes for water filtration will be elucidated. Several processing methods have been available for polymeric nanofiber fabrication. Several processing methods are available for nanofiber fabrication. However, electrospinning is a novel process and occupies a unique position due to its minimum investment and easy fabrication. Filtration technology has been markedly improved by the useful addition of electrospun nanofibers because they possess high surface area, small pore size, and flexibility.

3 Electrospinning

Electrospinning is a simple and novel process that produces micro and nano-sized fibers when an electrostatic field is applied to a polymeric solution or melt. There are many processes available to generate nanofibers, such as phase separation, self-assembly, and template synthesis. However, electrospinning is a method of choice due to its simplicity, minimum investment, and short period of processing time. Microfibers and nanofibers can both be produced via electrospinning. Electrospinning is often employed to create submicron and nanosized fibers with large surface areas. Electrospinning is similar to the principle of spinning polymer solutions or melting in a high electrostatic field. In all other processes, shearing or any other mechanical forces are used to generate fibers but in electrospinning, no shearing or mechanical forces are used, instead, electrostatic forces are used to generate fibers. Electrospinning also known as "electrostatic spinning" is not a new technology for processing polymer solutions or melting into micro and nanofibers.

Recent studies show that the influence of an electric charge on a liquid droplet has been reported in the 17th century. It was determined by William Gilbert around 400 years ago that a spherical drop of water is drawn into a conical shape when a piece of amber is placed over it [14]. Electrospinning is the formation of fibers under a high electric field. Almost all naturally occurring and synthetic polymers have been electrospun so far. In conventional spinning, inertia, shearing, rheological, gravitational, and aerodynamic forces act on the fibers. However, in electrospinning, only an electrical field is used to generate fibers. **Figure 1** shows the schematic of an electrospinning process and produced nanomembranes [82].

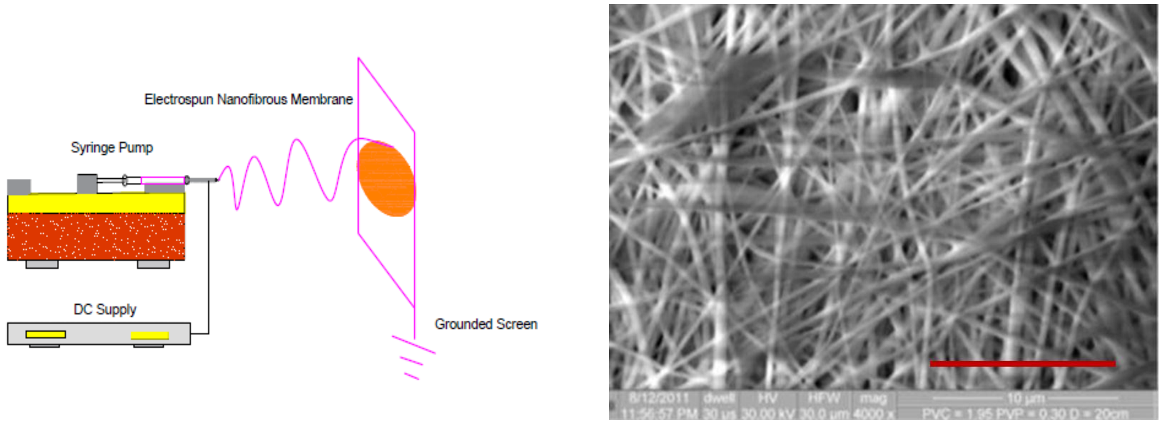

Fig. 1: a) A schematic diagram of electrospinning for polymeric nanomembrane fabrication and b) produced PVC nanomembrane.

Electrospun nanofibers possess high permeability, high surface area-to-volume ratio, high porosity, well-connected pore size, and flexibility [15]. The preferred materials for filtration applications are electrospun nanofibrous membranes (ENMs), due to their high filtration effectiveness, nanosized holes, high permeability, and cheap cost [16-19]. These outstanding properties of electrospun nanofibrous membranes are suitable for not only wet filtration but also dry filtration. Although these membranes are being successfully used in laboratory experiments, their commercial applications are yet to be explored. ENMs have a high flux, but the problem is that the membranes possess a large electrostatic charge during the electrospinning process. Furthermore, ENMs are mechanically weak and require some type of support to filter water. Therefore, nearly all applications of ENMs in membrane separation technology are generally based on hybrid systems [20-21]. In these systems, nanofibers are generally placed on a support (substrate) sandwiched between different layers or blended with micro-sized fibers [21]. ENMs are effective and low-cost methods of water treatment. However, they face fouling during filtration after some time. The accumulation of solutes on the membrane surface is generally termed as fouling and causes hindrance to mass transport, which lowers the productivity of the membrane. Surface modification is a known technique to mitigate fouling. Surface coatings, interfacial polymerization, grafting, and blending are generally used in surface modification. Plasma-induced graft polymerization is different from plasma polymerization. Plasma-induced graft polymerization is a very good technique for applying a selective layer of polymer on the top surface of a membrane. Surface modifications play an important role in increasing the performance of membranes. Many studies revealed that when the membranes are nano-sized, the fouling can be minimized [20]. The application of nanomembrane infiltration addresses several challenges, including the removal of organic and biological impurities. Furthermore, membranes made of nonreactive materials have been employed to break down contaminants such as 4-nitrophenol and bind metal ions in a water solution [20]. The use of a polysulfone ultrafiltration membrane coated with silver nanoparticles is particularly successful in virus removal [20].

ENMs have shown that they are very good candidates in water filtration. However, their large-scale commercialization has been facing many challenges such as environmental and health risks, and compatibility with the existing infrastructure. Recently, AMSOIL has designed a filter that is made of nanofiber for automobile applications [22]. DuPont has been using electrospun fabric products for automobiles, HVAC, bedding protection, and apparel applications [22]. ENMs can provide a drastic increase in filtration efficiency compared to conventional filters at the same pressure drop. ENMs having nano-sized diameters possess a higher capacity to gather the fine suspended particles since slip flow around the fibers increases the diffusion, interception, and inertia impact efficiency [23].

Recently, numerous scientific articles have been published on electrospun nanomembranes for water filtration applications [24]. Balamururgan *et al.* [25] elucidated the recent trends in nanofibrous membranes and their application in water treatment. Feng *et al.* [26] and Subramanian *et al.* [27] reported the new directions of nanofibers in nanofiltration. ENMs possess some unique features, such as high porosity, high surface area to volume ratio, and good water permeability, which present a good contribution towards water treatment [28]. To make electrospun nanofibrous membranes more effective some researchers mentioned the possibility of embedding a variety of polymers, biological agents, and nanosized particles such as carbon nanotubes and graphene nanoplatelets during electrospinning to develop nanocomposite/hybrid nanofibrous membranes having high efficiency and much higher range of applications [29].

4 Surface Modifications of Electrospun Nanomembranes

Membrane distillation technology is an excellent technology for treating saline water, but it suffers significant wetting problems due to contaminants generally present in wastewater. Wu *et al.* [30] reported an effective method of fabricating a polyvinylidene fluoride-co-hexafluoropropylene (PVDF-HFP) electrospun nanofiber membrane with improved anti-wetting properties against low surface-tension substances thereby mitigating fouling to a great extent. Surface modification is generally used to alleviate membrane fouling. Fouling is the unwanted accumulation of solutes on the membrane surface or within the pores thereby increasing the resistance to mass transfer and decreasing the membrane productivity [31]. By reducing fouling, the operational lifespan of the membrane increases, and the energy requirement decreases. The chemical and physical properties of the membrane's surface play a significant role in determining the flus and selectivity of a separation process. Surface modification is a very good method in membrane technology, which is used to increase the performance of the membrane. The polymers that are suitable for membrane applications should preferably be chemically stable and mechanically strong [32].

The surface modification of electrospun nanofibers can be done by two methods; one-step surface modification that can be performed during the electrospinning process (blending and nanocomposites) and post-treatment that can be performed after electrospinning (grafting, plasma treatment, wet chemistry, and coating etc.) as shown in **Figure 2.**

4.1. One-Step Surface Modification

4.1.1 Blending with Other Polymers

The properties of the polymer can be altered or enhanced by blending with other polymers in an appropriate ratio. These properties generally depend on the mixing ratio, molecular weight of the added polymer, and the solvent. The blending improves the surface properties of many hydrophobic polymers including polystyrene, polyvinylidene fluoride, polycaprolactone, and Polyethylene terephthalate.

Hydrophobic polymeric nanofibers that are usually employed for eliminating ionic pollutants from aqueous solutions are frequently modified by incorporating hydrophilic homopolymer or amphiphilic copolymer before electrospinning.

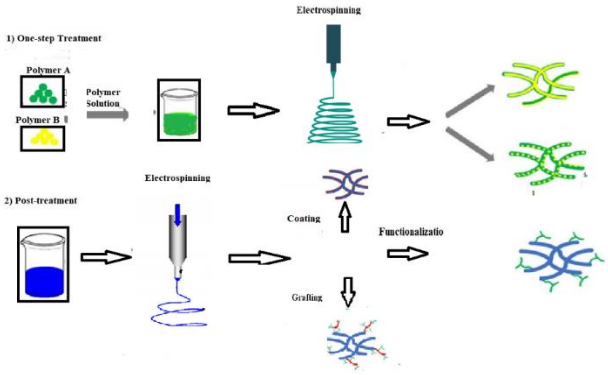

Fig. 2: Surface modification of electrospun nanofibers.

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4.1.2. Incorporating Nanomaterials

The addition of nanomaterials into polymeric solution before electrospinning is another method of modifying the surface properties, mechanical properties, and adsorption capacity and mitigating the fouling effect. The nanomaterials must have a good dispersion property in the polymeric solution, and they should segregate to the surface of nanofibers so that the anti-fouling and modification process is efficacious. Carbon nanomaterials such as carbon nanotubes and graphene oxide are commonly used as nanomaterials in modifying the surface of nanofibers. Other materials such as nano-clay and metal oxides are also used in the surface modification of nanofibers.

4.2. Post-Treatment Methods

4.2.1. Wet Chemistry

The functionalization of the surface of the nanofibers can be done via wet modification. In this process, a chemical reaction is carried out between the surface of the nanofiber and the functionalization agent. The nature of the functionalization agent determines the surface properties of the nanofiber. Generally, polar functional groups on the surface of nanofibers can be created via hydrolysis and aminolysis methods.

4.2.2. Surface Grafting

Grafting is a technique in which one or more polymer side chains are chemically attached to the main chain by using covalent bonds to alter the hydrophilic property, polymer chain, rheological property, and aggregation state of the polymer. Grafting enables nanofibers to absorb ionic pollutants due to the presence of multiple functional groups. There are two methods for grafting polymer: 1) grafting to, 2) grafting from, and 3) grafting through. In the “grafting to” method, the pre-polymerized chains are attached to the backbone polymer with reactive end-groups. This method needs pre-polymer preparation followed by reactions with the surface of the nanofiber. The grafting needs the creation of initiators onto the surface of nanofibers which can be created using wet-chemistry, UV radiation, and plasma or by embedding initiators onto the surface of nanofibers during electrospinning.

4.2.3. Surface Coating

The surface coating is performed by depositing functional materials onto the surface of nanofibers with a layer having a thickness ranging from a few nanometers to micrometers. The bonding between functional material and the surface of the nanofibers is physical such as hydrogen bonding, electrostatic interaction, and π-π interaction. Hydrophobicity, hydrophilicity, adsorption capacity, and fouling resistivity depend on the polarity, layer thickness, and uniformity of the coating materials.

4.2.4. Plasma Treatment

Plasma treatment is another post-treatment method generally employed to enhance the adsorption sites on the surface of the electrospun nanofibers. Argon (Ar), helium (He), nitrogen (N_2), and oxygen (O_2) are generally used in plasma generation for the surface modifications of nanofibers. These gases are used to create functional groups (-OH, -COOH, -OOH, -NH2) onto the surface of polymer nanofibers. These functional groups improve the hydrophilicity and adsorption capacity of nanofibers.

Generally, the polymers that can provide a convenient pore structure should be hydrophilic to be used as filter media [33]. Surface modification is done by blending, surface coating, grafting, and interfacial polymerization [34]. Plasma-induced graft copolymerization is also an advanced method for surface modification. The plasma-induced copolymerization results in the reduction of the surface pores of ENM without any compromise on its bulk porosity [34]. Kaur *et al.* [35] applied plasma-induced grafting on ENM surfaces to reduce pores while maintaining the base structure.

According to a study, electrospun PVDF membrane was first heated at 60^{o}C for 1 hour to eliminate organic solvent then subsequently heat treated again at 157^{o}C for 3 hours to improve the structural integrity of the membrane, then a polyamide thin layer was formed using interfacial polymerization reaction of p-phenylenediamine (PPD)/aqueous phase and trimesoyl chloride (TMC)/organic phase [33]. When PVDF polymer is blended with clay nanocomposites the hydrophobicity of the membrane increases and the static water contact angle reaches as high as 154^{o} [36]. The surface modification of PVDF membranes results in superhydrophobic characteristics. These modifications include dopamine surface activation, silver nanoparticle deposition, and hydrophobic treatment [36].

A Poly (vinyl alcohol) (PVA)/ Polyacrylonitrile (PAN) electrospun nanofibrous membrane was fabricated with a PVA layer electrospun on a PNA substrate then this membrane was melted by water vapors to form a barrier film and cross-linked in glutaraldehyde water/acetone solution. The highest flux of approximately 210 L/m^2 with a rejection rate of 99.5% for an operating pressure of 0.35 MPa was achieved [36]. Wu et al. [37] grafted polyethyleneimine (PEI) on electrospun PVC coated with polydopamine. This membrane was efficient in eliminating Cu^{+2} from an aqueous solution. Many electrospun fibrous membranes such as polyvinylpyrrolidone (PVP), polyamide, polymethyl methacrylate (PMMA), and polyvinyl chloride (PVC) are used in water purification. A mesoporous PVP/SiO2 membrane containing amino group polyvinylpyrrolidone/silica/3-aminopropyltriethoxysilane composite nanofibrous membrane was used for the removal of metal ions [38]. An electrospun polyamide nanofibrous membrane incorporated with Mg $(OH)_2$ and combined with a hydrothermal strategy (1–5 hr.) for the removal of Cr (VI) from an aqueous solution [39].

Huang et al. [40] applied a post-treatment method to enhance the mechanical properties of Polyacrylonitrile (PAN) and Polysulfone (PSU) membranes. The mechanical properties of these polymers were enhanced using solvent-induced fusion of inter-fiber junction points. The membrane showed good improvement in tensile strength and modulus of elasticity while maintaining high porosity and good permeability [40]. Yan et al. [41] studied the self-polymerization of dopamine on the surface of an electrospun polyvinyl alcohol/ polyacrylic acid fibrous membrane and produced water stable by post-heat treatment. The porous structure of this membrane was blocked by the agglomeration of polydopamine in between the nanofibers and the adsorption rate was reduced.

The adsorption capacity of polydopamine and electrospun PVA/PAA membrane is less than that of the surface-modified membrane and better adsorption of other membranes is due to the presence of both porous structure and polydopamine layer [38]. Adsorption generally occurs at lower pH and about 93% of adsorption is observed within 30 min. Cyclodextrin is an efficient modification agent because of the presence of a large number of coordinating groups therefore, many researchers used cyclodextrin as a modifying agent [38]. Surface modification of Poly (vinyl alcohol) employing synthetic and natural products is generally used to eliminate nanoparticles from water. This surface modification is done by using mercaptopropionic acid, mercaptosuccinic acid, and Lysine for eliminating Au and Ag nanoparticles from water. This process was developed and used by Mahanta and Valiyaveettil [42].

The electrospun nanofibrous PVA membrane produced by blending with polysaccharides can be used for the adsorption of nanoparticles from water whereas, heat treated membrane is used for the removal of nanoparticles such as Ag, Au, and Pt [38]. Electrospun PVA membrane blended with gum karya and subsequently heat treating induce esterification reaction between gum karya the membrane has shown a water contact angle of 54.5°. The plasma treatment of heat-treated membrane produces a rough surface with a static water contact angle reaching as high as 104.3°. The plasma-treated membrane is more effective and stable than the heat-treated membrane and has a stronger affinity towards nanoparticles from aqueous solution and eliminates nanoparticles such as Pt, Au, Ag, CuO, Fe_3O_4 [43].

A PVDF membrane was surfaced and modified by grafting with acrylic acid and methacrylic both chemically and with plasma treatment. A high-water flux of 150 Kg/h- m^2 at an operating pressure of 4Psig, and a 79% removal of polyethylene were achieved [44]. Stephen *et al.* [45] surface-modified nanofibrous membrane with oxolone-2, 5-dione to enhance the surface area of the membrane and help in eliminating heavy metals such as lead and cadmium. Schiffman *et al.* [46] modified chitosan polymer by crosslinking with Glutaratldehyde and shciffs imine. Likewise, Haider *et al.* [47] manifested the solubility of chitosan nanofibers by treating them with trifluoroacetic acid (TFA). Yoon *et al.* [48] studied the modification of poly (ether sulfone) nanofibers by using two solvents, such as N-Methyl-2-pyrrolidone (NMP) and Dimethylformamide (DMF) to improve mechanical properties and oxidation process. The surface property of the membrane, such as hydrophilicity was improved by treating it with 3% w/v of ammonium persulfate. As far as the improvement in modulus and strength are concerned, a profound increase of 570% and 360%, respectively, was attained by the addition of high boiling solvents, such as NMP and DMF.

The static contact angle values of 120° and 28° were observed before and after treatment, respectively [48]. Biorge *et al.* [49] exhibited the formation of silver ions by immersing various polymeric membranes in $AgNO_3$ and subsequent reaction with $NaBH_4$. Li *et al.* [50] electrospun microporous membrane and annealed it at around 100°C to control the size of the pores and increase the tensile strength. The membranes exhibited excellent performance in particle (nano and micro-sized) rejection. Wu *et al.* [51] applied Trimesoyl chloride, triethanolamine, and B-cyclodextrin (CD) as the additives in polymer solution with a concentration of 1.8% (*w*/*v*) of CD in the aqueous phase during interfacial polymerization. Their test results showed a 2-fold increase in the value of water flux of TFNC than normal polyester membranes. **Table 1** shows surface modifications performed by different researchers to mitigate fouling [52].

Table 1: Nanofiber Surface Modifications [52].

Substantial	**Adjustment**	**Active group**	**Objective metal**	**Exclusion**
Chitosan	Nullification with K_2CO_3	-NH_2-Amine	Copper(II), Pb(II)	485.44 mg/g, 263.15 mg/g
Silica	Zonal dissolution of polyacrylonitrile	-SH-Thiol	Mercury(II)	57.49 mg/g
Acetate ester of cellulose	*In situ* polymerization	Fluorinated polybenzoxazine	Oil water	Maximum
Polysulfone	Graft copolymerization	Carboxyl group	Toluidine blue O.BSA	380 nmol of TBO/mg of TBO
Poly ether sulfone	1. Solvent induced fusion, 2. oxidation	Carbonyl	Waste water	1. flux: 2,626 L/m^2hpsi, 2. flux: 2,913 L/m^2hpsi
PETE, PCTE, PTFC, PA	$AgNO_3$ lessening	Silver	Pathogen, Waste water	Turbidity exclusion: 99.25%, COD: 94.73%, NH_4: 93.98%
Poly lactic acid	Annealing	-COOH-	TiO_2 removal	85% elimination
Polyacrylo nitrile	Hot press interfacial polymerization	-CN-	Salt rejection, $MgSO_4$	86.5%
Polyacrylo nitrile	Coupling	-NH_2-	Antibacterial	53.7–99.9%

5 ENMs in Filtration Applications

Scientists and researchers have developed an ideal relationship between turbidity and diseases in humans. This relation shows that more turbidity means more toxic substances present in water, which would certainly increase diseases in humans. Several studies showed that ENMs reduced turbidity in water to a significant level. ENMs can rapidly and economically eliminate total dissolved solids (TDS) pathogens, monovalent and multivalent anions and cations, salts, minerals, and other suspended nanomaterials [53]. ENMs can remove protozoa (*ascryptosporidium and giardia*). **Figure 3** shows a schematic of ENMs filtration process.

As is seen in **Figure 3**, filtration is a process of eliminating suspended particulates from raw water by applying pressure to drive water through a porous or semi-porous permeable media. The polymer used in ENMs fabrication is mechanically and chemically stable with a hydrophilic surface for use as filter media. This ENMs filtration can easily remove pathogenic microorganisms in wastewater and water to prevent waterborne diseases. Many research studies have reported the applications of ENMs for eliminating bacteria, particles, or dye from water [54].

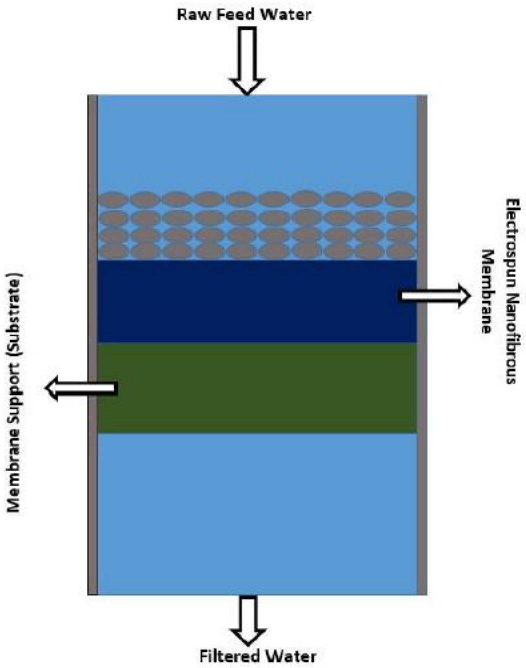


Fig. 3: The schematic of ENMs filtration process.

Generally, bacteria are in micro size, whereas viruses are in tens of nanometers. An ENM can easily remove bacteria; however, for virus removal, the size of the pore should be extremely small (50 – 500 nm). The electrospun membrane can be fabricated in nanosized by controlling the process parameters such as applied (spinning) voltage, tip-to-collector screen distance, the viscosity of polymer solution, and the molecular weight of the polymer, etc. The only problem with small pore size is that the water flux is reduced. Sato *et al.* [55] used cellulose fine fibers infused on a PAN ENM nonwoven substrate to fabricate a composite membrane for removing bacteria and viruses, as well. To remove viruses, they charge the membrane (polyacrylonitrile ENM membrane) by applying the cellulose fiber layer on the top surface, so the positive cellulose fibers attract and trap the negatively charged viruses. They achieved 99.99% efficiency in removing *Escherichia coli* (*E. coli*), which is in close agreement with the 2 cu/ml set up by the National Sanitation Foundation Standard [55]. ENMs can remove bacteria such as coliform, salmonella, cryptosporidium, giardia, and other water-borne microorganisms. Some researchers used the incorporation of silver nanoparticles in polyacrylonitrile membranes during electrospinning and testing for gram-positive Basillus cereus and gram-negative E. coli bacteria [56]. The studies confirmed that the membrane exhibited anti-bacterial activity. Almost every bacterium that can cause illnesses such as typhoid fever, flu, tetanus, polio, dysentery, cholera, meningitis, infectious hepatitis, and respiratory conditions can be effectively eliminated by ENMs.

Wastewater contains pieces of heavy metals. ENMs can eliminate heavy metals from wastewater. Chromium is a toxic pollutant in wastewater, which causes cancer. ENMs can eliminate chromium. Teha *et al.* [57] demonstrated the removal of chromium (VI) employing functionalized cellulose acetate/silica composite membrane with the removal rate reaching as high as 19.45 mg/g. Some researchers used a poly (vinyl alcohol) polymer matrix for removing Cr (II) up to 97 mg/g [58]. Lead and copper can be removed by chitosan nanofiber membrane [56]. Aliabadi *et al.* [59] reported the removal of other metal ions, including cadmium, copper, nickel, and lead by using ENMs. Generally, the important parameters that must be taken into consideration for determining the quality of filtered water are turbidity, total dissolved solids (TDS), chemical oxygen demand (COD), pH, and biochemical oxygen demand (BOD). ENMs reduced all these parameters to acceptable limits. Alharbi *et al.* [60] demonstrated the fabrication of an electrospun nanofibrous membrane with a blend of polyacrylonitrile and polyvinylpyrrolidone polymeric solutions incorporated with gentamicin sulfate to filter wastewater and dam water. Their results revealed that turbidity, TSS, COD, and BOD were reduced to acceptable limits as outlined by the World Health Organization. Alharbi *et al.* [60] Gentamicin is integrated into a highly hydrophilic electrospun polyacrylonitrile/polyvinylpyrrolidone nanofibrous membrane for wastewater treatment. The Tabuk Sewage Treatment Plant (Tabuk STP) provided the wastewater samples. It is situated in Saudi Arabia's Tabuk city. Tabuk STP design capacity is 100,000 m^3/day of raw wastewater. **Figure 4** shows the primary, secondary, and tertiary wastewater treatment used in the Tabuk STP plant in Saudi Arabia [60].

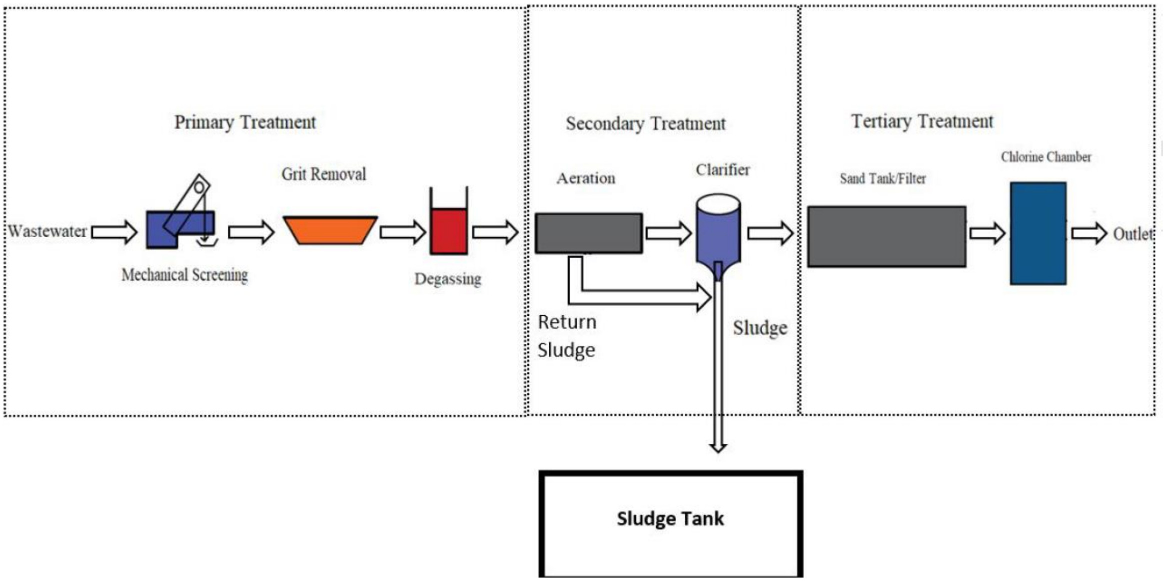


Fig. 4: Wastewater treatment processes used in Tabuk STP, Saudi Arab.

Table 2 contains a list of the filtered wastewater sample chemical and physical analyses. Turbidity, TSS, COD, and BOD are often the most essential factors considered when assessing the quality of filter water. Alharbi et al. [60] used PAN nanomembrane incorporated with gentamicin and varying proportion of PVP polymer and reduced turbidity, TSS, COD, and BOD 78.57%, 83.33%, 28.44%, and 67.62%, respectively. Oil and grease were completely removed. A similar study done by Makaremi et at. [61] showed similar results with PAN nanofibers, exhibiting superior oil-water separation. Alharbi *et al.* [60] demonstrated that the PAN nanomembrane incorporated with gentamicin and varying proportions of PVP polymer when used in wastewater filtration reduced pH to 7.70 after filtration. The average turbidity of the water after filtering was 14.22 NTU. TDS was determined to be 1589 mg/l. The ability to conduct or transmit electricity is referred to as electrical conductivity. Electrical conductivity was determined to be 3219 S/cm.

6 ENMs in Bacteria and Virus Elimination

Pathogens are microscopic biological organisms in drinking water that cause diseases. Pathogens include viruses, bacteria, and protozoa [62-63]. Water purification does not mean the removal of suspended particles, heavy metals, and other solid materials but also pathogens and chemicals. Various polymer solutions and polymer gels had been electrospun to try and remove these contaminates; the most common polymers are chitin, a chitosan derivative, poly (ethylene-co-vinyl alcohol), poly (glycolic acid) and chitin and chitosan/PVA [63-68]. ENMs can eliminate most bacteria such as coliform, salmonella, cryptosporidium, giardia, and other water-borne microorganisms. A study elucidated the embedding of silver nanoparticles (3-6 nm) in polyacrylonitrile electrospun membrane and tested for gram-positive Bacillus cereus and gram-negative E. coli microorganisms. The polyacrylonitrile membrane displayed antibacterial activity [62]. The methodology was used by amidoxime functionalized polyacrylonitrile. Tests were conducted to find microbes such as s. aureus and E. coli for Ag+ and its reduction to Ag nanoparticles. The ASFPAN-3, which were amidoxime functionalized nanofibers after immersion for 20 min in NH_4OH showed log 7 reductions (complete kill) [62]. A similar trend was observed for the $AgNO_3$ solution, in which polyacrylonitrile nanofibers were dipped in solution for 30 min, and Ag nanoparticles/PAN nanofibers, displayed log 7 bacteria reduction [62].

ENMs can practically eliminate any bacteria that cause a variety of diseases, including typhoid fever, influenza, tetanus, polio, dysentery, cholera, meningitis, infectious hepatitis, and respiratory ailments. To prevent waterborne infections, ENMs filtration can remove pathogenic microorganisms from both water and wastewater. Literature reviews have reported on the use of ENMs to remove bacteria, particulates, or dye from water [63]. Bacteria are generally in micrometers, whereas viruses are in the range of tens of nanometers. ENM can remove bacteria due to their micrometer size; however, for removing viruses the pore size should be very small (50-500nm). Electrospinning can produce fibers nanosized if the process parameters are controlled properly. The only problem with small pore size is that the water flux is reduced. Sato et al. [69] used cellulose fine fibers infused on a PAN ENM nonwoven substrate to fabricate a composite membrane for removing bacteria and viruses. To remove viruses, they charge the membrane (polyacrylonitrile ENM membrane) by applying the cellulose fiber layer on the top surface, so the positive cellulose fibers attract and trap the negatively charged viruses. They achieved 99.99% efficiency in removing Escherichia coli (E. coli), which is in close agreement with the 2 cu/ml set up by the National Sanitation Foundation Standard [69]. It has been determined that nanofibers can absorb viruses and remove bacteria by size exclusion. The nanofibers can obtain a 8.6 LRV for A.Laidlamii and a 9 LRV for B.Diminute, compared to a 9 LRV produced by two commercial microfilters, Durapore VV and Express SHR. The pore size of the commercial filters is 0.2 µm. The pore size of the nylon nanofiber filter was in nm [63]. The ENMs are antibacterial and antiviral having high filtration efficiency. A study shows that the synthesized active composite UiO-PQDMAEMA was embedded with the polyacrylonitrile (PAN) solution to produce an antibacterial nanofibrous filter, which also exhibits a high filtration performance [69]. **Table 2** shows the chemical and physical analysis of filtered wastewater conducted by Alharbi et al. [60].

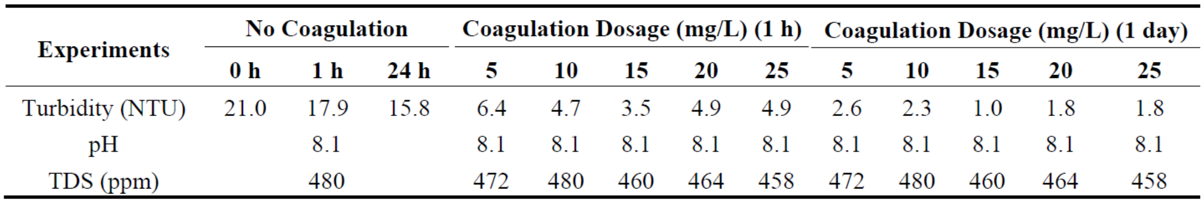
Table 2: Chemical and physical analysis of filtered wastewater samples [60].

Nanofibers	pH	Turbidity (NTU)	TDS (mg/l)	Cond. (µS/cm)	TSS (mg/l)	COD (mg/l)	BOD$_5$ (mg/l)	Phosphate PO_4^{3-} (mg/l)	Ammonia NH_3-N (mg/l)	Oil-Grease (mg/l)	DO (mg/l)
Raw Water Before Filtration	7.45	42	1658	3356	36	55	21	35.3	19	2	6.2
PAN+0 wt% PVP + 0 wt% Gent.	7.52	18	1599	3238	14	33	10.1	35.1	18	0	5.52
PAN+0 wt% PVP + 2.5 wt% Gent.	7.71	17	1597	3229	12	27	9.2	34.2	17	0	5.48
PAN+0 wt% PVP + 5 wt% Gent.	7.77	15	1604	3247	11	22	8.5	34.6	17	0	5.61
PAN+5 wt% PVP + 0 wt% Gent.	7.55	18	1587	3217	9	36.9	9.4	34.9	17	0	5.78
PAN+5 wt% PVP + 2.5 wt% Gent.	7.78	15	1607	3253	8	38.3	9	33.5	16	0	6.05

PAN+5 wt% PVP + 5 wt% Gent.	7.76	11	1591	3221	8	39.4	8.1	35	17	0	5.43
PAN+10 wt% PVP + 0 wt% Gent.	7.80	14	1601	3245	8	40.87	9.6	33.8	18	0	5.66
PAN+10 wt% PVP + 2.5 wt %Gent.	7.68	11	1570	3179	8	40.3	7.3	34.1	16	0	5.92
PAN+10 wt% PVP + 5 wt% Gent.	7.72	9	1553	3148	6	39.36	6.8	34.7	16	0	5.83
Average	7.70	14.22	1589.89	3219.67	9.33	35.24	8.67	34.43	16.89	0	5.70
Removal %	+3.62	78.57	6.33	6.20	83.33	28.44	67.62	1.70	15.79	100	5.97

Asmatulu at el. [53] fabricated nanomembrane with polyvinyl chloride blended with polyvinylpyrrolidone using an electrospinning technique for filtration of three different types of liquids such as lake water, water from jet cutting machine, and water containing magnetic nanoparticles. The major goal of their research was to create a highly hydrophilic nanomembrane and use it to filter the suspended liquids at an optimal level. They used a coagulation process to overcome the fouling/biofouling problem. Two chemical agents, Tanfloc (organic) and Alum (inorganic) were chosen for the flocculation/coagulation process. The removal efficiency was measured in terms of total dissolved solids (TDS), turbidity, and pH. **Table 3** shows the turbidity, pH, and total dissolved solids (TDS) values of lake water after filtration.

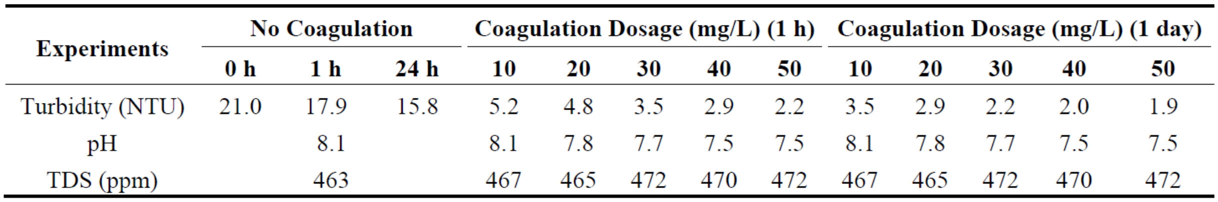
Table 3: Turbidity, pH, and total dissolved solids (TDS) values of lake water as a function of Tanfloc additions after 1 h and 24 h settlements by gravity [53].



Experiments	No Coagulation			Coagulation Dosage (mg/L) (1 h)					Coagulation Dosage (mg/L) (1 day)				
	0 h	1 h	24 h	5	10	15	20	25	5	10	15	20	25
Turbidity (NTU)	21.0	17.9	15.8	6.4	4.7	3.5	4.9	4.9	2.6	2.3	1.0	1.8	1.8
pH		8.1		8.1	8.1	8.1	8.1	8.1	8.1	8.1	8.1	8.1	8.1
TDS (ppm)		480		472	480	460	464	458	472	480	460	464	458

The test results display that the turbidity was reduced to a significant level by increasing the Tanfloc concentration. The initial value of turbidity was 21 nephelometric turbidity units (NTU), and one hour later, the turbidity values of the lake water were reduced to 6.4, 4.7, 3.5, 4.9, and 4.9 NTU at 5, 10, 15, 20, and 25 mg/L Tanfloc dosages, respectively. The turbidity values were further reduced to lower values such as 2.6, 2.3, 1.0, 1.8, and 1.8 NTU at the same Tanfloc concentrations one day later. The residual turbidity values of the lake water were reduced by about 15% after 1 h and about 25% after 24 h without any addition of the Tanfloc coagulants (only settlement due to gravity). It was obvious that the 15 mg/L of Tanfloc concentration and 1 h duration was sufficient to obtain clean water from the lake water. Table 4 shows the turbidity, pH, and TDS values of the lake water as a function of the addition of Alum after 1 h and 24 h settlements by gravity.

Table 4: Turbidity, pH, and, pH, and total dissolved solids (TDS) values of lake water as a function of Alum addition after 1 h and 24 h settlements by gravity [53].



Experiments	No Coagulation			Coagulation Dosage (mg/L) (1 h)					Coagulation Dosage (mg/L) (1 day)				
	0 h	1 h	24 h	10	20	30	40	50	10	20	30	40	50
Turbidity (NTU)	21.0	17.9	15.8	5.2	4.8	3.5	2.9	2.2	3.5	2.9	2.2	2.0	1.9
pH		8.1		8.1	7.8	7.7	7.5	7.5	8.1	7.8	7.7	7.5	7.5
TDS (ppm)		463		467	465	472	470	472	467	465	472	470	472

The test results display that after 1 h, the turbidity values of the lake water were reduced to 5.2, 4.8, 3.5, 2.9, and 2.2 NTU at 10, 20, 30, 40, and 50 mg/L Alum dosages, respectively. Turbidity values were further reduced to 3.5, 2.9, 2.2, 2.0, and 1.9 NTU at the same Alum concentrations after one day of settlement time. Similarly, the pH and TDS values of these tests remained almost the same [53].

Figure 5 (a-d) shows the SEM micrograms of a PAN nanomembrane used in this study. The average diameter of PAN fibers is around 100 nm. No significant change was observed when 5 wt% gentamicin was added, as can be seen in

Figure 5(b); that is, gentamicin has no significant effect on the fiber diameter. A slight increase in fiber diameter was observed with an average fiber diameter of approximately 150 nm, after a 5 wt% addition of PVP, as shown in **Figure 5(c)**. The average diameter of the fibers increased substantially (200 nm) after a 10 wt% addition of PVP, as shown in **Figure 5(d)** [83, 84].

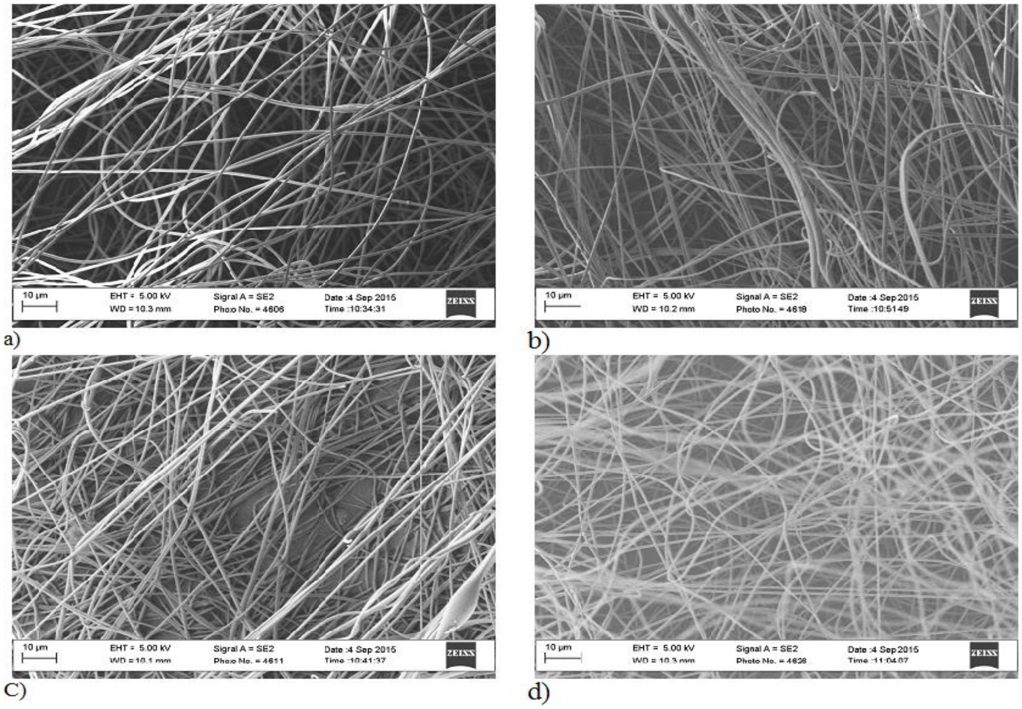


Fig. 5: SEM images of electrospun nanofibers before filtration: (a) PAN + 0 wt% PVP + 0 wt% gentamicin, (b) PAN + 0 wt% PVP + 5 wt% gentamicin, and (c) PAN + 5 wt% PVP + 5 wt% gentamicin, and (d) PAN + 10 wt% PVP + 5 wt% gentamicin.

7 Electrospun Nanofibers in Oil/Water Separation

Electrospinning generates fibers in the range of 100 nm to 1000 nm. Generally, fibers having a diameter of less than 500 nm are considered nanofibers. Wet filtration encompasses a wide range of methods employed to separate solid particles, gas and liquid droplets, and microorganisms from liquid. Wet filtration is generally used in petroleum, chemical industries, pigments, metallurgy, pharmacy, food, paper mills, and coal and water treatment. ENMs are replacing woven fabrics with more wet filtration applications, due to their higher efficiency and ability to retain fine particles. Water pollution is an important factor in recent times. The separation of oil and water is a challenging issue due to the increased amount of oily industrial wastewater and polluted water of the ocean. ENMs can filter oily wastewater. Oil pollution is due to textile industries, petrochemicals, food industries, and time-to-time oil spill incidents during offshore oil production, and marine transportation. This oil pollution damages the ecological environment and causes loss of energy resources, as well [69-71]. Conventional methods, such as flotation, ultrasonic separation, and skimming are not capable of oil/ water separation. Therefore, there is an urgent need for a cost-effective novel method to address this issue. ENMs provide an ideal solution for fabricating a wettable surface for oil/water separation. The conventional method used for decades for oil spill cleanup is mechanical extraction by appropriate sorbents such as nonwoven polypropylene fibrous mats. However, they are marred by a low sorption capacity (< 30 g/g) [72]. The ENMs have shown great promise in this regard. The sorption capacity of ENMs is high because the fibrous surface of ENMs not only drives oil into voids between fibers but also into their pores effectively. The membrane technology is an ideal technology for oily wastewater separation in recent years. The surface features of ENMS can be altered by changing the chemical composition and surface geometry by embedding nanoparticles in polymeric solution or by embedding other polymers in the polymeric solution. Fabrication of fibrous membranes having good wetting properties can be achieved by manipulating chemical composition and surface geometry [72]. The electrospun membrane can be categorized into three types: oil-removing, water-removing, and smart separation membrane. The oil-removing membrane (superhydrophobic and superoleophilic) with superwettable features can repel water and allow oil to flow through thereby achieving high efficiency and selectivity [73].

As far as the wettability of solid surfaces is concerned, the Wenzel and Cassie-Baxter model described that the introduction of roughness on the surface of a membrane can make a hydrophobic surface superhydrophobic and an oleophilic surface becomes superoleophilic [74]. Therefore, a membrane that exhibits both superhydrophobic and superoleophilic properties can be fabricated with high surface roughness and low surface energy [75]. These membranes are extremely useful in oil/water separation applications. Wang et al. [72] demonstrated the application of an in-situ polymerization for the fabrication of superhydrophobic and superoleophilic nanofibrous membranes for oil/water separation. They fabricated a nanofibrous membrane by combining an electrospun membrane and an in-situ polymerized fluorinated polybenzoxazine (F-PBZ) functional layers embedded with nanoparticles of SiO_2 or Al_2O_3. The polymers were either cellulose acetate or poly (m-phenylene isophthalamide) or poly (m-phenylene isophthalamide). By using the F-PBZ/nanoparticles modification, the surface of the membrane displayed superhydrophobicity with a water contact angle of 161□ and superoleophilicity with a water contact angle of 3□ [72]. Some researchers developed the oil-removing membrane by using Ag nanocluster or hydrophobic nano-silica on the surface of the nanomembrane [76-77]. ENMs are extremely promising in water filtration applications having low energy consumption and good permeability and can also be used in oil/water separation after some surface modification. However, the problem of fouling and low flux could be a problem that can be solved by depositing a thin layer of hydrophilic materials, such as poly (vinyl alcohol) (PVA), chitosan, or polyamide onto the surface of the membrane by physical absorption or interfacial polymerization [78-81]. Chu et al. [79-81] reported the surface modification of nanofibrous membrane by hydrophilic layer to attain high flux in water/oil separation. A smart nanofibrous membrane has been developed which is based on a wettability switch for treating oil-contaminated water [72]. **Figure 6** shows an electrospun nanofibrous membrane in oil and water separation.

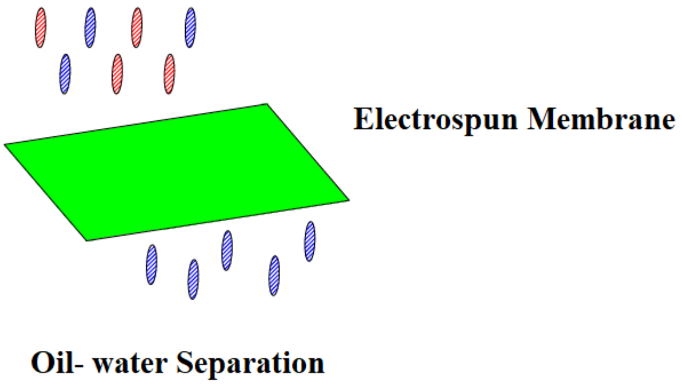


Fig. 6: Oil-water separation using Electrospun membrane.

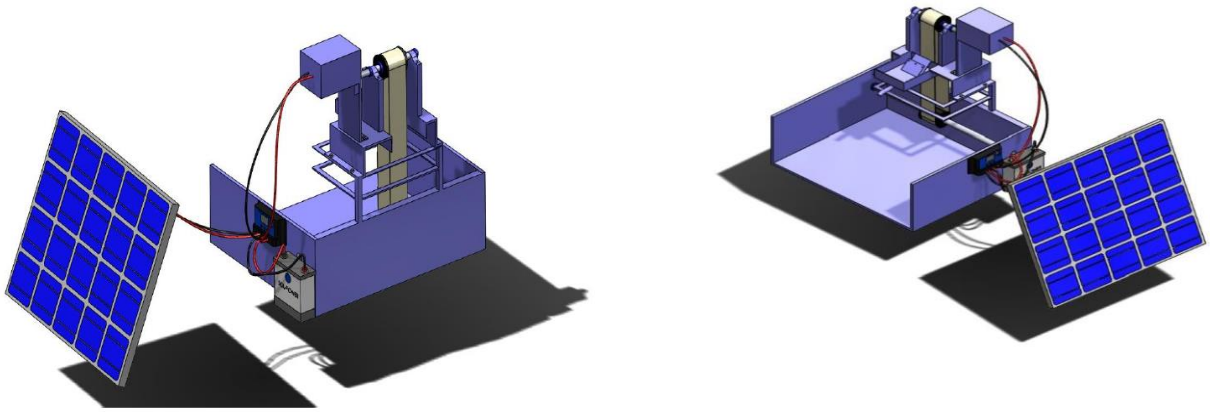


Fig. 7: Oil-water separation using an electrospun hydrophilic membrane.

Figure 7: shows an oil skimmer used to separate oil from water. A hydrophilic belt fabricated from electrospinning and running from solar cells is being used to separate oil from water.

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8 Conclusions

The electrospun nanomembranes are a very good addition to membrane technology for wet filtration. The electrospun nanomembranes possess extraordinary features such as porous structure, high surface area, flexibility, permeability, high pore connectivity, and high aspect ratio which make them an ideal candidate for filtration applications. The exotic properties of nanofibers and their incorporation with current technologies present a promising future for transmuting water/wastewater treatment. ENMs are cutting-edge technology which offers a high rejection rate and high flux compared to all other membranes being used now. ENMs are a breakthrough technology in water and wastewater purification. These membranes have shown auspicious results in laboratory experiments, however, their applications in commercial projects are yet to be explored. ENMs incorporated with metal oxide nanoparticles can decontaminate toxic gases, chemical contaminants, biological contaminants, and pesticides from the atmosphere for several different domestic and industrial applications. ENMs can also filter oily wastewater. ENMs have shown promising results in oil/ water filtration applications due to their surface chemistry and surface features.

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