

# Development of a Noble Fouling-Resistant Membrane for Wastewater Treatment

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**Abstract:** Membrane fouling is a serious matter in membrane operation that impacts the system's durability and cost. A noble fouling-resistant membrane was created to address this issue. A noble fouling-resistant membrane was made using copper oxide-graphene oxide (Cu<sub>2</sub>O-GO) and polyether sulfone. As copper has strong antimicrobial properties, the per-unit strength of graphene is the highest among the known materials in the world. The Cu<sub>2</sub>O-GO was incorporated into the polyether sulfone matrix using the phase inversion method. The effects of Cu<sub>2</sub>O-GO on the performance and antifouling properties of the membrane were investigated. The membrane structure and properties have also been characterized by using Scanning Electron Microscopy (SEM), Energy Dispersive X-Ray Spectroscopy (EDS), and Fourier-Transform Infrared Spectroscopy (FTIR). The SEM and EDX tests were carried out on the 0.1 wt %, 0.5 wt %, and 1 wt% of Cu<sub>2</sub>O membranes. Membrane performance in terms of wastewater treatment and fouling resistance of the prepared mixed matrix membranes was also studied. This project shows a new direction in the research as not many studies using Cu<sub>2</sub>O-GO has been reported worldwide.

**Keywords:** SEM, EDS, FTIR, Membrane Performance, Polyether sulfone, Copper Oxide, Graphene Oxide, Nano filtration, Antifouling Membrane.

## 1. INTRODUCTION

Oman is one of the countries in the world with the least amount of fresh water available. Drinking water is desalinated or treated using membrane filtration on the one hand. On the other hand, there is a lot of emphasis on wastewater recycling these days. Membrane filtration is one of the most effective processes for producing recyclable water all over the world. Every technological advancement has both advantages and downsides. The most prevalent issue that occurs throughout the membrane filtration process is membrane fouling. The life expectancy of the membrane reduces as its strength lowers. Furthermore, numerous approaches are currently in use to control severe fouling of membranes. The life span of the membrane reduces as its strength lowers. Furthermore, numerous approaches are currently in use to control severe fouling of membranes. These include chemical cleaning and membrane replacement, both of which raise operational costs. However, a fouling resistant membrane was not produced in this study. The performance of the created membrane will then be investigated. Membrane fouling occurs when a solution or particle deposits on the surface or pores of a membrane during membrane filtration procedures such as membrane bioreactor (MBR), reverse osmosis

(RO), membrane distillation, ultrafiltration (UF), microfiltration, or Nano-filtration (NF). As a result, the performance of the membrane declines. Fouling of membranes is thus a major impediment to the widespread deployment of membrane technology. It is an inherent effect of the membrane process. Cleaning, correct membrane selection, and operation condition selection can all help to reduce it. Furthermore, membrane fouling causes a significant decrease in flow and has an impact on the quality of the treated effluent produced. Severe membrane fouling typically necessitates either costly chemical cleaning or membrane replacement, both of which increase operational costs. Furthermore, fouling is classified as colloidal fouling (for example, clays or flocs), biological fouling (for example, bacteria or fungus), organic fouling (for example, oils or polyelectrolytes), and scaling fouling (for example, mineral residuals). This study looks into the efficacy of using a Cu<sub>2</sub>O-GO/polyethersulfone membrane to solve one of the most serious difficulties with membrane filtration: fouling. This has an impact on the membrane's performance as well as its longevity. Despite the fact that copper has the best anti-microbial properties of any metal [13] and that Cu<sub>2</sub>O is the most stable copper compound [25], no attention has been paid to the preparation of nano Cu<sub>2</sub>O-GO/polyethersulfone membrane for filtration application to the best of our knowledge. Polyethersulfone (PES) is a polymer that is widely used in the production of commercial and laboratory ultrafiltration and nanofiltration membranes. GO with Cu<sub>2</sub>O nanoparticles on polyethersulfone is

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predicted to offer a high-performance antifouling membrane. [22] Investigated the formation of CuO nanoparticles using a hydrophilic microporous PES ultrafiltration membrane with good antifouling and separation properties. [16] suggest that nanomaterial incorporation into ultrafiltration membranes appears to be a critical factor for evaluating membrane efficiency and productivity [16]. In this study, the researchers hoped to prolong the mixed matrix membrane by incorporating CuO hydrophilic nanoparticles into the PES matrix. The phase inversion method was utilised to analyse CuO/PES UF membranes in order to improve PES membrane properties such as hydrophilicity and anti-fouling. [16] plan to increase not only the hydrophilicity, permeability, and surface properties of PES membranes but also their antifouling capability and flux recovery ratio [16, 17]. The CuO nanoparticles were created using a technique developed by the researchers. The production of CuO nanoparticles is a straightforward and straightforward method of manufacturing nanoparticles. In a 30 mL distilled water bottle, the precursor was combined and diluted. The solution was then poured into a glass bottle to the necessary volume and statically stored at 80 °C for one day. The projected microporous membranes were created using Non-solvent Induced Phase Separation (NIPS) technologies. The functional groups of CuO nanoparticles were determined using Fourier Transform Infrared (FTIR) spectroscopy in the 4000-450 cm<sup>-1</sup> range. Microtrac (Nanotracer Wave) Distribution Light Scattering was used to assess particle size (DLS). The wavelength spans from 0.1 to 10000 nm. The roughness characteristics and surface morphology of the generated membranes were investigated using atomic force microscopy. The height distribution profile of AFM images was used to evaluate the surface pore size distribution of the membranes. Finally, the Flux Recovery Ratio (FRR) was employed to evaluate the antifouling capability of bare and CuO/PES membranes [22]. The researchers created CuO nanoparticles using a simple procedure, then mixed the synthetic nanoparticles with the PES casting solution to begin building the CuO/PES membranes. The purpose of this method was to improve permeability and reduce fouling. SEM images of CuO-containing PES membranes showed no significant or quantitative changes. The purpose of this method was to improve permeability and reduce fouling. SEM images of CuO-containing PES membranes showed no significant or quantitative changes. Contact angle analysis was used to study the AFM technique as well as the effect of CuO nanoparticles on surface

roughness and the improved hydrophilicity of mixed membranes. The increase in hydrophilicity boosted the permeability and antifouling capabilities of the PES membrane [19]. As a result of the research, CuO nanoparticles are an appropriate inorganic additive for PES membranes. [23] used transmission electron microscopy (TEM), scanning electron microscopy (SEM), zeta potential measurements, thermogravimetric analysis (TGA), Fourier Transform Infrared spectroscopy (FTIR), Atomic Force Microscopy (AFM), and contact angle observations to investigate N-CNTs and the N-CNT/PES blend membrane matrix. NMP was used as a moulding solvent in the Polyethersulfone (PES) membrane matrix to efficiently separate CNTs made using only a modest Chemical Vapor Deposition (CVD) method and oxidised with HNO<sub>3</sub>. According to FTIR spectroscopy, the generated membranes are made up of a mixture of PES polymers and N-CNTs that interact via hydrogen bonds. The AFM results demonstrate that as the amount of N-CNT processing increased, the surface roughness of the membranes reduced. Fouling is less frequent when the roughness is reduced. The findings suggest that N-CNTs have a lot of potential for improving membrane properties. [22] provide a strong theory for customising Mixed Matrix Membranes (MMM) to improve permeability, antifouling, and antibacterial capabilities with less GO. Extreme fouling in wastewater treatment could result in a significant decrease in water flux and a reduction in membrane life. As a result, several strategies, such as blending and surface modification, have been used to improve membrane antifouling performance. Because they can damage the cell membrane when they come into contact with germs, GO nanosheets have a high antibacterial efficacy. A considerable amount of research, according to the experts, is focused on incorporating GO into membranes to improve antifouling, antibacterial efficacy, and water permeability. The goal of this research is to use aqueous GO dispersion as a casting solution additive to create PES/SPSf/GO MMMs with improved permeability, antifouling, and antibacterial capabilities by using NIPS [10]. They looked at how different GO concentrations affected the microstructure and properties of MMMs. Raman analysis and molecular dynamic (MD) assays were used to identify the H-bonding interaction between SPSf, GO, and H<sub>2</sub>O. The antifouling and antibacterial properties of MMMs have also been studied. Membrane fouling causes a drop in separation efficiency as well as an increase in maintenance and operation expenses, making it one of the most significant barriers to the widespread use of

membrane technologies [24]. Membrane fouling has been studied, simulated, and controlled using a variety of approaches [2, 15, 21, 6]. Membrane fouling is generally caused by physical and chemical interactions between membrane surfaces and particulates, colloidal particles, or biomacromolecules present in separation solutions [27]. Nonspecific adherence of biomacromolecules and microorganisms occurs on the membrane surfaces as a result, resulting in reduced or blocked membrane pores and, as a result, a severe reduction in permeation or separation efficiency. The incorporation of inorganic nanoparticles into the membrane matrix has been discovered to be an effective way to reduce fouling in membranes, which can be attributed to an increase in hydrophilicity or a change in membrane morphology [11]. Due to its functional hydrophilic groups, graphene oxide (GO) inclusion can induce hydrophilic characteristics in membranes. Roughness and mechanical strength might also be affected. Due to its unique features, such as 2D carbon nanostructure, high specific surface area, and outstanding thermo-mechanical stability, GO is the most frequently utilised material in the preparation of nanohybrids [27, 1, 14, 20]. Many functional nanohybrids have been created by adding different nanoparticles onto the surface of GO, such as silica, gold, silver, zinc oxide, and titanium dioxide, because of the excellent nature of GO [9, 6, 13, 9, 25]. For the purpose of recycling the decanter water produced by the reactive distillation of ethyl acetate [28], the authors looked into a number of different water treatment methods. Saravanan *et al.* [29] looked at a simulator model for groundwater purification systems in Oman that is based on renewable energy. The authors [30] looked at the various energy options available for oil and gas water treatment. In order to remove water from the reactive stage of the distillation process more efficiently, the authors of [31] looked at the role of an entrainer in breaking the water-ethanol azeotrope.

## 2. EXPERIMENTAL METHODOLOGY

### 2.1. Material & Methodology

Copper (I) oxide nanoparticles, N, N-Dimethylacetamide, Polyvinylpyrrolidone, Polysiloxane and O-anisidine were procured from sigma Aldrich.

### 2.2. Preparation of Cu<sub>2</sub>O-GO Nano Composite

Add 40 ml of deionized water in a Pyrex bottle with a fixed amount of graphene oxide, 0.2% weight per cent of the total composition for all the samples. Cu<sub>2</sub>O

nanoparticles were calculated as a weight percentage for each sample (0%, 0.1%, 0.5%, and 1% for different samples). A micro-pipette was used to suspend all samples in 80 µl of O-anisidine. Stir each mixture for 30 minutes using the mechanical stirrer. Autoclave the samples for 6 hours at 120 °C. Ultra sonication was done using the Hielscher UP400St Ultrasonic Processor device. The maximum temperature during sonication was set to 80 °C. Then the sonicated solution was transferred into small centrifuging vials. The centrifugation was done at 15 °C temperature, 6000 rpm, 9 ms acc/dec, and for a 1-hour duration, in order to extract the solid particles from the solution. Then wash the extracted particles with ethanol by filling the ethanol into the centrifuge tubes and shaking them well. Later on, the ethanol was removed and the solid particles were transferred into ceramic bowls. Then dry the extracted solid particles in the oven using ceramic bowls at a temperature of 120 °C. The dried particles were crushed using a manual crusher.

### 2.3. Fabrication of Asymmetric Cu<sub>2</sub>O-GO/PES Membranes

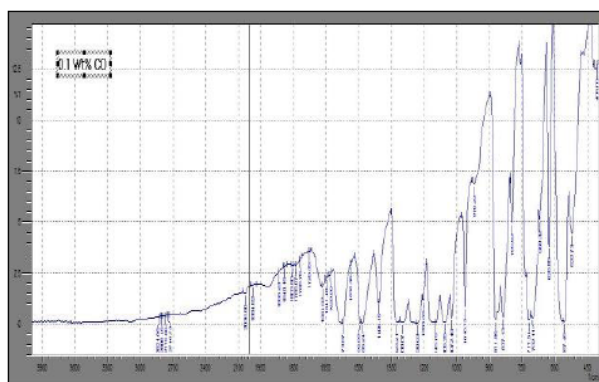
PES particles were dried in the oven to remove the moisture from the particles. Then add the NMP along with PVP and PES (PES should be added gradually in order to avoid the formation of conglomerates) until all the materials are dissolved. Later on, add the Cu<sub>2</sub>O-GO nanocomposite to the previous mixture and start stirring using a mechanical stirrer for 12 hours. This has to be continued for all the samples. Then a sonication bath was done for the samples in order to remove air bubbles and dissolve the small particles. The viscosity of the samples must be checked, then casting of membranes can be carried on. Fix three layers of sticky tape on the four sides of a rectangular glass sheet. The three layers provide the desired thickness of 200µm for the membrane. Pour a sufficient amount of the mixture solution on to the glass sheet. Cast the solution using a glass rod. The casting must be done in one stretch without repetition. Fill containers with distilled water at room temperature. Then, after casting the mixture, immediately immerse the glass sheet gradually into the water bath. And ensure that the water is stable in order to obtain a good membrane formation. Let the membrane be immersed in water for 10 minutes. Later on, separate the membrane from the glass sheet and immerse the membrane in new distilled water for 24 hours. Then remove the membranes after 24 hours from the distilled water. Place the membranes between the filter papers to let them dry.

## 2.4. Characterization of the Membrane

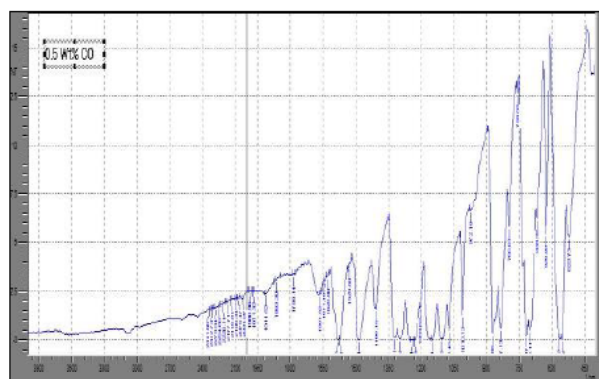
The following tests were conducted to understand the influence of  $\text{Cu}_2\text{O}$  particles on the membrane. FTIR test: This test aims to identify the presence of functionalized groups of  $\text{Cu}_2\text{O}$  nanoparticles in the structure of the prepared membranes. The test was conducted after placing a small piece of the membrane between two layers of sodium chloride plates. SEM: This test investigates the structure of the nanoparticles. In addition, the test was conducted using liquid nitrogen in order to obtain the cross-sectional image of the membrane. Moreover, in order to achieve conductivity of the membranes, the membranes were coated with gold. Energy Dispersive X-ray EDX: Illustrates the nanoparticle distribution within the depth of the membrane.

## 3. RESULTS AND DISCUSSION

The obtained results of the FTIR test as well as the SEM and EDX tests carried out on the 0 wt%, 0.5 wt%, and 1 wt% of  $\text{Cu}_2\text{O}$  membranes. And from the experimental work, the composition as well as the structure of the membrane were studied.



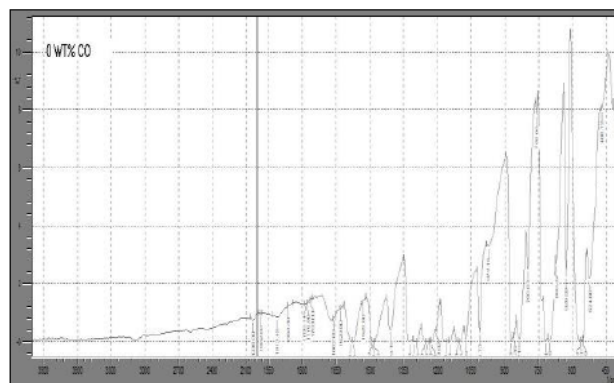
**Figure 1:** FTIR test Zero wt.% of  $\text{Cu}_2\text{O}$  membranes.



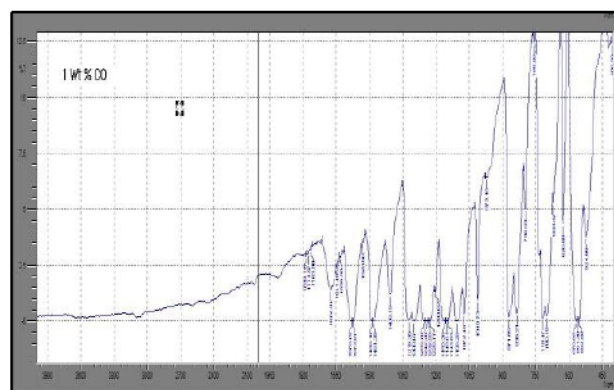
**Figure 1:** FTIR test 0.1 wt% of  $\text{Cu}_2\text{O}$  membranes.

According to the following figures, which describe the FTIR test findings, there are some specific bands for different materials in the FTIR test. It has been discovered that when the  $\text{Cu}_2\text{O}$  content increases, so does the transmittance % for specific bands.

### FTIR Test Results



**Figure 2:** FTIR test 0.4 wt.% of  $\text{Cu}_2\text{O}$  membranes.



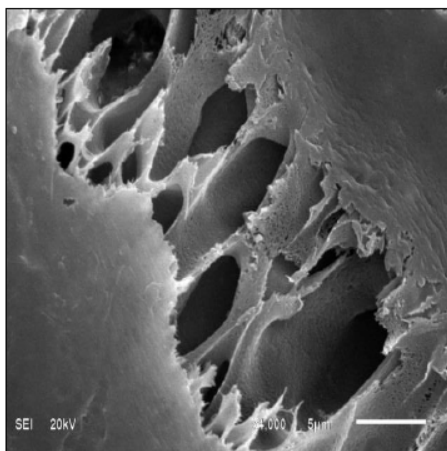
**Figure 3:** FTIR test 1 wt. % of  $\text{Cu}_2\text{O}$  membranes.

### SEM and EDX Test Results

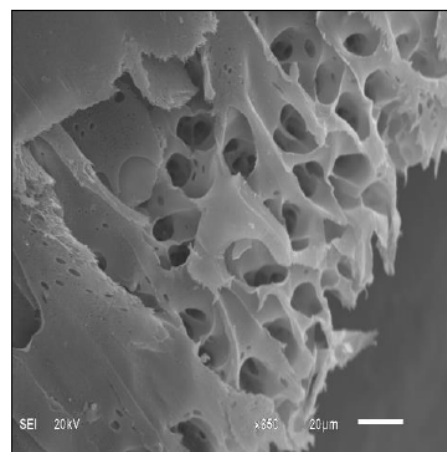
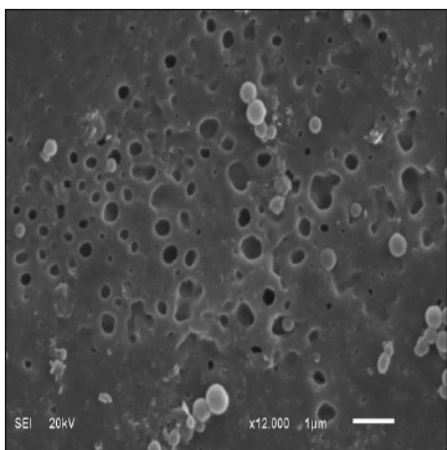
It was discovered that a porous layer had formed in the membrane based on the surface and cross-section photographs. The porous layer is quite appealing. Furthermore, the pores were plainly visible on the membrane's surface. There is also an active layer on top and a porous layer beneath it. The porous layer will encourage rapid permeability.

## 4. CONCLUSION

To summarize, the membrane in this study was created and manufactured using copper oxide-graphene oxide ( $\text{Cu}_2\text{O}$ -GO) and polyethersulfone. The major goal of this membrane is to increase the antifouling and strength properties. SEM was used to



**Figure 4:** SEM results for Cross Sectional area of membrane.



**Figure 5:** SEM results of membrane.

examine the membrane structure. The SEM results demonstrated that a porous layer had formed in the membrane, which is highly desired. Furthermore, based on the discovered pores, a significant penetration rate can be achieved.

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