Polymeric Nanocomposite For Membrane Applications Special Emphasis To Water Treatment

A dissertation submitted for the partial fulfilment of the requirements for the degree of

B. Sc. in Chemistry (Paper DSE-603) under Dibrugarh University

Under the Supervision of-

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CERTIFICATE

This is to certify that the dissertation entitled **"Polymeric nanocomposite for membrane applications special emphasis to water treatment"** *is submitted by* **Pinki Chutia, Roll No.-14720057 Reg.No.-S2205400,** *a B.Sc. 6th semester student of the Department of Chemistry, N. N. Saikia College, Titabar for the partial fulfillment of B.Sc. degree in Chemistry (Paper: DSE-603), is a record of original research work carried out by her under the supervision of* **Dr.Swapnali Hazarika.** *She has fulfilled all the requirements for submitting the dissertation for the B.Sc. degree. The results embodied with this dissertation have not been submitted to any other college or institute for any other degree or diploma to the best of my knowledge.*

Borah 29/05/25
(Jayanta Madhab Borah)

Date: 29/05/2025
Place: Titabar

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TO WHOM IT MAY CONCERN

*This is to certify that Pinki Chutia, a student of B.Sc. in Chemistry, Nanda Nath Saikia College, Titabar, Assam has accomplished her internship project work entitled **"Polymeric nanocomposite for membrane applications special emphasis to water treatment"** as a requirement for academic development, embodies the record of original investigations carried out by her under my supervision and guidance. She has fulfilled all the requirements for submitting the report.*

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CERTIFICATE

This is to certify that the training report entitled, ***"Polymeric nanocomposite for membrane applications special emphasis to water treatment"*** *is a bonafide record of research work done by* ***Pinki Chutia*** *under my supervision and submitted to Nanda Nath Saikia College, Titabar, for the partial fulfillment of her short-term internship programme. This work is carried out at Centre for Petroleum Research, CSIR-North East Institute of Science and Technology, Jorhat, Assam.*

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DECLARATION

I am hereby declaring that the project entitled, ***"Polymeric nanocomposite for membrane applications special emphasis to water treatment"*** *is submitted to Nanda Nath Saikia college, Titabar, Assam for the partial fulfillment of short-term internship research work carried out by me under the guidance of Dr. Swapnali Hazarika ma'am at CSIR-NEIST, Jorhat, Assam. I am further declaring that the work reported in this project is the outcome of my effort.*

Pinki Chutia

Signature of student

Date: 30-01-2025

ACKNOWLEDGEMENT

The training opportunity I had with CSIR-NEIST was indeed a great chance for learning and professional development. I am fortunate to be a part of it and to acquire various theoretical and practical skills. I am also grateful for having a chance to meet brilliant as well as intelligent mindsets and professionals who led me though this training period.

I express my deepest sense of gratitude and sincere regards to Dr. Virendra M. Tiwari, Director, CSIR-North East Institute of Science and Technology for giving me an opportunity to do Project work in this renowned institute.

I am greatly indebted and place my deep sense of appreciation to my supervisor, Dr. Swapnali Hazarika ma'am for providing me an opportunity to do the training on *"Polymeric nanocomposite for membrane applications special emphasis to water treatment"* and giving me all support and guidance, which made me complete the project duly.

I express my deepest thanks of gratitude to Mr. Chinmoy Bhuyan, Senior Research Fellow, Centre for Petroleum Research, CSIR-NEIST for his various helps in this study. I am also greatly thankful to Senior Research Fellows Miss Prathana Bora and Mr. Parashmoni Rajguru for their suggestions during this study, which was extremely valuable for my training, both theoretically and practically. Also, I would like to extend my sincere esteems to all the member of Centre for Petroleum Research Division and laboratory members for their timely support.

Lastly, but not the least I really want to thank honorable working Principal of Nanda Nath Saikia College, Dr. Litool Baruah, honorable HoD, Dr. Jayanta Madhab Borah as well as all other faculty members for giving us the opportunity and providing us the platform.

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Abstract

Wastewater treatment is one of the major global concerns to meet environmental sustainability. To meet this demand, water and wastewater treatment is of utmost important. The polymeric nanocomposite membrane is most advantageous in the membrane technology. Because of the low-cost, eco-friendly, energy efficiency, thermal stability, high porosity, high flexibility and permeability, membrane technology is extensively used in the treatment of water and wastewater compared to conventional technology. Nanomaterials have been widely used in polymer nanocomposite membranes due to incorporation of unique properties that induces water and wastewater treatment performance. The incorporation of nanoparticles improves membrane performance to a greater extent. This report highlights the use of polysulfone membrane fabricated with CuFe-LDHs. The CuFe-LDHs incorporated in the pristine membrane surface are biocompatible, adsorbent specific and possess antibacterial and antifouling properties. Overall, characterization of the membrane shows enhanced and promising performance such as increase in hydrophilicity, high permeability along with high mechanical strength. The best membrane shows very good results for desalination of magnesium sulphate with flux of 7 $L.m^{-2}.h^{-1}$ and 57% rejection for separation of magnesium sulphate from saline water.

1. Introduction

Water, the most precious natural resource covers about three-fourths of the Earth's surface. Only 2.5% of the Earth's water is freshwater out of which only 1% is fit for drinking. The demand for water is increasing due to continuous rise in population growth, urbanisation, industrialisation and climate change. The scarcity of clean water is a major global issue and possess tremendous challenges to the survival of all living species [1]. There are several causes contributing to water scarcity worldwide. Advanced development, rapid urbanisation and industrialisation have led to increased pollution of water bodies, making them unfit for consumption [2,3]. Inefficient agricultural practices have depleted the groundwater bodies causing a serious threat to all the organisms present in the water bodies as well as for human use [4-6]. Further due to anthropomorphic activities, climate change causes irregular rainfall patterns resulting in decrease in agricultural products, drought like conditions and famine. The effluents, toxic dyes and heavy metal ions in wastewater from industrial activities can result in serious damage to human health and the environment [7,8]. Innumerable noxious contaminants including bacteria, fungi, virus, protozoa, pathogens, and chemical pollutants can be found in wastewater. When untreated wastewater is discharged into water bodies or used for other purposes several waterborne diseases like diarrhoea, cholera, typhoid, hepatitis, amebiasis, gastroenteritis, etc. transmits causing a serious threat for public health. Discharge of wastewater into natural waterbodies can have detrimental effects on aquatic ecosystems. The contaminants present in wastewater can bio-accumulate in the food chain thereby negatively impact the environment of the aquatic organisms resulting in adverse effect on their health [9,10]. To overcome from all these obstacles, treatment of wastewater is the alternate route to meet environment sustainability, safeguarding public health and ensuring the sustainable use of water resources. Earlier, conventional technologies such as thermal treatment, ion exchange, adsorption, electrochemical degradation, coagulation, flocculation, chemical precipitation etc were done for the treatment of wastewater [11,12]. But the above-mentioned techniques have several disadvantages such as production of undesirable by-products, high energy consumption, high maintenance and cost, large amount of material handling etc. Wastewater treatment can be performed by implementing membrane technology [13]. A membrane is a semi-permeable barrier that selectively allows desired material to pass through it by retarding the unwanted substances leaving behind it. Compared to conventional technologies for the treatment of wastewater, membrane filtration processes offer several benefits including easy operation, orientation and selectively removes contaminants by altering its structure and pore size [12]. Hydrophobic membranes such as polyvinyl chloride (PVC), polyvinylidene fluoride (PVDF), polysulfone (PSF), polyethersulfone (PES), cellulose acetate (CA), polyacrylonitrile (PAN), polyethylene (PE), polypropylene (PP) etc. are used due to effective separation. Polymeric membranes are economic and good in applications [14-16]. Polymeric nanocomposite membranes are fabricated by dispersing nanoparticles, nanotubes, nanofibers or nanosheets into the polymer matrix via several techniques including phase inversion (PI), interfacial polymerization (IP), physical coating, electrospinning and cross-linking, self-assembly, layer by layer assembly and chemical grafting [17]. Nanocomposite membranes have emerged as promising water purification technologies to overcome the limitations associated with conventional polymeric membranes by offering enhanced hydrophilicity, thermal and mechanical stability, permeability, targeted degradation, solute rejection, and magnetic, antimicrobial, and antifouling properties [18-20]. Layered double hydroxide (LDH) are one such promising two dimensional nanomaterials which are incorporated into the polymeric membrane via interfacial polymerization, dispersing in polymer dope solution or crosslinking with membrane. LDHs are a class of ionic solids with a layered structure with the generic formula $[M_{1-x}^{2+}M_x^{3+}(OH)_2]\ [A^{n-}]_{x/n}.zH_2O$.The divalent metal ions comprise of Mg^{2+},Zn^{2+},Ni^{2+},Ca^{2+},Co^{2+},Fe^{2+},Mn^{2+},Cu^{2+} etc while the trivalent metal ions include Al^{3+},Fe^{3+},Co^{3+},Ni^{3+},Ga^{3+},Mn^{3+}etc.A^{n-} is a non-framework charge compensating interlayer inorganic or organic anion such as CO_3^{2-},Cl^-,SO_4^{2-},RCO_2^-,NO_3^-,OH^-,Br^-,I^- etc. z denotes the number of solvent molecules present between two metal hydroxide layers. And x denotes the ratio between trivalent and total cations including both bivalent and trivalent $(M^{3+}/(M^{2+} + M^{3+}))$ metal ratio represents surface charge which ranges between 0.2-0.4.LDH may also contain monovalent and quadrivalent cations such as Li^+ and Ti^{4+},Zr^{4+},Sn^{4+}respectively[21-24].LDH materials are adsorbent-specific, biocompatible, fire-retardant and exceptionally tunable and exchangeable anionic particles with sensible interlayer spaces. Depending on the requirements of phase purity, high crystallinity and high porosity LDH can be synthesized by different techniques such as coprecipitation method, urea hydrolysis, sol-gel method, hydrothermal method, microwave assisted synthesis, ion exchange etc. [25].

In this work a thin film nanocomposite membrane has been designed by incorporating CuFe-LDHs on the membrane surface by interfacial polymerization method. LDH was synthesised by most common precipitation method [26-28]. A polysulfone membrane have been fabricated by non-solvent induced phase separation method. Designed membranes and nanomaterials have been characterized by FTIR analysis, contact angle analysis and tensile strength measurements. Membrane shows promising performance like very good hydrophilicity, high water flux as well as high mechanical stability.

2. Materials

$Cu(NO_3)_2.3H_2O$ was obtained from Sigma-Aldrich, Spain. $Fe(NO_3)_2.9H_2O$ obtained from Central Drug House(P) Ltd.Na_2CO_3, N-Methyl-2-pyrrolidone(NMP) were obtained from Tokyo Chemical Industry Co., Ltd. The NaOH pellets used were obtained from SRL chemicals. Polysulfone, m-phenylenediamine (MPD) were obtained from Sigma Aldrich Co. made in USA and China respectively. Trimesoyl Chloride (TMC) was obtained from GLR Innovations, New Delhi and n-hexane used was obtained from Avantar Performance Materials Inc. USA.

3. Charaterization techniques

3.1. FTIR

Based on a material's ability to absorb infrared light, Fourier Transform Infrared (FTIR) spectroscopy is a technique used to analyze materials both qualitatively and quantitatively. Infrared light is sent through a sample in the FTIR and the intensity of the transmitted or reflected light is measured as a function of wavelength. The molecular rotations and vibrations of the various chemical bonds within the sample are represented by the resultant spectrum. We have used FTIR-ATR mode for the analysis.

Sample preparation: The membranes were cut into small pieces of 1 cm^2 size and dried well before analysis and kept in desiccator.

3.2. Tensile strength measurement

A Universal Testing Machine applies a controlled and measurable force upon the material to ascertain its mechanical properties, like tensile strength, elongation and modulus of elasticity. Conducting a tensile test on a membrane, the universal testing machine generates a uniaxial tensile force that increases until the membrane fails. Force applied and corresponding elongation are recorded with the machine during testing.

Sample preparation: The membranes were cut into rectangular pieces of 1 cm× 10 cm prior the analysis.

3.3. Contact angle

The contact angle is a measure of membrane wettability by the liquid. It is the angle formed at the interface where the liquid droplet meets the membrane surface. The underlying principle is the balance of forces acting at the three-phase boundary between solid, liquid and gas (generally air). The contact angle gives information concerning the surface energy of the membrane and the interaction of the membrane with water. It is one of the methods used in assessing properties such as hydrophobicity or hydrophilicity of surfaces.

Sample preparation: The membranes were cut into small pieces of size 2cm ×3cm and dried prior the analysis.

4. Methods

4.1. LDH Synthesis

The Layered Double Hydroxide (LDH) was synthesized by the most used traditional method via coprecipitation method [26-29]. Metal salt of copper (II) nitrate trihydrate and iron (III) nitrate nonahydrate were taken for the synthesis of LDH and a concentration of 0.15M (3.624gm) and 0.05 M (2.02gm) were prepared in 100 mL deionized water respectively. Then 0.1M (0.6gm) and 0.2M (3.1797gm) of NaOH and Na_2CO_3 were prepared in 150 mL deionized water. Both the solutions were stirred properly about 15 minutes using magnetic stirrer at 600rpm in room temperature. After that, the aqueous solution of the metal salt was added into the alkaline solution dropwise and stirred properly for almost 2 hours at 600 rpm. A constant pH 11 is maintained to speed up the co-precipitation. The precipitate is aged at room temperature for a minimum 24 hour and then washed with deionized water several times until the solution becomes neutral.

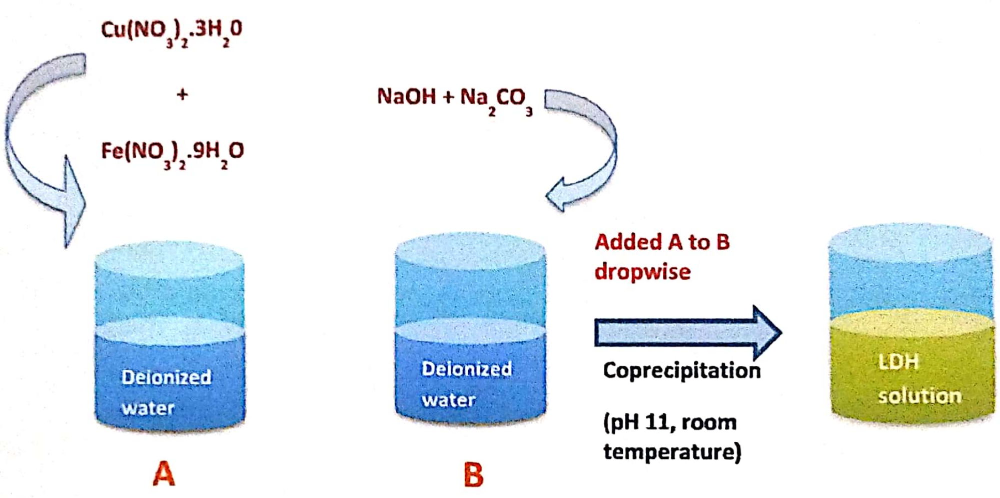


Figure 1: Schematic diagram for preparation of LDH solution

4.2. Preparation of Polymeric Solution

To make the polymeric solution,23% of polysulfone was dissolved in 40mL N-methyl-2-pyrrolidone (NMP) in a reagent bottle. The solution was stirred properly using magnetic stirrer at 600rpm for 8 hours at 50°C.

4.3. Membrane Casting

The membranes were prepared from the polymeric solution by the non-solvent induced phase separation technique (NIPS) [30-31]. In this technique, the polymeric solution was dispersed on a clean and dried glass plate and the solution was cast by using a specially arranged glass rod to maintain a constant thickness (10 cm) of the polymeric layer. After casting, the glass plate was immediately immersed in a non-solvent bath containing deionized water. Phase inversion occurs immediately and a thin solid polymeric membrane was obtained in water bath within a few seconds. After 1 hour the membranes were taken out from the water bath and then dried.

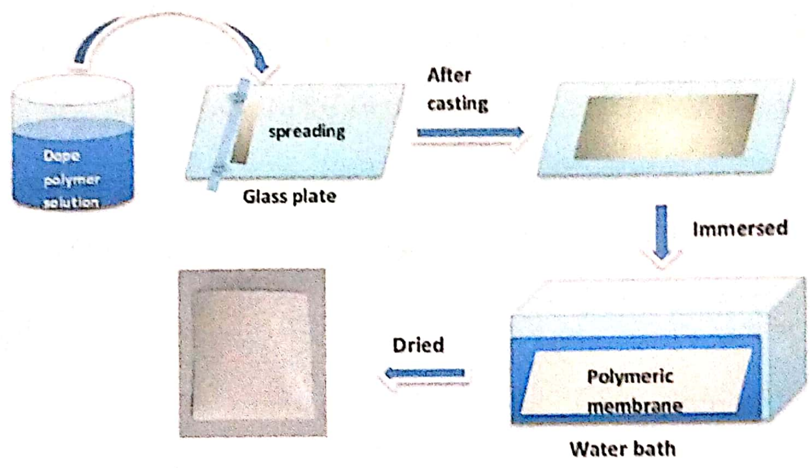


Figure 2: Schematic diagram of casting of membrane

4.4. Membrane Coating

Coating of membrane was done through interfacial polymerisation (IP) [32-34]. This technique involves reaction of two monomers, one in the aqueous phase and the other in the organic phase at the interface of the polymeric membrane. The aqueous phase was prepared by mixing 15 mL LDH aqueous solution (432 mg) with 85 mL deionized water in a beaker and stirred properly for 5 hours in a magnetic stirrer at 500 rpm. After that, 0.2 gm of m-phenylenediamine (MPD) is added into the aqueous solution and then stirred properly at 40°C for 6 hours at 350 rpm. And the organic phase was prepared from 0.5% trimesoyl chloride (TMC) solution. From 0.5 % TMC, 0.1 %

solution was made by dissolving 10 mL of 0.5 % solution in 40 mL n-hexane and then stirred for 2 hours using magnetic stirrer at 450rpm.Both the aqueous solution and the organic solution were poured in a petri dish separately. The membrane was first immersed in aqueous solution for 2 minutes and then dried for settling of the LDH particles. After that it was dipped in the organic phase for 1 minute and left for drying. The same procedure was repeated once again so that the LDH particles get incorporated into the polymeric membrane efficiently and this membrane was denoted as M1. Similarly, another membrane was fabricated by immersing four times both in the aqueous solution and in the organic solution for a time interval of 2 and 1 minutes respectively and it was denoted as M2. This process undergoes a special type of reaction known as Schotten-Baumann reaction where formation of polyamide bond takes place.

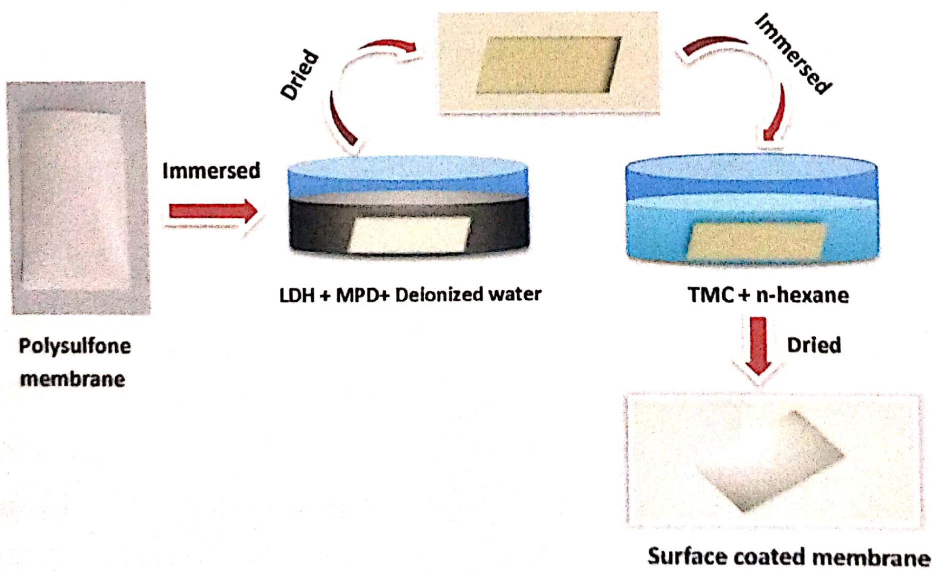


Figure 3: Schematic diagram of coating of membrane

4.5. Membrane's performance study

The membrane's performance study is analysed by using cross-flow filtration mode. For this a membrane with cross-sectional area 0.003314 m^2 is placed between the membrane cell.

The flux and % rejection of the membrane is calculated by using the following equation:

$$\text{Flux} = \frac{V}{A.t} \quad \ldots\ldots. \quad (1)$$

Where V is volume of permeate, A represents membrane area, t is time. Flux is determined in the unit $L.m^{-2}.h^{-1}$.

The rejection is calculated by using equation 2.

$$\text{Rejection (\%)} = \frac{C_f - C_p}{C_f} \times 100 \ldots\ldots. \quad (2)$$

where C_p and C_f represent concentration of permeate and feed (mg/L) respectively.

The feed used for the permeation study is 1200 ppm solution of magnesium sulphate. Magnesium sulphate is a common salt that is found in the sea water whose removal is necessary in desalination of sea water.

5. Results and discussions:

5.1. Characterization of the CuFe-LDHs: FTIR Analysis

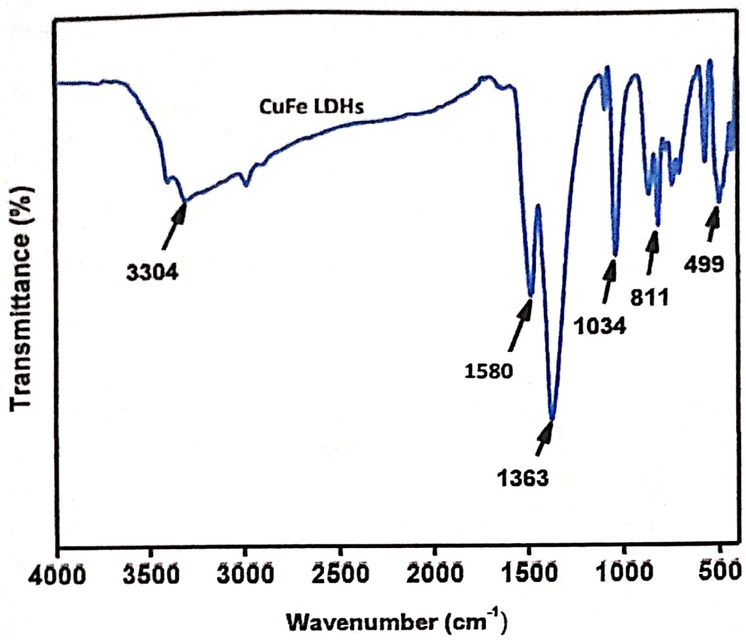


Figure 4: FTIR spectra of CuFe-LDH

The synthesized nanoparticle is characterized with FTIR analysis. The spectra obtained have been depicted in Figure 4. The broad peak 3304 cm^{-1} is ascribed to the presence of OH bonds in the LDHs. Similarly, the peak at 1580 cm^{-1} is for the C=O stretching obtained for the interacted carbonate ions; peak at 1034 cm^{-1} is due to C-O stretching. Peak at 811cm^{-1} and 499 cm^{-1} are due to Cu-O stretching and Fe-O stretching respectively. Hence, from the FTIR spectra the successful synthesis of CuFe-LDHs can be validated.

5.2. Characterization of the membranes: FTIR analysis

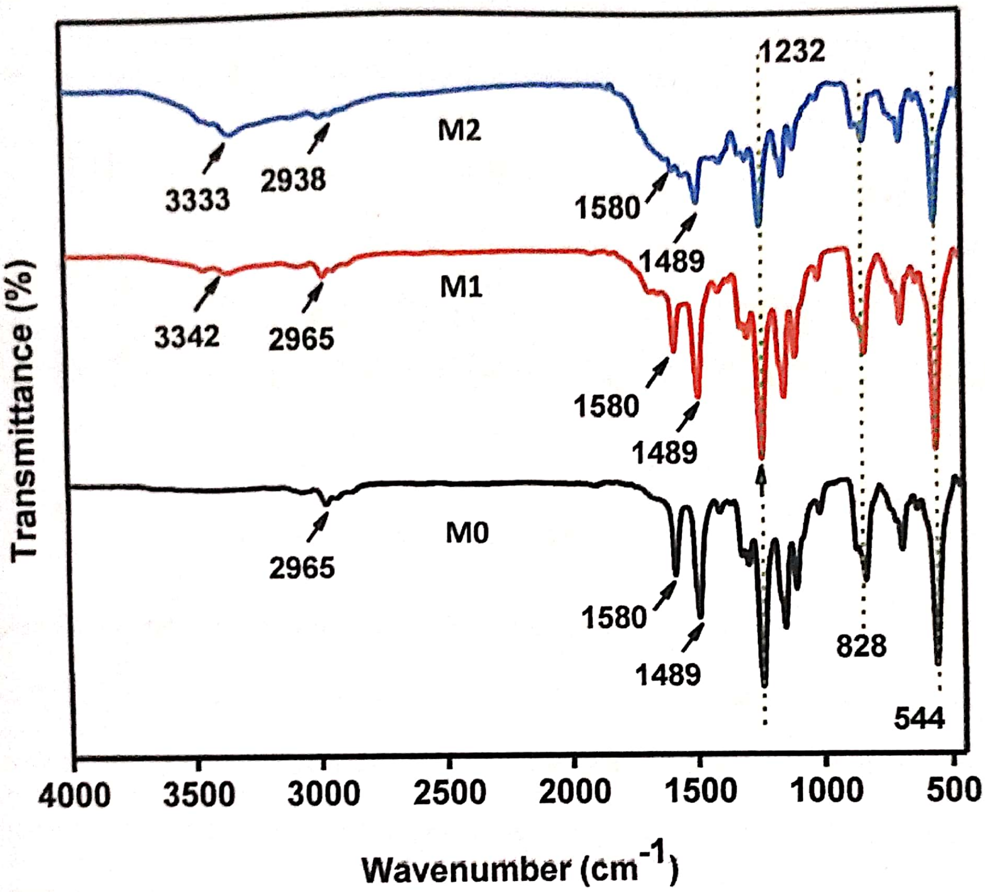


Figure 5: FTIR spectra of the membranes

The FTIR spectra obtained for all the designed membranes have been depicted in Figure 5. The peak at 2964 cm^{-1} is ascribed due to the presence of C-H stretching; 1580 for C=O stretching, peak at 1232 cm^{-1} is for S=O stretching, 1489 cm^{-1} is for aromatic C=C. Due to absence of O-H bond we have not observed any such peak.

However, in case of M1 and M2 due to presence of LDH broad peaks at 3342 cm^{-1} for M1 and 3333 cm^{-1} M2 is observed. Similarly, two distinct peaks near 3000 cm^{-1} confirms the presence of

N-H bond due to formation of polyamide layer. This confirms the successful design of the membranes M1 and M2.

5.3. Characterization of the membranes by contact angle analysis:

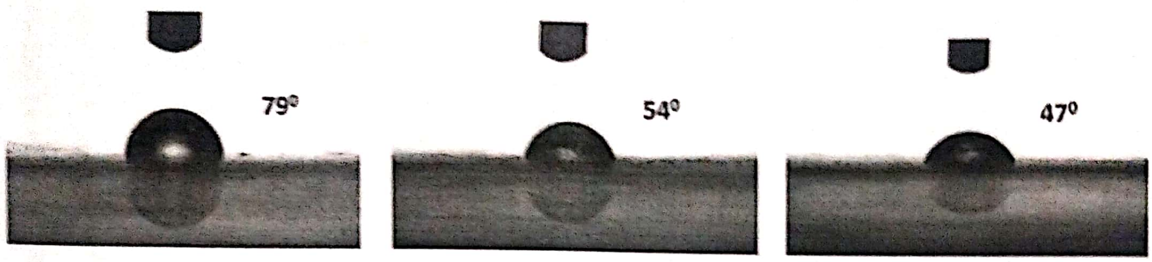
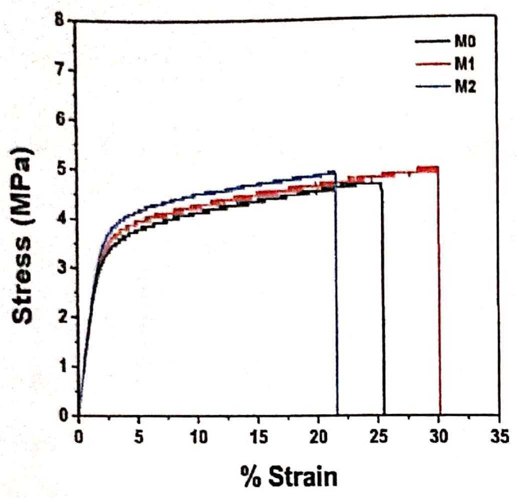


Figure 6: Contact angle analysis of the membranes

To analyse the wettability (hydrophilicity or hydrophobicity) of the membranes water contact angle analysis is employed. It is observed that from M0 to M1 the water contact angle decreases which indicates the increase in the hydrophilicity of the membranes upon addition of CuFe-LDHs. Due to hydrophilic nature of the LDHs it has impacted the wetting ability of the membranes also. It is important to induce hydrophilicity in membranes used for water treatment. A membrane with more hydrophilicity will exhibit more flux and less fouling as compared to a less hydrophilic membrane. Hence, it is one of the major successes in this study.

5.4.Tensile strength measurement of the membranes:



Membrane	Ultimate stress (MPa)
M0	4.67
M1	4.91
M2	4.94

Figure 7: Stress-strain curves of the membranes

Tensile strength measurement of the membrane is also done for analysing the mechanical stability of the designed membranes. As we can see in the Figure 7 the ultimate stress of the membranes is increases from M0 to M2. This is due to the formation of polyamide layer and successful incorporation of LDHs over the membrane. In terms of mechanical strength all the designed membranes show very high stability. Hence, the fabricated membranes can be useful in harsh condition of pressure also.

5.5. Performance evaluation of the membrane using cross flow experiment

The best membrane M2's performance is analysed by employing cross-flow filtration method. The cross-flow permeation of the membrane is performed applying a pressure of 1 bar at 158 rpm. The cross-sectional area of the membrane used for the permeation is 0.003314 m^2. The performance of the membrane was carried out continuously for 6 hours and the permeate was collected for every

1-hour interval respectively. The selectivity of the membrane is analysed measuring the conductivity of the feed solution and the permeate collected in each hour. The results have been presented in Table 1.

Table 1: Flux and % rejection of the membranes

Time (hour)	Flux ($L.m^{-2}.h^{-1}$)	% rejection
1	7.54	56.76
2	7.09	54.14
3	6.63	55.35
4	6.48	57.67
5	6.03	55.56
6	5.12	55.46

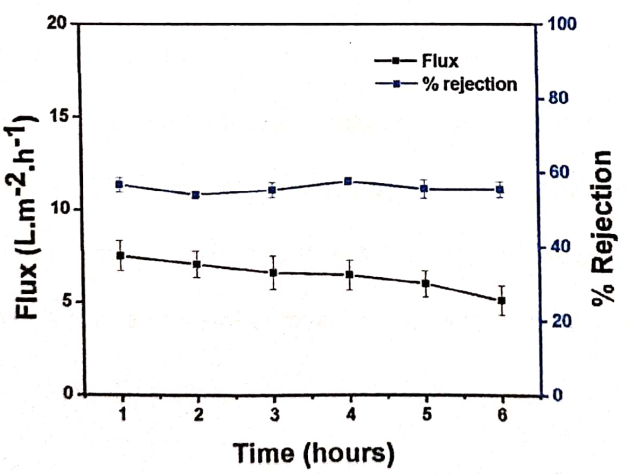


Figure 8: Performance analysis of the membrane.

It is observed that the flux of the membrane gardually decreaes which indicates the accumulation of the cations and anions on the membrane pores as well as LDHs interlameller spaces. However, we have not observed any drastic change in the % rejection of the membrane. The best rejection of 57.67 % is obtained for removal of magnesium sulphate. Therefore, the designed membrane can be utilized in desalination of seawater.

6. Conclusion

Thus, it could be concluded that polymeric nanocomposite membranes are very efficient in the treatment of wastewater because of the enhanced unique characteristics like hydrophilicity, thermal and mechanical stability, permeability, targeted degradation, solute rejection, and magnetic, antimicrobial, and antifouling properties. Polysulfone, the polymer of our interest possesses high mechanical strength and chemically, thermally, and oxidatively more stable. Incorporation of nanoparticles provides membrane with immense properties of nanomaterials and induces the membrane performance as compared to pristine membrane. Fabrication of membrane with iron-copper LDH increases the hydrophilicity, water permeability and mechanical strength of the polymeric membrane. Hence, the synthesis of nanocomposite membranes for the treatment of wastewater has been successfully done. Moreover, the membrane shows stable flux of nearly 7 $L.m^{-2}.h^{-1}$ and rejection of nearly 57% which indicates its excellent desalination capability. In the future, the designed membrane can be further modified for use in seawater desalination.

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Photo Gallery

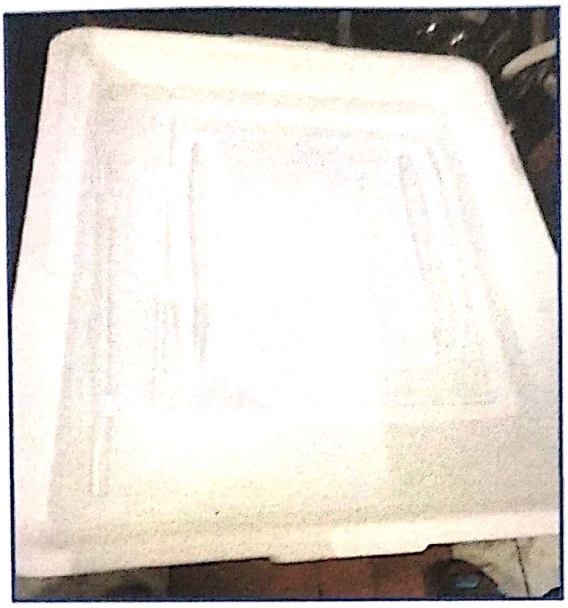

Photo 1: Preparation of membrane via NIPS

Photo 2: LDH solution

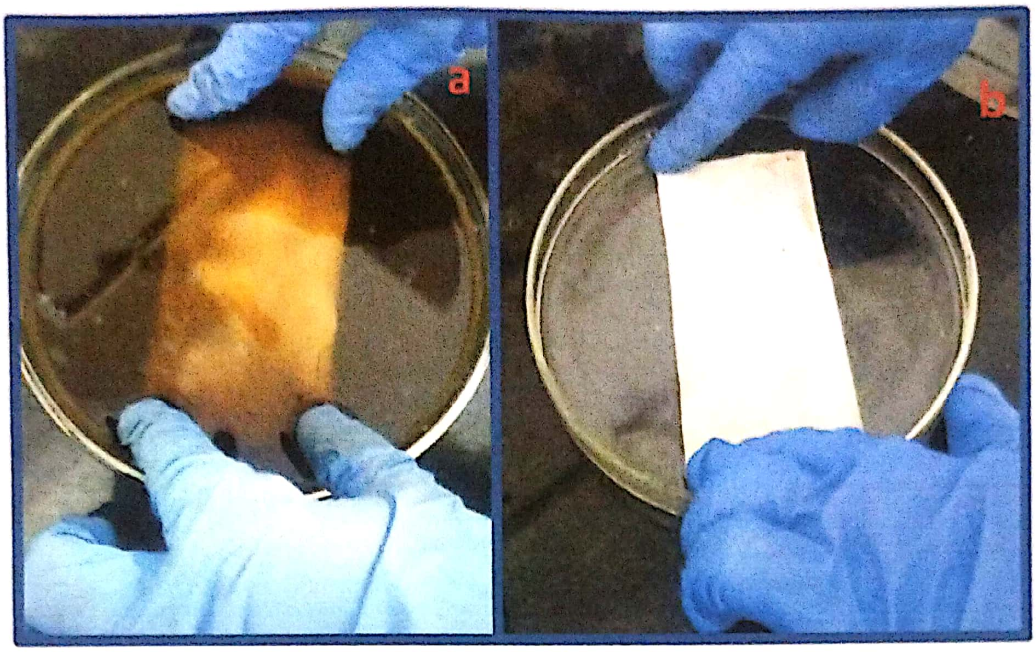

Photo 3: a) Membrane immersed in aqueous solution; b) Membrane immersed in organic solution

Pristine membrane	2 times LDH fabricated membrane	4 times LDH fabricated membrane

Photo 4: Membranes fabricated via interfacial polymerization.

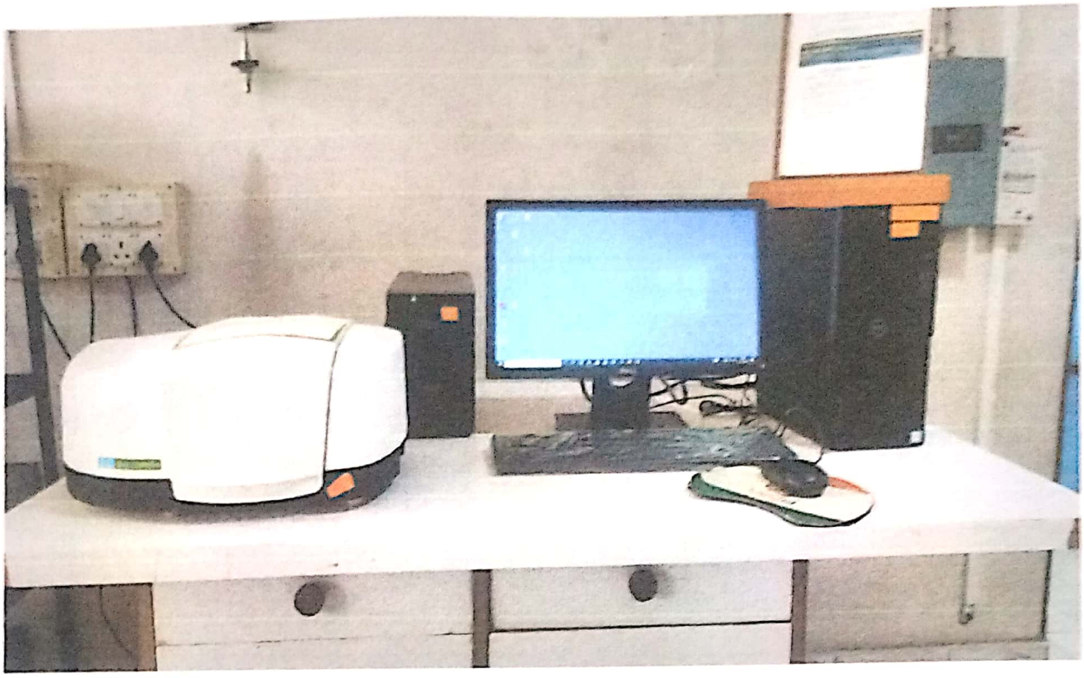

Photo 5: FT-IR Instrument

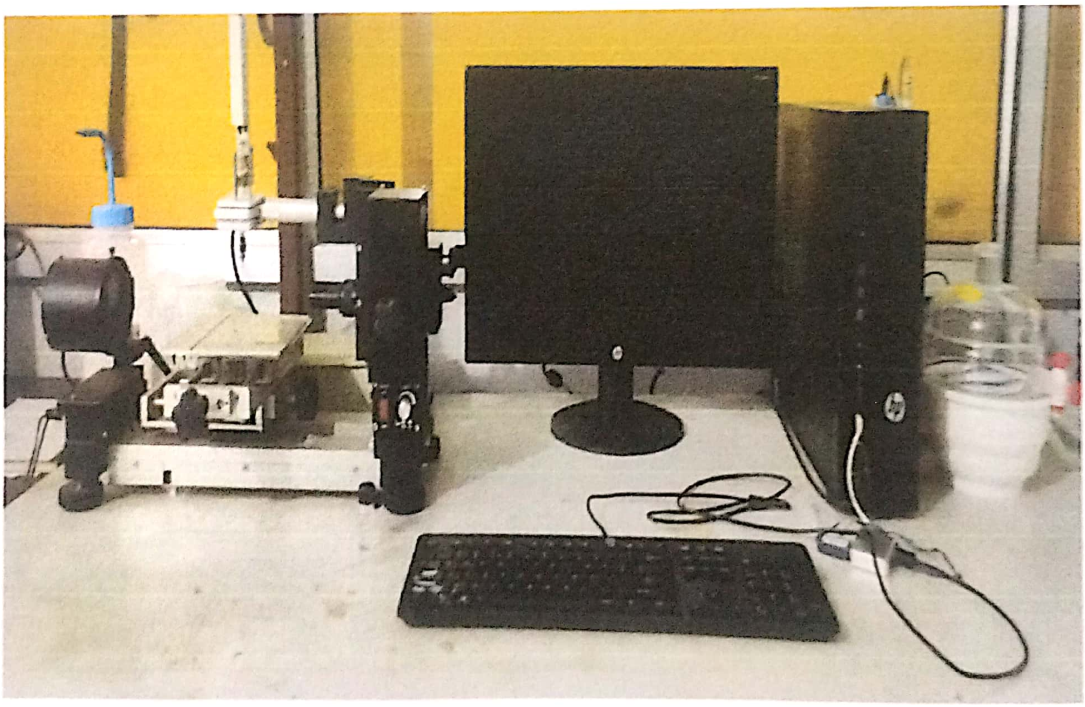

Photo 6: Contact angle measurement instrument

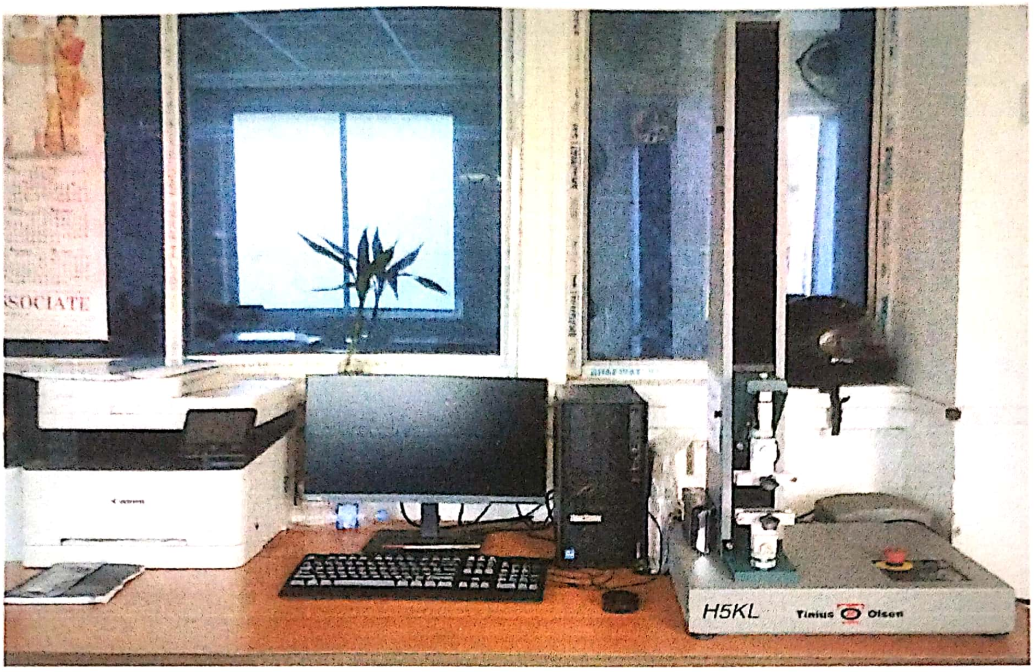

Photo 7: Tensile strength measurement instrument

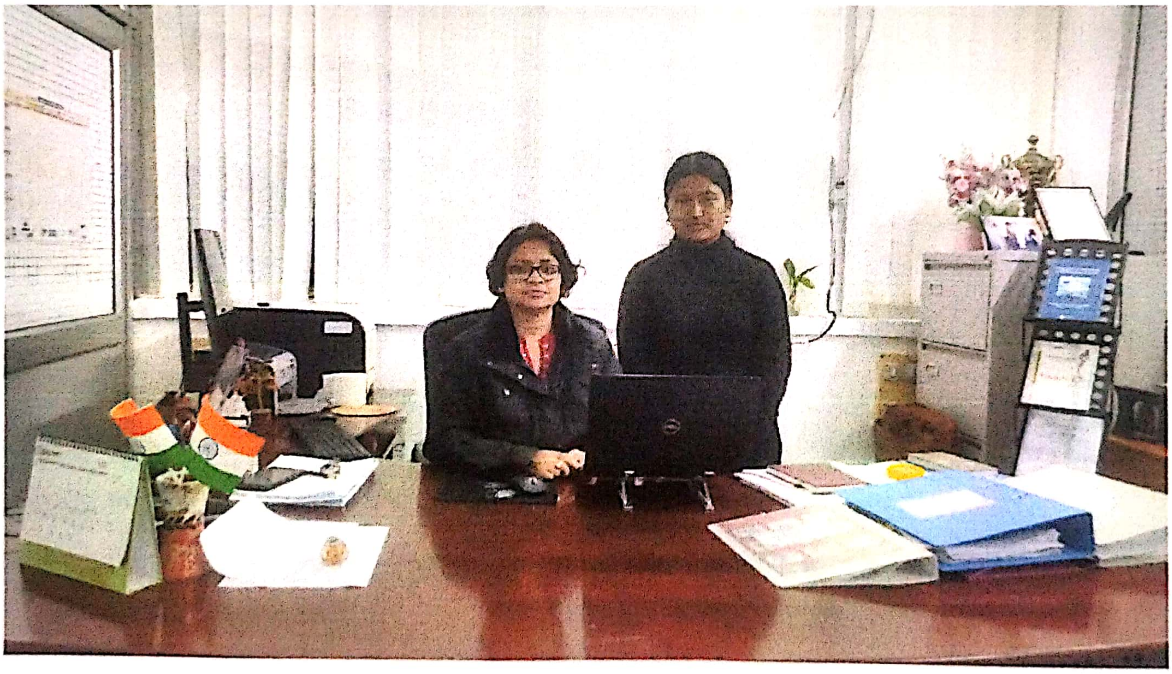

Photo 8: With Supervisor Dr. Swapnali Hazarika Ma'am.

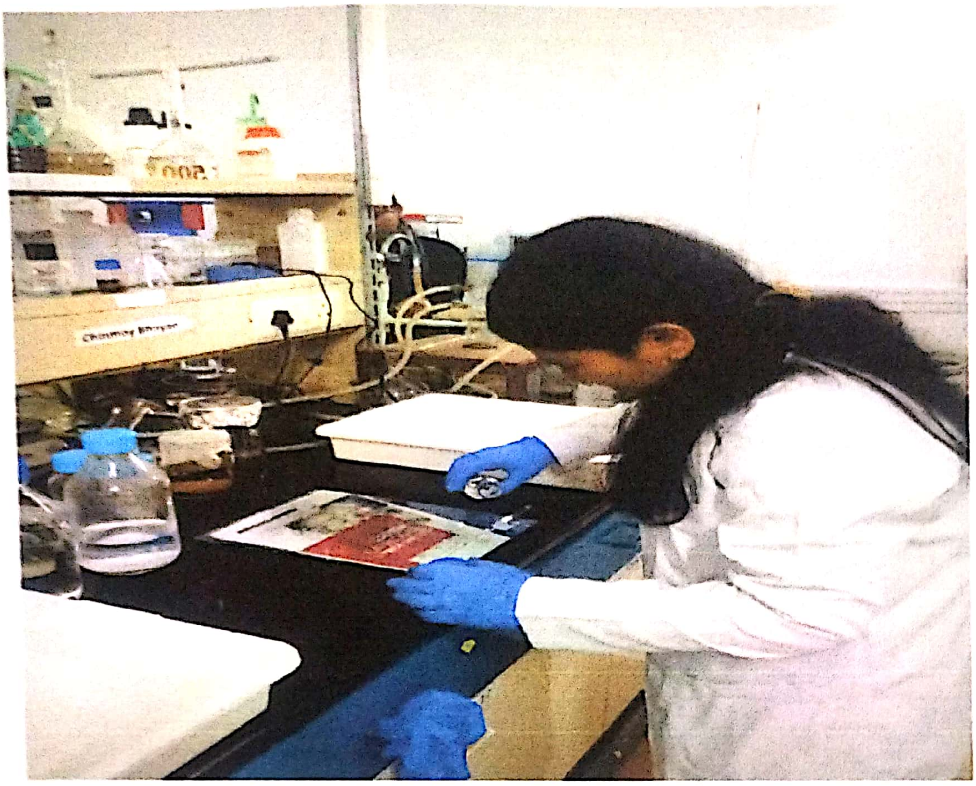

Photo 9: Dispersing polymeric solution in the clean and dry glass plate

Photo 10: Immersing the glass plate into the water bath

Photo 11: During fabrication of pristine membrane