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Research Article

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Preparation and characterization of low cost mixed matrix membrane for wastewater treatment

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ABSTRACT

Nowadays the treatment of wastewater is must due to modern Urbanization and Industrialization. We have a plenty of techniques to treat these kinds of wastewater. Even though the conventional waste water cleaning processes literally outsmarted in treating a relative high amount of pollutants, only a little amount of dye and oil were effectively treated so far, where which the treated effluent lacked the claimed purity in various standards. Membrane technology is one of the major and recent advanced techniques in the treatment methods. We have polymeric, ceramic or ceramic incorporated polymeric types (Mixed Matrix types) for a better filtration process in wastewater management. Since a century, the phase inversed membranes are established as a predominant component in a regime of solid-liquid separations. It is proven that if done with constant improvements in the material combinations and other required conditions such as temperature, setting time etc., it is possible to eradicate the lags inherent in the membranes of today. The work encompasses all the details beginning with the ceramic incorporated polymeric membrane preparation, its characterization and the test followed with the textile effluent samples to evaluate the efficiency of the formulated membrane highlighting its future prospects.

Key words: Textile effluent, Effluent treatment, Membrane Filtration, Nanofiltration, Ceramic membrane

INTRODUCTION

Membrane technology covers all approaches for the transport of substances between two fractions. In general, mechanical separation processes for separating gaseous or liquid streams use membrane technology. The application of membrane science has been in prior use since the late nineties. The wide spectrum is now a subject to continuous modulation and improvement in search of better prototypes development for various types of filtration in various fields of effluent management. In near future, the emergence of new type of membrane materials and simple fabrication techniques may lead to preparation of low cost membranes.

The usage of homogeneous membranes is of limited range due to its higher cost and fouling nature. The new researches are to find out the better blend of ceramic materials on the polymeric skeleton to study the probabilities of flux improvement, porosity and effluent management. This work focuses on the post effects and their study after the incorporation of ceramic feldspar in the asymmetric polymeric membrane matrix and the studies on the extension of filtration the fabricated micro porous membrane offers in the wide stream of textile effluent management. In this research work, effort has been given in utilizing a liquid textile effluent for gainful.

A large quantity of oily colored phenolic effluent are generated from various process: dyeing and textile sectors day by day due to commercial processing. It is highly essential to separate the toxic contaminants before being discharged into a municipal sewage system to protect the environment by maintaining government regulations. Additionally, it might yield some value added product. Conventional methodologies like evaporation, gravity settling, reverse osmosis etc. take longer time between batches, requires large spaces and higher logistical supports. Membrane technology on the other hand follows a compact design and relatively low operational costs. The prepared module in the thesis work is seen to be a small model of the bigger module that is expected to be used in waste water management in the future.

1.1 Textile Sector – An Overview

Textile or cloth is one among the basic necessity of human being. The textile industries therefore have great economic significance by virtue of its contribution to the overall industrial output to meet the population and trending needs and employment generation of a country. This sector has wide spectrum of industries ranging from small scale units that uses traditional manufacturing processes to large integrated mills using modern machineries and equipments. According to a recent survey that focuses on composite and process houses, so far there are 2324 textile industries in India.

1.2 List Of Harmful Chemicals Used In Textile Industries

1. Detergents mainly nonyl-phenol ethoylates : generates toxic metabolites that are poisonous to aquatic flora and fauna

2. Stain remover: carry solvents like CCl4; causative constituent in ozone depletion.

3. Oxalic acid used for rust stain removal: are toxic to aquatic organisms and also boots COD.

4. Sequestering agents: Polyphosphates like Trisodiumpolyphosphate and sodium hexametaphosphateare banned chemicals.

5. Printing gums: preservatives like pentachlorophenol can cause dermatitis, liver and kidney damage for which they are all restricted in processing sector.

6. Fixing agent: Formaldehyde and Benzidine that are banned internationally due to their toxicity.

7. Bleaching: Chlorine, that aids the bleaching operations causes itching and are harmful to skin.

8. Dyeing: Azodyes that release amines are banned due to their carcinogenic nature.

A discharge with these pollutants in a reasonable extend when discarded into the water bodies turns fatal unless properly treated before its disposal. The existing conventional waste water treatment is mostly done by primary and secondary processes. However, these treatment systems are not very effective in the removal of pollutants such as dissolved solids, color, trace metals etc. The replacement of these classical methods by advance treatments pave an alternative for the reduction of the pollutants to a favorable extend at first hand and give scope for recovery - recycling of water and the chemicals.

The table 1 given below outlines the permissible limits of various prominent contaminants discharged from the textile sectors of India.

PARAMETER	PERMISSIBLE LIMIT-CPCB (mg/l, except pH)
Ph	5.5-9.0
Bio-chemical oxygen demand (BOD)	30
TSS	100
Chemical oxygen demand (COD)	250
Chloride	350
Dissolved Oxygen	6.52
Conductivity	98
Sulphate	20-403
Zinc	0.5-0.05

Table 1.1 Effluent standards for discharge into streams

2. EFFLUENT TREATMENT METHODOLOGIES

2.1 Primary and Secondary Treatment

The conventional treatment systems like physico-chemical treatment followed by biological treatment are the most common installations in many textile industries. Different stages of primary and secondary treatment are:

- 1. Mixing.
- 2. Equalization.
- 3. Screening.
- 4. Clarification.
- 5. Clariflocculation.
- 6. Filtration.

7. Advanced treatment methods like adsorption, Ion exchange, and membrane filtration.

2.2 Ceramic Incorporated Polymer Membrane (Advanced Membranes)

Apart from the homogeneous membranes for the conventional waste water filtration and textile effluent management, a limited research articles also highlight the preparations of advanced membrane types, a combination of hydrophobic and philic counter parts to attain the required properties of fluxing and surface wet ability. Blending a polymer matrix with hydrophilic components during the phase inversion process is an effective method for advance membrane prototypes that can be easily achieved, and this process has been well investigated. In this process, hydrophilic polymers, amphiphilic copolymers and inorganic nanoparticles as the three main types of additives have been extensively employed to modify the porous filtration membrane. Although the effects of different additives on the membrane performance differ, the main objective of blending is to enhance the membrane permeability and antifouling property. Especially for the application of purification of oily wastewater, polymer-dominated membranes must be more hydrophilic to prevent the adhesion of oil droplets on the membrane surfaces.

Many studies have demonstrated the effectiveness of blending of these hydrophilic components into polymeric membranes in improving membrane performance. However, because of the water-favoring property of these additives and the weak interaction between the additives and the polymer matrix, the additives tend to release from the membranes during long-term use. Therefore, the stability of the blending membranes remains an issue that needs to be addressed. In addition to various polymers, inorganic particles, such as Al2O3, TiO2 SiO2, feldspar and Quartz have also been used to blend with host polymers.

It is well known that these particles have large surface areas and abundant surface-active groups. The additives of inorganic nature have been demonstrated to be helpful to membrane permeability and fouling resistance by either changing the pore structure or increasing the hydrophilicity of the membranes. A simple and commonly used approach is to directly blend pre-prepared particles with polymers in solution.

2.3 Phase Inversion And Asymmetric Membrane Preparation

Phase inversion refers to the process in which a homogenous solution of a polymer in a solvent (or solvent mixture) inverts from a single phase into a two-phase system. The two-phase system consists of a solid (polymer rich) phase which forms the membrane structure and a liquid (polymer-poor) phase which forms the pores in the final membrane. Phase inversion membranes can be prepared from any polymer-solvent mixture which forms a homogenous solution under certain conditions of temperature and composition, but separates into two phases when these conditions are changed. For example, phase inversion can be induced by the evaporation of a volatile solvent from a homogenous polymer solution, or by cooling a casting solution which is homogenous only at elevated temperatures. Phase separation can also be brought about by the introduction of a third component (non solvent) into a homogenous polymer solution. This particular variation is referred to as the wet phase inversion technique. The general procedure for the preparation of a wet phase inversion membrane is as follows.

(a) An amorphous polymer is dissolved in an appropriate solvent to form a solution containing 10 to 30 weight% polymer.

(b) The homogenous polymer solution is cast into a thin film. The film is typically 100 to 400 (micrometer in thickness)

(c) The film is then quenched in a coagulation bath to precipitate the polymer membrane out of the solution. The coagulation medium is typically water, which acts as a strong non solvent for most of the polymers used in the preparation of these membranes.

In some cases, the quenching step is preceded by a short evaporation period. However, the principal structureforming processes which influence the transport properties of these membranes occur in the quenching step, during which nonsolvent exchange takes place followed by various phase transformations in the polymer film.

Membranes with different morphologies and, different transport characteristics can be obtained by the use of various combinations of polymer, solvent, nonsolvent and processing conditions. During the phase inversion process, the combination of steps leading to a given membrane structure involves a complex interaction of thermodynamic and mass transfer processes. However, in all cases, the basic membrane formation mechanism is governed by similar thermodynamic and kinetic parameters, such as the chemical potentials and diffusivities of the individual components and the Gibbs free energy of mixing of the system. An accurate description of the thermodynamic and kinetic phenomena occurring during the phase separation process is vital to understand the membrane formation mechanism and, therefore, to optimize the membrane structure and transport properties.

EXPERIMENTAL SECTION

3.1 Chemicals Used

Polysulfone, N Methyl Pyrolidone, Potash Feldspar (bulk), Alginic Acid, Sodium Lauryl Sulphate, Effluent Water are used in the experiment.

3.2 Fabrication of Membrane

The modular preparation of the filter medium is done keeping in a detailed view on the literature accessed on the given headings. The raw materials used constitute Polysulfone as the polymeric counterpart and feldspar as the ceramic component. Binary solutions were prepared by the dissolution of PSU (Polysulfone) in NMP (N-Methyl Pyrolidone) at room temperature and ternary dispersions were obtained by the addition of different amounts of feldspar (from 0.25 to 1 wt %). A measured quantity of alginic acid say 0.04375 gm is added to control the porosity of the expected asymmetric matrix.

Initially 3g of alginic acid is dissolved in 75 ml of distilled water to obtain a homogeneous solution which is in turn added with various ranges of polymer sample (100-98%) and ceramic (0.25-1%). An amount of 4.375 g of the given polymer alone is added to the alginic acid solution to prepare a polymer alone or homogeneous type membrane on the first hand followed by 5 other samples with polymer mass ranging 4.33125, 4.3203, 4.3094, 4.2984, 4.2875 and quantities of feldspar ranging 0.01094, 0.021875, 0.03281and 0.04375gm to study the effect of the ceramic fluxing agent addition in the properties of polymeric membrane.

The 4 samples hence prepared are added with equal amount of solvent (20 ml of N-Methyl Pyrolidone) and is kept for 1 day to facilitate homogeneous dissolution. On the completion of one day the samples are thoroughly examined for its homogeneity and solubility extend. The fully dispersed solutions are hence subjected to precipitation casting where a solvent non solvent exchange is undertaken thermodynamically.

The solution and dispersion are first cast onto a glass plate to form a thin film that in the case of membrane preparation, after a 30-s exposure period in a controlled environment (60% relative humidity) are immersed in water bath holding 3g dissolved Sodium Lauryl Sulfate in it to induce precipitation of the polymer and porous membrane formation. After a 5-min immersion period, the membranes are detached from the plate and leached overnight underwater and is then preserved for future use using 0.1% formalin before characterization.

Table given below shows the various compositions of the 4 sample asymmetric membranes prepared by phase inversion method

DOI VMEP		ADDITIVES					
CAMDLE	POLIMER		ALGINIC ACID		FELDSPAR		SOLVENT
SAMPLE	PERCENT	WEIGHT	PERCENT	WEIGHT	PERCENT	WEIGHT	(ml)
	(%)	(gm)	(%)	(gm)	(%)	(gm)	
А	99	4.33125	1	0.04375	0	0	20
В	98.75	4.3203	1	0.04375	0.25	0.01094	20
С	98.5	4.3094	1	0.04375	0.5	0.021875	20
D	98	4.2875	1	0.04375	1.0	0.04375	20

Table 3.1 Formulations for membrane A, B, C and D

3.3 CONDITIONS APPLIED FOR MEMBRANE PREPARATION

The table given below details the optimum conditions that are required to prepare in lab the membrane of size 25 gm.

Table 3.2 Film casting conditions

CONDITIONS	RANGE
Temperature of casting solutions (0C)	28 ± 2
Temperature of casting atmosphere (0C)	24±1
Humidity of casting atmosphere (0C)	55±2
Period of casting (s)	3–6
Solvent evaporation time (s)	30±5
Temperature of gelation bath (0C)	12±2
Period of leaching membranes in gelation bath (h)	1–3
Thickness of film on glass plate (mm)	0.22±0.02

3.4 THE PREPARED MEMBRANE

Figure given below shows the prepared membrane kept for phase inversion in water bath



Figure 3.1 Asymmetric membrane prepared

4. MEMBRANE CHARACTERIZATION

The surface morphology study was done by SEM analysis for the prepared samples of 4 that varies in the amount of ceramic counter part in it.

4.1 PERMEABILITY AND FILTRATION TESTS

The spectrum of membrane science and its quality study relies on the outcomes and results of the filtration and the permeability tests performed with the specific sample and the literature comparison .The efficiency of a membrane is a function of its separation efficiency that is an indication of its future prospects. Here the 2 important experiments conducted to study the permeability extend and the filtrating capability is discussed in detail. The filtration experiments were carried out in a batch type dead end cell (UF cell-S76-400-Model, Spectrum, USA)with a diameter of 76 mm, fitted with Teflon coated magnetic paddle.

4.2 COMPACTION

The prepared membranes were cut into desired size needed for fixing it up in the ultra filtration kit of 37.5 cm2 area and initially pressurized with distilled water at 4000 kPa for 15 minutes. The water flux was measured at every one hour. The flux generally declines initially and attained steady state with the increase in time. The pre pressurized membranes were used in subsequent ultra filtration experiments at 4100 kPa for a 10 minute operation.

4.3 PURE WATER FLUX STUDY

Membranes after compaction were subjected to pure water flux at trans- membrane pressure of 345 k Pa. The flux was measured under steady state flow i.e., after every 1 h for 4 h. The pure water flux was determined using the given equation

ie.
$$J_W = \frac{Q}{A.\Delta T}$$

Where, Jw is the water flux (lm-2 h-1), Q is the quantity of permeate (l), A is the membrane area (m2) and DT is the sampling time (h).

4.4 FILTRATION STUDY

Separation experiment was performed using the permeation set up shown below using UF cell-S76-400-Model, Spectrum applying dead end cross flow filtration technique.

Feed solution was carefully poured and whole module was perfectly sealed using Teflon tape to avoid leakage of pressure and liquid. For the experiments, liquid feed was pressurized using compressed air from compressor. Pressure was maintained at throughout the process. To assess the quality of separation, quantitative estimation of the effluent important parameters were done before the operation and the permeates was checked at regular intervals taking spectrophotometer reading and corresponding time was noted down. Sampling was performed at regular time interval in order to ascertain if there was any variations of quality of permeate obtained with respect to time. Permeate volume was also noted to find the variation of flux with respect to time since possibility of chocking of

membrane due to accumulation couldn't be ruled out. It should be noted that the experiments were performed in batch process with no recycle.



Figure 4.1 Cross Flow dead end ultra filtration module used for the permeability and filtration tests

4.5 MEMBRANE RECOVERABILITY

Attempts to recover the support and membrane were pursued by back washing and rinsing with de-ionized pure water at room temperature several times to retrieve back the membrane efficiency. Performance of the recovered membranes was checked by performing hydraulic permeability studies once again to determine the flux.

4.6 PRE AND POST ANALYSIS OF EFFLUENT WATER

Both samples before and after treatment were segregated and stored in a unit which was further subjected to parameter analysis. The specific conditions to avoid sample fouling were strictly maintained. The parameters studied in the work comprises conductivity, Zinc presence, DO range, Ph ,BOD(Biological Oxygen Demand),COD (Chemical Oxygen Demand),TSS(Total Suspended Solids, Sulphate and Chloride content which were found to be in higher amount from the Textile industrial sectors of India.

The conductivities were measured by a PHWE conductivity meter. TDS of all samples were measured by taking 10 ml 0f each sample in a watch glass and drying by keeping in an oven. For COD the sample was refluxed with Potassium Dichromate and Sulphuric Acid in presence of Mercuric Sulphate and Silver Sulphate .The excess of Potassium Dichromate was titrated against Ferrous Ammonium Sulphate using Ferroin as an indicator. The amount dichromate used is the equivalent of the oxidizable organic matter present in the sample. pH indication is done by the Electrometric method, Chlorine by lodometric method, Sulphate turbidimetric method and Zinc by ICP AES.

RESULTS AND DISCUSSION

Morphology of the blend membranes were analyzed by SEM with different Ceramic compositions ranging from 0 to 1 percent from membrane A to D and the results are depicted in Figs.4.1 and 4.2. At 0 wt.% of feldspar, membrane exhibit smaller pores distributed evenly rather more adjacent and traces an average pore size in 0.6 to 0.8μ m. As, the ceramic feldspar composition is increased in the casting solution, the pore size increased proportionally in the way 0.86, 1.23 and 1.45μ m.

The cross-section of membrane confirms the asymmetry in the infrastructure of the membrane. The addition of the ceramic constituent greatly reduced the formation of macro voids. The presence of the low range micro voids in the surface can be considered as an indication of the nano-porosity in the membrane structure if traced down to bottom which can hence contribute for a blend of micro nano-porous asymmetric membrane structure. The nano porosity underneath is a promising factor for the further filtration experimentations.



Figure 5.1 SEM images of membrane A (0 % Feldspar) and B (0.25 % 0f feldspar)

Figure 5.2 SEM images of membrane C(0.50 % Feldspar) and D (1.0 % 0f feldspar)



5.1 Pure Water Flux Test Or Permeability Test

The values of pure water flux are presented in the table. The water flux increases from 9.132 to 45.662 l m-2 hr-1. This apparent linear trend due to the increase in ceramic content in the blend may be due to the hydrophilic nature of the ceramic group present in the membranes as well as the efficient fluxing property of the bulk potash feldspar.

Table 5.1	Permeability	data -	pure	water	test
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SL NO	MEMBRANE	VOLUME OUT FLOWED (× 10-3) (l)	DURATION (\Deltat)(hr)	FLUX (<i>l m-2 hr-1</i>)
1	A (0% feldspar)	2.5	0.073	9.132
2	B (0.25% feldspar)	10	0.073	36.52
3	C (0.5% feldspar)	10.5	0.073	38.356
4	D (1% feldspar)	12.5	0.073	45.662

The asymmetricity of nano and micro controls the liquid flow and extend of filtration through the prepared membrane that increases its efficiency. The average permeability value is ascertained to be equivalent to 40 (lm-2 hr-1) which is at a great difference from the polymeric dominated membrane permeability (say the 1 sample).

5.2 Pre And Post Analysis Of Effluent Water

The details on the various impurities, the prior contaminants, toxicants etc., were found out from literature and the ranges were studied thoroughly. Analyses of the derived important parameters before and after the treatment are given below to come up with the comparative data regarding the membrane efficiency. The post analysis value is the average value of the analysis data on the water sample flowed out after treatment with the sample D membrane.

The table 4.2 given below shows the pre and post analysis data of the effluent water sample taken from the industry.

Sl. NO	Parameters	Water Analysis before treatment (mg/l)	Water Analysis after treatment (mg/l)
1.	Conductivity	700	150.54
2.	Zinc	38.3	14
3.	Dissolved Oxygen	6.52	2.45
4.	pH	9.0	7.97
5.	BOD	560	25
6.	COD	312	60
7.	Total Suspended solids	16	11
8.	Sulphate	1400	200
9.	Chloride	1170	500

Table 5.2 comparison before and after effluent water analysis

From the above given data it is observed that there is a drastic decrease in the value of BOD, COD, TSS, DO, Sulphate and Chloride value before and after treatment. The value of pH is balanced to the neutral level with a slight decline in alkalinity of the sample solution. Comparing with the stringent recommendations and regulations of the Central Pollution Control Board regarding the permissible level of the contaminants in the effluents, the values obtained for BOD, COD, pH, DO and TSS are quite promising. Whilst the other values can vary if the process is subjected to proper improvement that can in turn to be worth advantageous in the treatment of textile water.

When dealing with the efficiency factor of the membrane the values except Zn can be worth considered to state the above satisfactory performance of the membrane. The low cost membrane the blend of the ceramic with the polymeric is a new idea in the spectrum of membrane filtration.

5.3 Cost Analysis Of The Membrane

The given table 5.3 depicts the amount spend for the modular preparation of the membrane. From the given data it is clear that the membrane prepared, the lab scale prototype is an example of a low cost efficient multi dimensional filtration module. The average cost of the membrane from the individual market price of the constituents is Rs 340.

Sl No:	Raw materials	Weight (gm)/ml	Unit price(Indian Rs)
1	N-Methyl Pyrolidone	20 ml	38
2	Algenic Acid	3 gm	9.69
3	Sodium Lauryl Sulphate	3 gm	6.774
4	Polysulfone	4.38 gm	268.5
5	Feldspar	0.05 gm	0.1
6	Formalin(preservative)	500 ml	8.968

CONCLUSION

Filtrating membranes A,B, C, D were prepared from Polysulfone with the addition of a small amount of bulk feldspar in the hope to conduct a detailed study about the flux and porosity changes. The pure water permeability tests indicated the increase in flux with increase in the ceramic counter parts added and the SEM analysis for the 4 samples dictated the promising asymmetric arrangement; the lower range micro pores followed by nano pores underneath. It was observed that the porosity had a slight positive acceleration when the amount of the ceramic constituent added was increased. However the ranges were approximately same. Mean while the properties of hydrophilicity had a drastic effect due to the ceramic counterpart and raw material specialty. The filtration tests of the samples with the 4 membranes and the succeeded pre and post analysis gave out the spectrum of contaminants that the membrane is capable to manage along with its efficiency. The data of filtration of the 4 membranes were tabulated on an average basis in a single tabulation. A proper comparison on the CPCB limits was also taken into consideration to evaluate the performance of the prepared membrane.

The cost analysis relying on the market price gave out an approximate cost of the membrane fabricated. It is observed that the membrane prepared is cheaper than commercially available ceramic homogeneous membranes.

These results are the indications of the significant opportunity in future to develop these filtration membranes with flexible pore sizes for industrial treatment applications in the field of textile effluent management.

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