Volume : 5 | Issue : 6 | June 2016 • ISSN No 2277 - 8179 | IF : 3.508 | IC Value : 69.48

Friendly Membrane Preparation and Its Application Studies

Chemistry

KEYWORDS : Green membrane, Filtration, Physico-chemical parameters.

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ABSTRACT

The aim of the study is to prepare the green membrane by low cost and eco friendly method. The prepared membrane has been characterized by using FT-IR, SEM & TGA analysis. The green membrane has been applied to salty water filtration. The filtrated water is used to study the physico-chemical parameters. Most of the parameters correlated with the value prescribed by WHO.

Introduction

An increasing scarcity in fresh water sources fuelled a push towards alternative resources such as ocean water. In the 1970 exploration began into using membranes for water desalination. Implementation of membranes for water treatment has progressed using more advanced membranes made from new materials and employed in various configurations1. Reverse osmosis is nowadays the most extended technology for desalination globally. Besides it has become a viable technology for wastewater reclamation2. These advances mainly include developments in membrane properties and module design, process design, feed pre-treatment, energy recovery devices, and operational strategies focused in energy consumption reductions3-4. Membrane separation processes, an energy saving and high efficiency technology, have been widely used for separation tasks in the chemical, food and biotechnological industries [illegible] cellulose acetate (CA) as a membrane material has found extensive commercial applications because of its relatively low cost, high selectivity, high permeability and easiness of controlling pore size [illegible] various tribes use the different parts of the [illegible] and [illegible] for many of their ailments such as mouth ulcer, fever, boil, epilepsy, burn, snakebite, scorpion sting, rheumatism, fever, headache, etc[9]. The preservation of water resources to prevent their pollution by toxic elements has become one of the most important challenges for the human race due to the exuberant growth of textile, leather, surface treatment, mining, motorcar and chemical industries which generate toxic heavy metals are released into the environment. Heavy metals are dangerous due to bioaccumulation10. The increased concerns in the environment health and the strengthened regulations demand for strict treatment of water and wastewater. Traditional methods for elimination, concentration and/or recovery of heavy metals such as precipitation, ion exchange, electrodeposition, crystallization, evaporation, liquid-liquid extraction etc. have great disadvantages by operating in succession of steps of heterogeneous reactions or distribution of substances between different phases which usually require a lengthy operating period11.

MATERIALS AND METHODS

Chemicals required

[illegible] All [illegible] chemical reagents [illegible] [illegible] chemicals limited, [illegible] powder (purchased from market), Di [illegible] (Loba chemie), Ammonium chloride and EDTA(LR) (Sd fine chem limited), Eriochrome black T indicator(LR) (Emar chemicals limited), potassium dichromate (0.250M), Standard ferrous ammonium sulphate (FAS) (0.25N), conc. sulphuric acid.

Preparation of membrane -1

About 1g of cellulose acetate and 50mg of [illegible] powder was mixed with about 8ml of dimethyl acetamide and it was stirred in mechanical stirrer for about half an hour with a minimum heat of about 30 then it was cooled for 2 minutes and was poured in a flat tile then after 10 minutes the tile was then immersed in water and then the formed membrane was peeled off and then dried for half an hour at room temperature in open air atmosphere. The membrane thickness was found to be 0.53mm.

Preparation of membrane 2

1g of cellulose acetate was mixed with 8ml of dimethyl acetamide followed by 50mg of [illegible] powder and then it was subjected to vigorous stirring for 40mins using a magnetic stirrer at 30oc. The solution was completely dissolved by stirring and then coated with flat tile. Then the coated solution was immersed into water for 2 to 3 mins. Afterwards membrane were peeled out and then dried for half an hour at room temperature in open air atmosphere. The membrane thickness was found to be 0.55mm.

Preparation of membrane 3

1g of cellulose acetate was mixed with 8ml of diethyl succinate (biosolvent) followed by vigorous stirring with the help of magnetic stirrer at 25oc. After 40mins the solution was completely dissolved and then coated with flat tile. Then the coated solution was immersed into water for [illegible] mins. Afterwards membrane were peeled out and then dried for half an hour at room temperature in open air atmosphere. The membrane thickness was found to be 0.76mm.

Characterization of membranes

The prepared membrane was characterized by the following techniques.

Compaction

The thickness [illegible] using WIKA Digital [illegible] WIKA instrument (I) K). The thickness of the membrane used in this study was 0.53-0.2mm. The prepared membrane were cut into effec-

tive membrane area 55.3896 cm² and it was initially pressured with distilled water at 250mmHg for 2min 38sec. The thickness of the membrane 2 used in this study was [illegible] mm. The prepared membrane were cut into effective membrane area 58.465 cm² and it was initially pressured with distilled water at 250mmHg for 2min 45sec. The thickness of the membrane 3 used in this study was 0.7mm. The prepared membrane were cut into effective membrane area 58.465 cm² and it was initially pressured with distilled water at 250mmHg for 2min 45sec. These pre-pressurized membranes were used in subsequent water filtration process.

Pure water reflux:
Membranes after compaction were subjected to pure water flux studies at a trans membrane pressure of 250mm Hg. The pure water flux is determined by using the formula

$$Jw = Q/A\Delta V \quad (1)$$

Where JW water flux (ml/cm²/min)
Q quality of water permeate (l)
A Membrane area (m2)
T the sampling time (hours)

Water uptake:
Percent water content of the membrane was obtained after soaking the membrane in water for 24hrs and the membranes were weighed followed by mapping it with filter paper. The wet membrane was placed in opened air for 1hr and the dry weights of the membranes were determined. From the wet and dry weights percentage water content was determined by

% Water content (wet sample weight-Dry sample weight/wet sample weight ×100 - (2)

FT-IR Analysis:
The membrane structure of the prepared membranes was characterised by using FT-IR (Model IR Affinity-1)

SEM Analysis:
The surface morphology of the prepared membranes was viewed through scanning electron microscope studies. (SCANNING ELECTRON MICROSCOPY XL 20 PHILIPS)

TGA Analysis:
Using thermo gravimetric analysis the weight loss of the membrane was found out (EXSTAR SII TG/DTA6300)

Water filtration by membranes:
The water was filtered using carbon (height-2.5cm & width 0.5cm) covered with the prepared membranes. The sample water was added drop wise to it so the water is filtered through the membranes. The filtered water was then collected and then analysed the physico chemical parameters.

RESULTS AND DISCUSSION
FT-IR spectrum of membrane 1
The peaks corresponding to [illegible] and [illegible] are [illegible] peak for prepared membrane and cellulose acetate. The peak at [illegible] indicates [illegible] stretching. The peak at [illegible] indicates [illegible] (ketone) and for [illegible] indicate C-H bending (strong). The additional peak found for membrane 1 indicated in table 1 and the membrane structure is shown in figure 1.

Table 1
FT-IR Peaks of membrane 1 compared to cellulose

S.No	Frequency (cm⁻¹)	Stretching
1	3834.19	O-H
2	3394.72	N-H(Medium)(primary amines [illegible] bands, secondary have one band [illegible] very weak O-H(alcohol phenol)(free)(strong)
3	3340.71	O-H(streach,free)(strong band) N-H(medium)(primary amine [illegible] bands; secondary have one band [illegible] very weak)
4	3255.84	O-H(H bonded)(strong,band) (alcohol),(dimer)
5	3194.12	O-H (dimer)(COOH) N-H(ammonium ion)(broad peak)
6	3132.40	N-H(ammonium ion)(broad peak) O-H(dimer)(broad)
7	3078.39	O-H(dimer)(broad), C-H(aromatic rings)
8	2924.09	C-H(alkane)(strong) O-H(dimer)(broad)(COOH)
9	2854.65	C-H(alkane)(strong), O-H(carboxylic acid), N-H(ammonium ion)(multiple [illegible] peak)
10	2725.49	N-H(ammonium ion), (multiple broad peak), O-H(carboxylic acid)
11	2584.61	N-H(ammonium ion) (multiple broad peak)
12	2407.16	N-H(ammonium ion) (multiple [illegible] peak)
13	2314.58	O-H
14	2144.84	C≡N(Nitrile)(sharp),N=C=O,C=C[illegible] C≡C(alkynes),N=C=S,N=C=N
15	1913.39	C-H(phenyl ring substitution[illegible])
16	1620.21	N-H (scissoring)(primary amine [illegible]) C=C(aromatic bending)
17	1549.91	C=C(aromatic bending) N-H(amine) NO (asymmetrical)
18	1373.32	C-H(alkalenes),(scissoring and be[illegible]) C-I(alkyl halide)(strong) NO (symmetrical)
19	1234.44	C-F(stretch),C-O(ether) N-H(primary amine)(strong band)
20	1041.56	C-N(amine),C-O(ether),N-H(prim[illegible] amine)(strong band)
21	779.24	C-H (rocking)

Figure 1 FT-IR spectrum of membrane 1

Figure 2 FT-IR spectrum [illegible]

Figure 3 [illegible]

Figure 4 [illegible]

Volume - 5 | Issue - 6 | June 2016 • ISSN No 2277 - 8179 | IF : 3.508 | IC Value : 69.48

Table-2 - FTIR Spectrum of Membrane-2

S.NO	Frequency(cm^{-1})	Stretching
1	3911.64	X-H(Halogen groups)
2	3834.49	N-H(Amines)
3	3765.05	N-H(Amines)
4	3379.29	O-H(H-bonded)(stretch free strong) (alcohol phenol) N-H(Primary amines)
5	3340.71	O-H (stretch free),(strong bond) N-H(primary amines)
6	3263.56	N-H(primary amines) O-H(stretch free),(strong bond)
7	3155.54	N-H(primary amines), C-H(Alkene),(strong)
8	3024.38	C-H(alkene)(strong) O-H(dimer)(broad) COOH(alcohol-phenol)
9	2924.09	C-H(alkane)(strong), O-H(dimer),(COOH),(broad)
10	2877.79	C-H(Alkane),(strong) O-H(Dimer)(broad)(COOH)
11	2738.32	C-H(Alkane)(strong), O-H(broad)(COOH)
12	2538.32	N-H(ammonium ion),(multiple broad peak) O-H(broad)(COOH)
13	2376.30	C=C(Alkenes)
14	2322.29	$C\equiv C$ (Alkynes)
15	2152.56	$C\equiv C$(Alkynes), N=C=O,N=C=S,N=C=N,C=C=O(isocynates,isothiocynates,dimide,ketones)
16	1620.21	C=C(aromatic bending) N-H(primary amine) C-H(Phenyl ring)(substitution overtones)
17	1504.48	C=C(Aromatic bending) NO_2(asymmetrical stretch)
18	1404.18	C=C(Aromatic bending,medium-weak) CH_2(Bending) C-H(Alkanes)
19	1373.32	C-H(Alkanes,Scissoring and bending) C-F(alkyl halide,strong) NO_2(Nitrocompounds,symmetrical) CH_3(rocking)
20	1234.44	C-F(Alkyl halide) C-N(Medium-weak) C-0(Ether,weak)
21	1041.56	C-N(Amine) C-O(Ether) N-H(Primary amine)(strong,broad)
22	171.53	O-H(Stretch,free)

FT-IR spectrum of membrane-3:

The peak corresponding to 1381.03 is found to be common peak for membrane(II) and cellulose acetate. The peak for 1381.03 indicate C-F (Alkyl halide),C-H(Alkane,bending),N-O(Nitro, strong band).

The additional peak found for membrane-3 are indicated in table- 3

Table 3 FTIR Spectrum of Membrane-3

S.NO	Frequency(cm^{-1})	Stretching
1	3990.51	X-H(Halogen groups)
2	3957.93	X-H(Halogen groups)
3	3888.49	O-H(H-bonded)(stretch free strong) (alcohol phenol)
4	3826.77	O-H(H-bonded)(stretch free strong) (alcohol phenol)
5	3703.33	N-H(amide)
6	3479.58	N-H(primary amines) O-H(alcohol)(phenol) C=0(Aldehyde,ester,ketone,carboxylic acid)
7	3379.29	N-H(primary amines), C=0(Aldehyde,ester,ketone,carboxylic acid) C-H(Alkane) O-H(alcohol,phenol)
8	3240.41	C=0(Aldehyde,ester,ketone,carboxylic acid) O-H(Alcohol,phenol)
9	3163.26	C=0(Aldehyde,ester,ketone,carboxylic acid) O-H(Alcohol,phenol)
10	2924.09	C-H(Alkane),(strong) O-H(Carboxylic acid)
11	2854.65	C-H(Alkane)(strong), O-H(broad)(COOH)
12	2785.21	O-H(Carboxylic acid)
13	2646.34	O-H(Carboxylic acid)
14	2245.14	.C=N(Nitrile)
15	2222.00	C=C(Nitrile)
16	2167.99	C
17	1959.68	C-H(Phenyl substitution overtones)
18	1897.95	C-H(Phenyl substitution overtones)
19	1851.66	C-H(Phenyl substitution overtones)
20	1666.50	C=C(Alkenes) C=O(Amide)
21	1527.62	N-H(Amine)(Scissoring,bending)
22	1442.75	C-H(Alkanes) CH_2(Bending)
23	1226.73	C-O(Aldehyde,ketone.carboxylic acid,ester) C-N(Amine)
24	1157.29	C-O(Aldehyde,ketone,carboxylic acid,ester)
25	1041.56	C-N(Amine) C-O(Aldehyde,ketone.carboxylic acid,ester)
26	956.69	C-H(Alkenes)
27	840.96	C-H(Alkenes)(Arenes,C-H bending and ring puckering)
28	779.24	CH_2(Bending) C-H(Alkenes)(Arenes C-H Bending and ring puckering)
29	748.38	C-H(Alkenes)(Arenes C-H Bending and ring puckering)

Water uptake

Membrane-1

%Water content=wet sample weight-Dry sample weight/wet sample weight X100

=[0.8624-0.3323/1.33] x100/(3.14x4x4.2x4.2x4.2)

=0.04279.

Membrane-2

W_1=0.8634; W_2=0.4053; dwater=1.33; v =πr3

= [0.5301/1.33] ×100/3.14×4.2×4.2×4.2

= 0.0427

Membrane-3

W_1=2.6944; W_2=2.0424; dwater=1.33; v =πr3

=[0.6520/1.33] ×100/3.14×3.5×3.5×3.5

= 0.0909

Water compaction

Membrane-1

J_w=Q/AΔV

19/ (3.14x4.2x4.2x (3/60))

=6.8606

Membrane-2

Q=19 ; A=πr2 ; π=3.14 ; r=3.5;

J_w= 19/3.14×3.5×3.5×0.033

= 14.8344

Membrane-3

Q=19 ; A=πr2 ; π=3.14 ; r=3.5;

J_w= 19/3.14×3.5×3.5×0.0500

= 9.8793

Membrane thickness:

The thickness of the cast membrane was measured using

WIRA Digital thickness tester (WIRA Instrument, UK). The thickness of the membrane-1,2,3 was found to be 0.53mm,0.55mm and 0.76mm.

TGA Analysis:

The weight loss of the membrane was analysed by thermo gravimetric analysis. It was measured from a temperature of 30 up to 210 and it is indicated in fig-5,6&7.

Figure- 5 Thermo gravimetric analysis of membrane-1

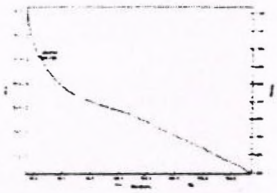
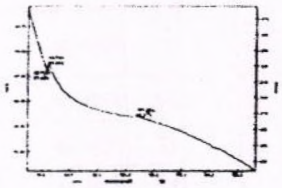


Figure - 6 TGA images of Membrane-2



The membrane was found to be stable till a temperature of 40and then it is found to be steadily decreasing. At 40the membrane was 97.12%

The membrane was found to be stable till a temperature of 43.7and then it is found to be steadily decreasing. At 43.7the membrane was 97.22%.

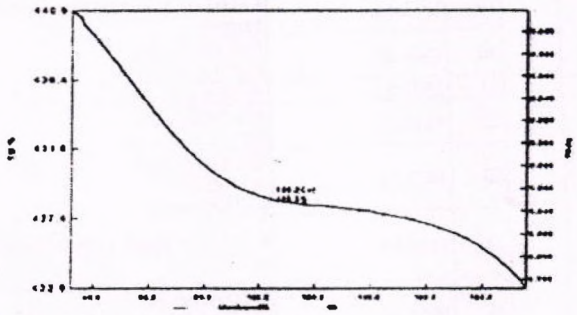


Figure 7 TGA images of Membrane-3

The membrane was found to be stable till a temperature of 40and then it is found to be steadily decreasing. At 40the membrane was 97.12%.

X-Ray diffraction:

X-ray diffraction pattern of the membrane-1 was analysed, and no peaks were found. The membrane -2 showed a sharp peak at 65.6872nm(fig-8) and no peak was observed for membrane-3.

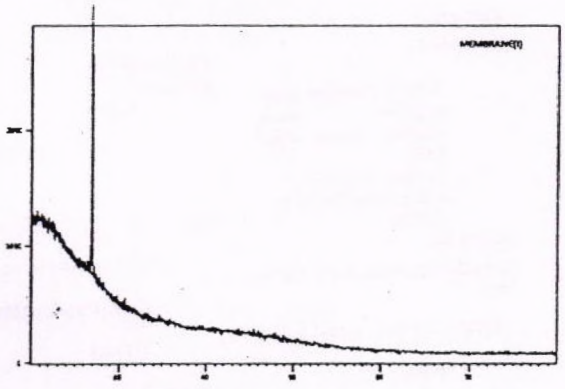


Figure 8 X-Ray diffraction images of membrane-2

5.7 SEM:

The SEM analysis of the membrane -1,2,3 are shown in fig-9,10 &11.

Figure 9 Scanning Electron Microscopic Image of the membrane-1

Figure 10 SEM image of the

Figure 11 SEM Images of the membrane-3

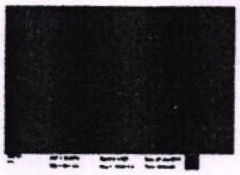


Table 4

Physico-chemical parameters of Sample water and filtered water using membrane-1,2 & 3

S.NO	PARAMETER	SAMPLE WATER	MEMBRANE-1	MEMBRANE-2	MEMBRANE-3
1	PH	7.58	8.20	7.96	7.58
2	Electrical conductivity(ds/m)	1.21	1.19	1.19	1.19
3	Calcium(mg/L)	52.80	41.6	43.2	38.4
4	Magnesium(mg/L)	59.41	48.07	50.78	36.2
5	Sodium(mg/L)	132.25	53.59	75.21	53.59
6	Potassium(mg/L)	14.04	9.36	14.04	9.36
7	Carbonate(mg/L)	1.60	0.08	1.60	1.60
8	Bicarbonate(mg/L)	4.40	3.20	3.20	3.20
9	Chloride(mg/L)	77.8	9.52	9.52	8.16
10	BOD	3.8	4.2	4.6	3.8
11	COD	12.1	10	15	12.1
12	Dissolved oxygen	7.9	5.6	6	5.2
13	Bacteria	11×10^5	9×10^5	9×10^5	4.5×10^5
14	Fungi	9×10^3	5.5×10^3	5.5×10^3	1.5×10^3

Table 5

Hardness measurement for sample water and filtered water using membrane-1,2&3

S.NO	PARAMETER	SAMPLE WATER	MEMBRANE-1	MEMBRANE-2	MEMBRANE-3
1	Calcium carbonate	1.60	0.80	1.60	1.60
2	Magnesium chloride	2.73	0.59	0.24	-
3	Magnesium bicarbonate	3.12	2.16	2.92	2.16
4	calcium bicarbonate	1.28	1.04	0.28	-
5	Sodium chloride	1.81	0.09	0.09	1.90

pH:

pH is an important indicator which indicates acidic and alkaline nature of water. pH should be in the level of 6.6 to 8.4(World Health Organisation).The pH of the sample water was found to be 7.58. The sample water was filtered using membrane-1, membrane-2 and membrane-3 and tested for pH. (table-4)

The obtained values were compared with the standard value of World Health Organisation and it was found that the values were in good agreement.

Electrical conductivity (EC):

Electrical conductivity (EC) is used to determine the cell

Volume : 5 | Issue : 6 | June 2016 • ISSN No 2277 - 8179 | IF : 3.508 | IC Value : 69.48

...ue. The electrical conductivity of the sample ...e value 1.21(ds/m). The sample water after fil...gh membrane-1,2,3 the electrical conductivity ...ed and the value obtained was 1.19(ds/m) for ...1.19(ds/m) for membrane-2 and 1.19(ds/m) for ...3(table-4).

...ardness:
... water, Calcium hardness should be in the level ...(World Health Organisation). The calcium hard...mple water was found to be 52.80mg/L. The sam...was filtered using Membrane-1,2,3 and tested for ...ardness.(table-4)

...ed values were compared with the standard val...ld Health Organisation and it was found that the ...re in good agreement.

...m Hardness:
...ing-water, Magnesium hardness should be in ... of 150mg/L (world health organization 2003). ...nesium hardness of sample water was found to ...ng/L. The sample water was filtered using Mem...3 and tested for Magnesium hardness. It was ob...hat the Magnesium hardness of filtered water was ...o be 48.07mg/L for membrane-1 50.78 mg/L for ...ne-2 and 7.58mg/L for membrane-3.(table-4)

...ined values were compared with the standard val...orld Health Organisation and it was found that the ...were in good agreement.

...um hardness:
...king-water, potassium hardness should be in the level ...g/L (world health organization 2003).The Potassium ...ss of sample water was found to be 14.04mg/L. The ... water was filtered using Membrane and tested for ...um hardness. The sample water was filtered using ...rane-1,2,3 and tested for potassium hardness. (table-4)

...btained values were compared with the standard val...World Health Organisation and it was found that the ... were in good agreement.

...m:
...nking-water, sodium hardness should be in the level ...0mg/L (world health organization 2003).The sodium ...ess of sample water was found to be 132.25mg/L. ...ample water was filtered using membrane and tested ...odium hardness. The sample water was filtered using ...brane-1,2,3 and tested for sodium hardness. (table-4)

...obtained values were compared with the standard val...f World Health Organisation and it was found that the ...es were in good agreement.

...onate:
... carbonate hardness should be in the level of 500 mg/L ...rld Health Organisation 2008). The value of carbonate ...the sample water is found to be 1.06 mg/L. The sample ...er was filtered using Membrane-1,2,3 and tested for car...ate hardness. (table-4)

... obtained values were compared with the standard val...of World Health Organisation and it was found that the ...ues were in good agreement.

...rbonate:
... bicarbonate hardness should be in the level of 100 ...L (World Health Organisation 2008). The value ob...tained for the bicarbonate in the sample water is 4.40mg/L. The sample water was filtered using Membrane-1,2,3 and tested for bicarbonate hardness. It was observed that the bicarbonate hardness of filtered water was found to be 3.20mg/L for membrane-1 3.20 mg/L for membrane-2 and 3.20mg/L for membrane-3. (table-4)

The obtained values were compared with the standard value of World Health Organisation and it was found that the values were in good agreement.

Dissolved oxygen:
In drinking-water, Dissolved oxygen should be in the level of 30mg/L (World health organization 2003).The Dissolved oxygen of sample water was found to be 7.9mg/L. The sample water was filtered using Membrane and tested for Dissolved oxygen. The sample water was filtered using Membrane-1,2,3 and tested for dissolved oxygen. (table-4)

The obtained values were compared with the standard value of world health organisation and it was found that the values were in good agreement.

Biological oxygen demand:
Biological oxygen demand is the measure of degradation of organic matter present in water. The Biological oxygen demand refers to the oxygen used by the micro-organisms in the aerobic oxidation of organic matter. The Biological oxygen demand of sample water was found to be 3.8 mg/L. The sample water was filtered using Membrane-1,2,3 and tested for biological oxygen demand. It was observed that the biological oxygen demand of filtered water was found to be 4.2mg/L for membrane-1 4.6 mg/L for membrane-2 and 3.8mg/L for membrane-3. (table-4)

The obtained values were compared with the standard value of world health organisation and it was found that the values were in good agreement.

Chemical oxygen demand:
Chemical Oxygen Demand is another measure of organic material contamination in water specified in mg/L. Chemical Oxygen Demand is the amount of dissolved oxygen required to cause chemical oxidation of the organic material in water. The Chemical Oxygen Demand of sample water was found to be 12.1 mg/L. The sample water was filtered using Membrane-1,2,3 and tested for chemical oxygen demand. (table-4)

The obtained values were compared with the standard value of world health organisation and it was found that the values were in good agreement.

Chloride:
Chloride is an important parameter in accessing the water quality. The values of chlorides in normal drinking water are range 250 mg/L (world Health organization 2003). The sample water has the chloride value of 77.8mg/L. The sample water was filtered using Membrane-1,2,3 and tested for Magnesium hardness. It was observed that the Magnesium hardness of filtered water was found to be 9.52mg/L for membrane-1 9.52 mg/L for membrane-2 and 8.16mg/L for membrane-3. (table-4)

The obtained values were compared with the standard value of world health organisation and it was found that the values were in good agreement.

Bacteria:
The bacterial growth was observed in the sample water and

was having the value 11x105mg/L. The sample water was filtered with the membrane-1,2,3 and the bacterial growth was observed for the filtered water and the value obtained was 9 x105mg/L. (table-4)

Fungi:

The fungal growth was observed in the sample water and was having the value 9x103mg/L. The sample water was filtered with the membrane and the fungal growth was observed for the filtered water and the value obtained was 5.5 x 103mg/L. (table-4)

SUMMERY AND CONCLUSION

The aim of this study is to prepare the Green membrane using low toxicity solvent.

The physico-chemical parameter values of the filtered water using the prepared membrane were compared with the values described by the world health organisation and the values mostly is in good agreement.

The membrane preparation is an eco-friendly method and it is easy to handle and it is a low cost method also.

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