

CELLULOSE ACETATE –STARCH BLEND GREEN MEMBRANE PREPARATION AND ITS APPLICATION

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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 and TGA analysis. The green membrane has been applied to salty water filtration. The filtered water is used to study the physio-chemical parameters. Most of the parameters are correlated with the values prescribed by World Health Organisation.

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

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 configurations (Amjad, 1993). Reverse osmosis is nowadays the most extended technology for desalination globally. Besides, it has become a viable technology for wastewater reclamation (Zhao *et al.*, 2010). 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 reductions (Yang *et al.*, 2009 and Kumar *et al.*, 2006). Membrane separation processes, an energy saving and high- efficiency technology, have been widely used for separation tasks in the chemicals, food and biotechnological industries (Yang *et al.*, 1998 and Zhang *et al.*, 2000). Cellulose acetate (CA) as a membrane material has found extensive commercial application, because of its relatively low cost, high selectivity, high permeability and easiness of controlling pore size. Amla fruit is widely used in the Indian system of medicine as diuretic, laxative, liver tonic, refrigerant, stomachic,

restorative, anti-pyretic, hair tonic, ulcer preventive, common cold, fever, etc (Hao and Wang, 1997). 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 bioaccumulation (Grimshaw, 1993). Pure starch is insoluble in cold water or alcohol. It consists of two types of molecules, the linear and helical amylose and the branched amylopectin, (Brown, 2005). The increased concerns in the environment, health and the strengthened regulations demand more 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 a succession of steps of heterogeneous reactions, or distribution of substances between different phases which usually require a lengthy operating period (Caflizares, 2002).

MATERIALS AND METHODS

Chemicals required

Cellulose acetate, Starch (AR Spectrum chemical

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reagents), Dimethyl acetamide (Finar chemicals limited), Amla powder (purchased from market), Ammonium chloride and EDTA (LR Sd fine-chem limited), Eriochrome black-T indicator (LR Finar chemicals limited), potassium dichromate (0.250M), Standard ferrous ammonium sulphate (FAS) (0.25N), concentrated sulphuric acid.

Preparation of membrane

About 0.5g of cellulose acetate, 0.5g of starch and 50mg of Amla powder was mixed with about 6ml of dimethyl acetamide and it was stirred in mechanical stirrer for about half an hour with a minimum heat of about 30°C 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 0.43mm.

Characterization of membranes

The prepared membrane was characterized by the following techniques.

Compaction

The thickness of the cast membrane was measured using WIRA Digital thickness tester (WIRA Instrument, UK). The thickness of the membrane used in this study was 0.47 ± 0.2 mm. The prepared

membrane were cut into effective membrane area 45.3896 cm^2 and it was initially pressured with distilled water at 250mmHg for 2 min 38 sec. These pre-pressurized membrane 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

$$J_w = Q / A \Delta T \quad \dots (1)$$

Where, J_w - water flux ($\text{mL}/\text{cm}^2\text{min}$)

Q - quality of water permeate (l)

A - Membrane area (m^2)

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

$$\% \text{Water content} = \frac{\text{wet sample weight} - \text{Dry sample weight}}{\text{wet sample weight}} \times 100 \quad \dots (2)$$

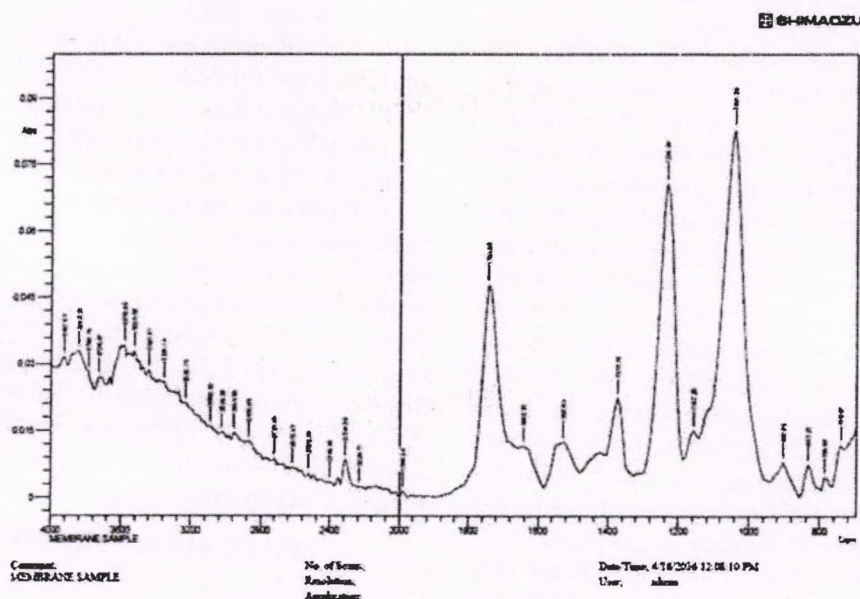


Fig. 1. FT-IR spectrum of the membrane

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

Sl.No	Frequency(cm ⁻¹)	Stretching
1	3927.07	O-H(Halogen groups)
2	3842.20	N-H(Amines)
3	3788.19	N-H(Amines)
4	3726.47	C-H
5	3579.88	O-H (free O-H),(Broad, sharp peaks)
6	3525.88	O-H(Broad absorption)
7	3441.01	N-H(Secondary amines, amide),(free1-band, medium)
8	3356.14	N-H (Secondary amines), (medium)
9	3232.70	C=OO-H(Alcohol, Phenol)
10	3093.82	Alkenes
11	3024.38	Alkyne,aromatic,C-H stretching,heteroaromatic
12	2954.95	C-H(alkyne)(strong)
13	2870.08	C-H stretching(alkanes)(strong)O-H(carboxylic acid)
14	2723.49	N-H(ammonium ion),(multiple broad peak),O-H(carboxylic acid)
15	2615.47	O-H (carboxylic acid),(broad)
16	2522.89	N-H(ammonium ion),(multiple broad peak),H(broad) peak)
17	2399.45	N-H(ammonium ion),(multiple broad peak)
18	2314.58	O-H
19	2229.71	N-H*stretching (sharp),(broad bands)
20	1990.54	C=C=CH ₂ (allenes),asymmetric C=C=C
21	1743.65	C=O (anhydride, strong)
22	1643.35	C=O (amides, strong)
23	1527.62	N-H(amine) (scissoring, bending)
24	1373.32	C-H (alkenes), (scissoring and bending)C-F(alkylhalide), (strong), NO ₂ (symmetrical)
25	1234.44	C-F (stretch),C-O (ether)
26	1157.29	C-O(aldehyde, ketone, carboxylicacid, ester)
27	1041.56	C-N (amine)C-O(aldehyde, ketone, carboxylic acid, ester)
28	902.69	C-C stretching vibration (weak and broad)
29	833.25	C-H(alkenes), (arenes), C-H (bending and ring puckering)
30	786.96	N-H wagging, (out of plane bending)
31	740.67	C-H(alkenes) (arenes),C-H (bending and ring puckering)

FT-IR Analysis

The membrane structure of the prepared membranes was characterised by using FT-IR (Model IR Affinity-1) in Fig. 1 and values are Tabulated in Table 1.

SEM Analysis

The surface morphology of the prepared membranes was viewed through scanning electron microscope studies. (SCANNING ELECTRON MICROSCOPY XL 20 PHILIPS). Fig. 3

TGA Analysis

Using thermo gravimetric analysis the weight loss of the membranes was found out. (EXSTAR SII TG/DTA6300) Fig. 4.

Water filtration by membranes

The water was filtered using carbon rod(height-2.5cm & width-0.2cm) covered with the prepared membrane. The sample water was added drop wise to it so the water is filtered through the membrane. The filtered water was then collected and then analysed the physio-chemical parameters.

RESULTS AND DISCUSSION**FT-IR spectrum of membrane**

The peak corresponding to 1527.62, 1157.29, 902.69 and 833.25 is found to be the common peak for prepared membrane and cellulose acetate. The peak at 1527.62 indicates N-H stretching, the peak at 1157.29 indicates C-O (aldehyde), (ester), (carboxylic acid) and (ketone), for 902.69 it indicates C-C stretching vibrations (weak and appear in broad region). The

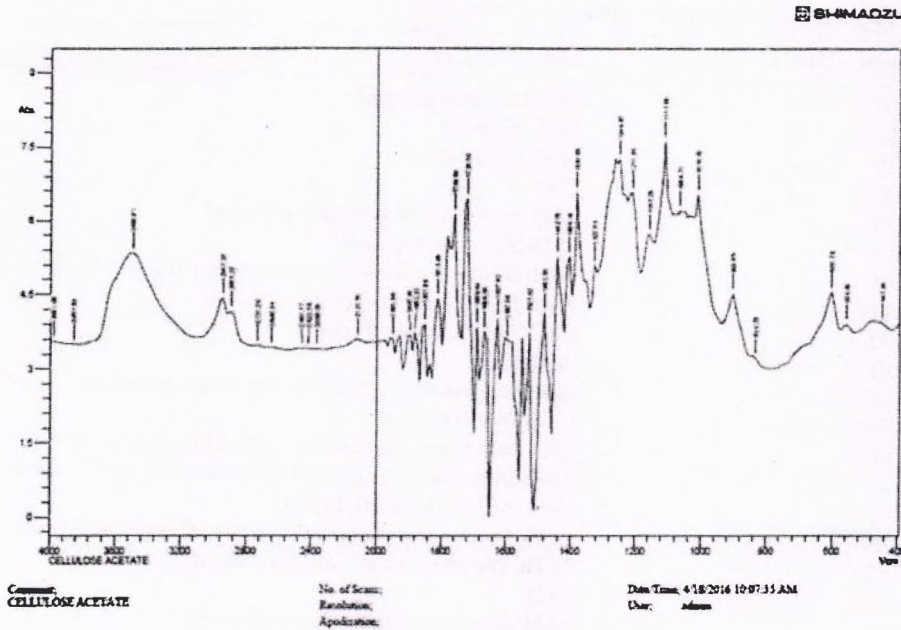


Fig 2. FT-IR spectrum of cellulose acetate

Table 2. Hardness measured for sample water and filtered water using membrane

S.no	Parameter	Sample Water	Memb Rane
1	Calcium carbonate	20	0.65
2	Magnesium chloride	2.55	0.50
3	Magnesium bicarbonate	3.00	2.23
4	Calcium bicarbonate	1.35	1.00
5	Sodium chloride	1.89	0.04

additional peak values

Indicated that Amla powder is incorporated in the formation of membrane

Water uptake

% Water content = $\frac{\text{Wet sample weight} - \text{Dry sample weight}}{\text{Wet sample weight}} \times 100$
 $= \frac{[2.1518 - 1.6577 / 1.33] \times 100}{(3.14 \times 4 \times 4.2 \times 4.2 \times 4.2)}$
 $= 0.03992.$

Water compaction

Table 3. Physio-chemical parameters of Sample water and filtered water using membrane

S.NO	PARAMETER	SAMPLE WATER	MEMBRANE
1	pH	7.75	0.34
2	Electrical Conductivity(ds/m)	1.02	0.44
3	Calcium (mg/L)	40	25
4	Magnesium(mg/L)	28.8	17.8
5	Sodium (mg/L)	68	45
6	Potassium (mg/L)	12	3.1
7	Carbonate (mg/L)	-	-
8	Bicarbonate (mg/L)	84	70
9	Chloride (mg/L)	97	31
10	BOD	3.5	4
11	COD	11	10
12	Dissolved oxygen	4.00	5.50
13	Bacteria	18×10^5	10×10^5
14	Fungi	5×10^3	5×10^3

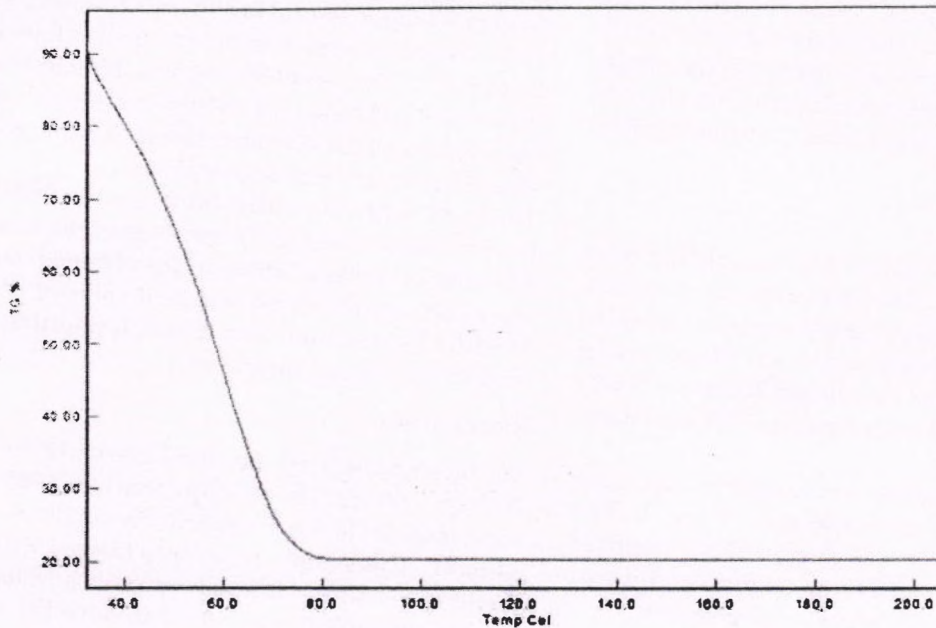


Fig. 3. Thermo gravimetric analysis of the membrane

$$\begin{aligned}
 J_w &= Q / A \Delta T \\
 &= 18 / (3.14 \times 4.2 \times 4.2 \times (3/60)) \\
 &= 5.0560.
 \end{aligned}$$

Membrane thickness

The thickness of the cast membrane was measured using WIRA Digital thickness tester (WIRA Instrument, UK). The thickness of the prepared membrane was 0.47 mm.

TGA

The weight loss of the membrane was analysed by thermo gravimetric analysis. It was measured from a temperature of 30°C upto 210°C and it is indicated in The prepared membrane was found to be stable till a temperature of 60°C and then it is found to be steadily decreasing.

SEM

The SEM analysis of the membrane is shown in the fig. 4.

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 Organization). The

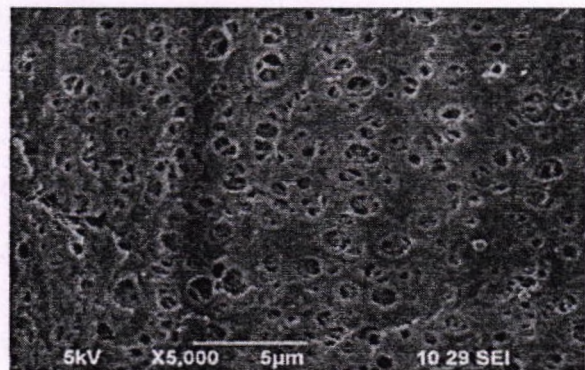


Fig. 4. Scanning Electron Microscope image of the membrane at 5µm

pH of the sample water was found to be 7.75. The sample water was filtered using membrane and tested for P4". It was observed that the pH of sample water was found to be 0.34.

The obtained value for the filtered water was found to be 0.34. The obtained values were compared with the standard value of World Health Organization and it was found that the values were in good agreement.

Electrical Conductivity (EC)

Electrical conductivity (EC) is used to determine the cell constant value. The electrical conductivity of the sample water has the value 1.02(ds/m). The sample water after filtration through membrane, the electrical conductivity was measured and the value obtained was 0.44 (ds/m).

Calcium Hardness

In drinking water, Calcium hardness should be in the level of 75mg/L (World Health Organization). The calcium hardness of sample water was found to be 40mg/L. The sample water was filtered using Membrane and tested for calcium Hardness. It was observed that the Calcium hardness of filtered water found to be 25 mg/L.

The Calcium hardness of the sample water after filtration using membrane was 25 mg/L. The obtained values were compared with the standard value of World Health Organization and it was found that the values were in good agreement.

Magnesium Hardness

In drinking-water, Magnesium hardness should be in the level of 150mg/L (World health Organization 2003). The Magnesium hardness of sample water was found to be 28.8mg/L. The sample water was filtered using Membrane and tested for Magnesium hardness. It was observed that the Magnesium hardness of filtered water was found to be 17.8mg/L.

The Magnesium hardness of the sample water after filtration using the membrane was found to be 17.8mg/L. The obtained values were compared with the standard value of World Health Organization and it was found that the values were in good agreement.

Potassium hardness

In drinking water, potassium hardness should be in the level of 82mg/L(world health organization 2003). The Potassium hardness of sample water was found to be 12mg/L. These sample water was filtered using Membrane and tested for potassium hardness. It was observed that the potassium hardness of the filtered water was found to be 3.1 mg/L

The potassium hardness obtained after the filtration of the sample water using membrane is 3.1mg/L. The obtained values were compared with the standard value of World Health Organization and it was found that the values were in good agreement.

Sodium

In drinking water, sodium hardness should be in the level of 200mg/L (world health organization 2003). The sodium hardness of sample water was found to be 68mg/L. The sample water was filtered using membrane and tested for sodium hardness. It was observed that the sodium hardness of sample water after filtration was found to be 45mg/L.

The Sodium hardness after filtration was found to have the value 45mg/L. There is a good decrease in the value of sodium hardness. The obtained values were compared with the standard value of World Health Organization and it was found that the values were in good agreement.

Bicarbonate

The bicarbonate hardness should be in the level of 100mg/L (World Health Organisation 2008). The value obtained for bicarbonate in the sample water is 84mg/L. This sample water was filtered with the membrane and the filtered water was tested for bicarbonate and it was found to have the value 70mg/L.

The obtained values were compared with the standard value of World Health Organization 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 4.00mg/L. The sample water was filtered using Membrane tested for Dissolved oxygen. It was observed that the Dissolved oxygen of filtered water was found to be 5.50mg/L. The value of dissolved oxygen has been increased.

The Dissolved oxygen of the sample water after filtration using membrane was found to have the value 5.50mg/L. The obtained values were compared with the standard value of world health organization 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 3.5mg/L. The sample water was filtered using membrane and tested for

Biological oxygen demand. The obtained value was 4.00mg/L.

The Biological oxygen demand of the sample water after filtration using membrane was found to have the value of 4.00mg/L. The obtained values were compared with the standard value of world health organization 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 11mg/L. The sample water was filtered using membrane and tested for Chemical Oxidation Demand of water, the value obtained was 10mg/L.

The Chemical oxygen demand of the sample water after filtration was found to have the value 10mg/L. The obtained values were compared with the standard value of world health organization 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 250mg/L (world health organization 2003). The sample water has the chloride value of 97mg/L. The sample water was filtered using membrane and tested for Chloride, the value obtained was 31mg/L.

The Chloride value of the sample water after filtration using membrane was 31mg/L. The obtained values were compared with the standard value of world health organization 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 18×10^5 mg/L. The sample water was filtered with the membrane and the bacteria growth was observed for the filtered water and the value obtained was 10×10^5 mg/L.

Fungi

The fungal growth was observed in the sample water and was having the value 5×10^3 mg/L. The sample water was filtered with the membrane and the fungal growth was observed for the filtered water and the value obtained was 6×10^3 m.

SUMMARY AND CONCLUSION

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

The physio-chemical parameter values of the filtered water using the prepared membrane were compared with the values described by the World Health Organization and the values are mostly 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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