Cellulose acetate based Eco friendly membrane preparation and its application studies

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Abstract

The aim of the study is to prepare the cellulose acetate & amla based on 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.

Key words: Cellulose acetate, Amla, Green membrane, Filtration, Physico-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 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 chemicals, food and biotechnological industries⁵,⁶. 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 size⁷,⁸. Various tribes use the different parts of the Amla plant for many of their ailments such as mouth ulcer, fever, boil, epilepsy, burn, snakebite, scorpion sting, rheumatism, fever, headache, etc⁹. 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 more strict treatment of water and wastewater. Traditional
methods for elimination, concentration and 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.\(^1\)

**Material and Methods**

**Chemicals required:**
- Cellulose acetate (AR Spectrum chemical reagents)
- Dimethylacetamide (Finar chemicals limited)
- Amla powder (purchased from market)
- Diethyl succinate (Lobachemie)
- 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)
- Conc sulphuric acid

**Preparation of membrane:**
- About 1g of cellulose acetate 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 0.43±0.2mm. The prepared membrane were cut into effective membrane area 45.3896 cm\(^2\) and it was initially pressured with distilled water at 250mmHg for 2min 38sec. 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
      \[ J_w = \frac{Q}{A \Delta T} \]  
      \[ J_w - \text{water flux (ml/cm}^2\text{ min)} \]
      \[ Q - \text{quality of water permeate(l)} \]
  - **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
    \[ \% \text{Water content} = \frac{\text{wet sample weight} - \text{Dry sample weight}}{\text{wet sample weight}} \times 100 \]
  - **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.
  - **TGA Analysis:**
    - Using thermo gravimetric analysis the weight loss of the membranes was found out. (EXSTAR SII TG/DTA6300).
  - **Water filtration by membrane:**
    - The water was filtered using carbon rod (ht-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 membranes. The filtered water was then collected and then analysed the physico-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 common peak for prepared membrane and cellulose acetate. The peak at 1527.62 indicates N-Hstreaking. amines, (scissoring & bending). Peak at 1157.29 indicates C=O,Aldehyde, carboxylicacid, ketone, Ester and for 902.69 it indicate C-H (bending) (strong). for Peak at 833.25 indicates C-H alkanes,arenes,C-H bending&ring puckering.The additional peak found for membrane is indicated in table -1 and the membrane structure is shown in figure-1.
Table 1
FT-IR Peaks of membrane compared to cellulose acetate

| Sl.No | Frequency(cm⁻¹) | Stretching |
|-------|----------------|------------|
| 1     | 3927.07        | O-H(halogen group) |
| 2     | 3842.20        | N-H (amines) |
| 3     | 3340.71        | O-H(streak, free) (strong band) N-H(medium) (primary amine have two bands; secondary have one band, often very weak) |
| 4     | 3255.84        | O-H (H-bonded) (strong,band) (alcohol), (dimer) |
| 5     | 3132.40        | N-H(ammonium ion)(broad peak)O-H(dimer)(broad) |
| 6     | 3078.39        | O-H(dimer)(broad),C-H(aromatic rings) |
| 7     | 2924.09        | C-H(alkane)(strong)O-H(dimer)(broad)(COOH) |
| 8     | 2854.65        | C-H(alkane)(strong),O-H(carboxylic acid),N-H(ammonium ion)(multiple broad peak) |
| 9     | 2723.49        | N-H(ammonium ion),(multiple broad peak),O-H(carboxylic acid) |
| 10    | 2584.61        | N-H(ammonium ion)(multiple broad peak) |
| 11    | 2407.16        | N-H(ammonium ion),(multiple broad peak) |
| 12    | 2314.58        | O-H |
| 13    | 2144.84        | C≡N(Nitrile)(sharp),N≡C=O,C≡C=O C≡C(alkynes),N≡C=S,N≡C=N. |
| 14    | 1913.39        | C-H(phenyl ring substitution)(overtones) |
| 15    | 1620.21        | N-H2(scissoring)(primary amine)C=C(aromatic bending) |
| 16    | 1519.91        | C=C(aromatic bending)N-H(amine)NO2(asymmetrical) |
| 17    | 1373.32        | C-H(alkenes), (scissoring and bending)C-F(alkyl halide)(strong) NO2 (symmetrical) |
| 18    | 1234.44        | C-F(stretch),C-O(ether)N-H(primary amine)(strong band) |
| 19    | 1041.56        | C-N(amine),C-O(ether),N-H(primary amine)(strong band) |
| 20    | 779.24         | CH2(rocking) |

Figure 1: FT-IR spectrum of membrane
Water uptake

Membrane:

\% Water content = wet sample weight - Dry sample weight / wet sample weight \times 100

\[=\frac{2.1518-1.6577}{1.33}\times100\]

Water compaction

Membrane

\[J_w=\frac{Q}{A\Delta T} \frac{I_b}{(3.14\times4.2\times4.2\times(3/60))}
\]

5.0560

Membrane thickness:

The thickness of the cast membrane was measured using WIRA Digital thickness tester (WIRA Instrument, UK). The thickness of the membrane was found to be 0.43mm.

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-2

![Thermo gravimetric analysis of membrane](image)

The membrane was found to be stable till a temperature of 60°C and then it is found to be steadily decreasing. At 60°C the membrane was 97.12%

SEM analysis of the membrane are shown in fig-3

![Scanning Electron Microscopic image of the membrane](image)

Table 2. Physico-chemical parameters of Sample water and filtered water using membrane.

| S. No. | Parameter          | Sample water | Membrane |
|-------|--------------------|--------------|----------|
| 1     | pH                 | 7.75         | 0.44     |
| 2     | Electrical conductivity (ds/m) | 1.02         | 0.42     |
| 3     | Calcium (mg/L)     | 40           | 20       |
| 4     | Magnesium (mg/L)   | 28.8         | 18.8     |
| 5     | Sodium (mg/L)      | 68           | 47       |
| 6     | Potassium (mg/L)   | 12           | 3.2      |
| 7     | Carbonate (mg/L)   | 2.8          | 1.4      |
| 8     | Bicarbonate (mg/L) | 84           | 79       |
| 9     | Chloride (mg/L)    | 97           | 41       |
| 10    | BOD                | 3.5          | 4        |
| 11    | COD                | 11           | 10       |
| 12    | Dissolved oxygen   | 4.00         | 5.50     |
| 13    | Bacteria           | 18x10^3      | 12x10^3  |
| 14    | Fungi              | 5x10^3       | 6x10^3   |

Table 3. Hardness measurement for sample water and filtered water using membrane

| S. No. | Parameter          | Sample water | Membrane |
|-------|--------------------|--------------|----------|
| 1     | Calcium carbonate | 20           | 0.75     |
| 2     | Magnesium chloride| 2.73         | 0.59     |
| 3     | Magnesium bicarbonate | 3.00   | 2.33     |
| 4     | calcium bicarbonate| 1.35         | 1.10     |
| 5     | Sodium chloride    | 1.81         | 0.07     |

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 7.75. The sample water was filtered using membrane and tested for pH, (table-2)

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 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 0.42(ds/m).
**Calcium Hardness:**

In drinking water, Calcium hardness should be in the level of 75mg/L (World Health Organisation). The calcium hardness of sample water was found to be 20mg/L. The obtained values were compared with the standard value of World Health Organisation 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 18.8mg/L for membrane.

The obtained values were compared with the standard value of World Health Organisation 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. The sample water was filtered using Membrane and tested for potassium hardness. The sample water was filtered using Membrane & found to be 3.2 mg/L.

The obtained values were compared with the standard value of World Health Organisation 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. The sample water was filtered using Membrane & the value found to be 47 mg/L.

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

**Carbonate:**

The carbonate hardness should be in the level of 500 mg/L (World Health Organisation 2008).

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

Similarly all other physico-chemical parameters in Tables are which is in good agreement with the value prescribed by the WHO.

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**Summary 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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