

RESEARCH PAPER

Cation Exchange Nanocomposite Membrane Containing Mg(OH)₂ Nanoparticles: Characterization and Transport Properties

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ABSTRACT

In this study, ion exchange nanocomposite membranes was prepared by addition of Mg(OH)₂ nanoparticles to a blend containing sulfonated polyphenylene oxide and sulfonated polyvinylchloride via a simple casting method. Magnesium hydroxide nanoparticles were synthesized via a facile sono-chemical reaction and were selected as filler additive in fabrication of ion exchange nanocomposite membranes. Nanoparticles and nanocomposites were then characterized using scanning electron microscopy, Fourier transform infrared spectroscopy and X-ray diffraction. The effect of nanoparticles loading on physicochemical and electrochemical properties of prepared cation exchange nanocomposite membranes was studied. The membranes performance was evaluated by membrane potential, transport number, permselectivity, ionic permeability, flux of ions and membrane oxidative stability. Various characterizations revealed that the addition of different amounts of inorganic fillers could affect the membrane performance. The inorganic nanoparticles not only created extra pores and water channels that led to ion conductivity enhancement, but also improved transport number, permselectivity and flux of ions.

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INTRODUCTION

Ion exchange membrane (IEM) has mostly been used in solutions containing multiple components, including electrodialytic demineralization of saline water, treatment of industrial effluents containing metal ions and desalination of cheese whey solution [1–3]. The composite membranes represent the essential properties of organic polymeric matrix and inorganic fillers and put forward specific advantages for the fabrication of new membranes with suitable thermal and chemical resistance, excellent separation performances and compatibility to harsh environments [4–11]. Thus, organic–inorganic composite materials have

attracted more concern. Poly (2,6-dimethyl-1,4-phenylene oxide) (PPO) is a poly-aryl compound which has suitable membrane-forming properties, appropriate thermal and chemical stability [12]. Among the PPO derivatives, aryl substituted sulfonated PPO (SPPO) is an appropriate structure which has been used as a membrane for reverse osmosis, gas separation, ultra-filtration and cation exchange membranes [13–21]. The dimensional instability of SPPO is a disadvantage which prevents its practical applications in fuel cells and electro-membrane processes [22]. One of the attractive procedure for improving the membrane properties (e.g., water swelling and dimensional stability) is

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blending of a mechanical and dimensional stable material into hydrocarbon based polymers.

Polyvinyl chloride is an outstanding material because of its high mechanical strength, reasonable cost and excellent chemical properties (resistance against acid, alkali and organic solvents) [17].

Preparing the new type of cation exchange nanocomposite membranes with appropriate physicochemical properties for application in electro-dialysis process was main target in this work. For the purpose, SPVC/SPPO blend cation exchange membranes were prepared by solution casting techniques using tetrahydrofuran (THF) as solvent.

Also, Mg(OH)₂ nanoparticle was employed as inorganic additive in membrane fabrication in order to improve the IEMs physicochemical properties. Currently no reports have considered incorporating Mg(OH)₂ nanoparticles into ion exchange membranes. The concentration effect of Mg(OH)₂ nanoparticles on the physicochemical properties of prepared homogeneous cation exchange nanocomposite membranes was evaluated. During this experiment, sodium chloride was employed as monovalent ionic solutions for the membrane characterization. The results are applicable for electro-membrane processes especially in electro-dialysis process for waste water treatment and water recovery.

MATERIALS AND METHODS

Poly (2,6-dimethyl-1,4-phenylene oxide) (PPO) with inherent viscosity of 0.57 dl/g in chloroform at 25 °C was obtained from Institute of Chemical Engineering of Beijing (China); SPPO was prepared by sulfonation of PPO according to the literature [22]. Polyvinylchloride (PVC) purchased from BIPC, Iran, grade S-7054. Tetrahydrofuran (THF) LR grade as solvent, sodium chloride, sulfuric acid (98%), sodium dodecyl sulfate (SDS) and Mg(NO₃)₂.6H₂O were supplied from Merck Company. Throughout the experiment, distilled water was used.

The test cell used in evaluation of membrane electrochemical properties consists of two cylindrical sections (vessel, each 140 cm³) made of Pyrex glass which are separated by membrane. The membrane was fixed between rubber rings. One side of each vessel was sealed by Pt electrode supported with a fragment of Teflon (Polytetrafluoroethylene) and the other side was equipped with a piece of porous medium to support the membrane. There are two orifices

on the top of each compartment for feeding and sampling aims. In order to minimize the influence of boundary layer during experiments and to diminish the concentration polarization on the vicinity of membrane's surface, both sections were stirred vigorously by magnetic stirrers (Model: Velp Scientifica Multi 6 stirrer). The membrane area was also 13.85 cm². The experiments were randomly repeated in triplicate and a desirable confidence limit (around 95%) was attained.

Morphological investigations of the membranes were performed via scanning electron microscopy (SEM) from Philips Company at an acceleration voltage of 25 kV. The samples were sputtered with gold to obtain a conductive surface. FT-IR spectra were recorded on Galaxy series FTIR5000 spectrophotometer. XRD patterns were recorded by a Philips, X-ray diffractometer using Ni-filtered Cu K α radiation.

Characterization of prepared membranes

Transport number and permselectivity

For potential measurements, the circular membrane was placed between the two half-cells containing NaCl solutions (0.01 and 0.1 mol dm⁻³). The membrane/solution interface potential was measured using two calomel reference electrodes (through KCl bridges) with the aid of a digital auto multi-meter and the NaCl solutions in the compartments were stirred mechanically. The membrane potential developed between the solutions contacting with both membrane surfaces is expressed via the Nernst equation which was employed to estimate the transport number of ions as follows:

$$E_m = (2t_i^m - 1) (RT/nF) \ln (a_1/a_2) \quad (1)$$

Where t_i^m is transport number of counter-ions in membrane phase, T is the temperature, R is gas constant, n is the electrovalence of counter-ion and a_1 , a_2 are electrolyte activities in the solutions specified by Debye–Huckel limiting law. The higher transport number of the counter-ions t_i^m in a membrane shows more permselectivity. The ionic permselectivity of membranes also is quantitatively expressed on the basis of the counter-ion migration through the IEMs [23-29].

$$P_s = (t_i^m - t_0) / (1 - t_0) \quad (2)$$

Where, t_0 is the transport number of counter-

ions in solution phase [30].

Permeability and flux

For the measurements of ionic permeability and the flux of ions, one side of the cell was filled with 0.1 M NaCl solution and another side with a 0.01 M solution. Using two stable platinum electrodes connected to the end of the compartments, a DC electrical potential (Dazheng, DC power supply, Model: PS- 302D) with optimal constant voltage was applied across the cell. By applying of electrical potential during the experiment, Na⁺ ions permeate through the membrane to the cathodic compartment and the pH of this region is increased as a result of hydroxide ions production. Therefore, in order to calculate the transported cations through the membrane the number of produced hydroxide ions in the cathodic section can be used. So the pH change in the cathodic region is a measure of the ions permeation (Δn) through the membrane.

In order to establish the equilibrium condition in two solution– membrane interfacial sections and to minimize the effect of boundary layers, both sections were strongly stirred via magnetic stirrers [31-33].

Membrane oxidative stability

To evaluate the oxidative stability of prepared membranes, they were soaked into 3% H₂O₂ aqueous solution containing 4 ppm Fe³⁺ at 25 °C for up to 60 h. The weight of the dried membranes (dried at 65 °C for 3 h) before and after the experiment was measured (using Mettler Toledo Group, Model: AL204). The percentage of the reduced weight can be attributed to the oxidative

stability of membrane [18, 20].

Synthesis of Mg(OH)₂ nanoparticles

Mg(NO₃)₂.6H₂O and SDS (mole ratio of 1:2) were dissolved in water were dissolved in 200 mL of distilled water. Under ultrasonic waves (100 W), 100 mL of NaOH solution 1 M (or ammonia) was then slowly added to the solution during 40 minutes. The precipitate was centrifuged and washed with distilled water, and later dried at 70 °C for 20 h in a vacuum dryer.

The chemical reaction involved in the formation of magnesium hydroxide is as follow:



Preparation of cation exchange nanocomposite membranes

The cation exchange nanocomposite membranes were prepared by casting solution technique. The membrane fabrication proceeded by dissolving the polymer binders (SPPO and PVC) in THF solvent. The mixture was mixed severely at room temperature to obtain a homogenous mixture. A certain amount of Mg(OH)₂ nanoparticles was dispersed in 10 ml THF with ultrasonic waves. The nanoparticles dispersion was then gradually added to the polymer solution. The new solution was mixed and stirred for 5 hours, followed by casting it on a clean and dry glass plate at 25 °C and was placed at room temperature. After the evaporation, the samples were treated at 50 °C, 65 °C, 85 °C, 105 °C (every temperature lasted 2 h). Finally, nanocomposite membranes were pretreated by dipping in HCl and NaCl solutions. A digital caliper device was applied

Table 1. Compositions of casting solutions in preparation of ion exchange nanocomposite membranes.

Membrane	Mg(OH) ₂ nanoparticle (additive:total solid) (w/w)
Sample 1	(0.5:100)
Sample 2	(1:100)
Sample 3	(2:100)
Sample 4	(3:100)

Polymer binder (SPPO:SPVC) (w/w), (7:3); solvent (THF:Polymer binder) (v/w), (10:1);

for measuring membranes thickness which confirmed that the thicknesses were maintained about 30-40 micrometers. The compositions of casting solutions are shown in Table 1.

RESULTS AND DISCUSSION

Mg(OH)₂ nanoparticles characterization

Scanning electron microscopy images of magnesium hydroxide nanoparticles, prepared by sodium hydroxide and ammonia addition are shown in Figs. 1 and 2, respectively. In both

conditions nanoparticles with average diameter size less than 100 nm were obtained.

The SEM images of Mg(OH)₂ nanoparticles after calcination are shown in Fig. 3. It seems by applying calcination a little agglomeration occurred; SEM images confirm nanoparticles with average diameter of 51nm have been obtained.

The XRD pattern of Mg(OH)₂ nanoparticles is shown in Fig. 4. XRD pattern of magnesium hydroxide is indexed as a pure hexagonal structure with suitable agreement to literature value

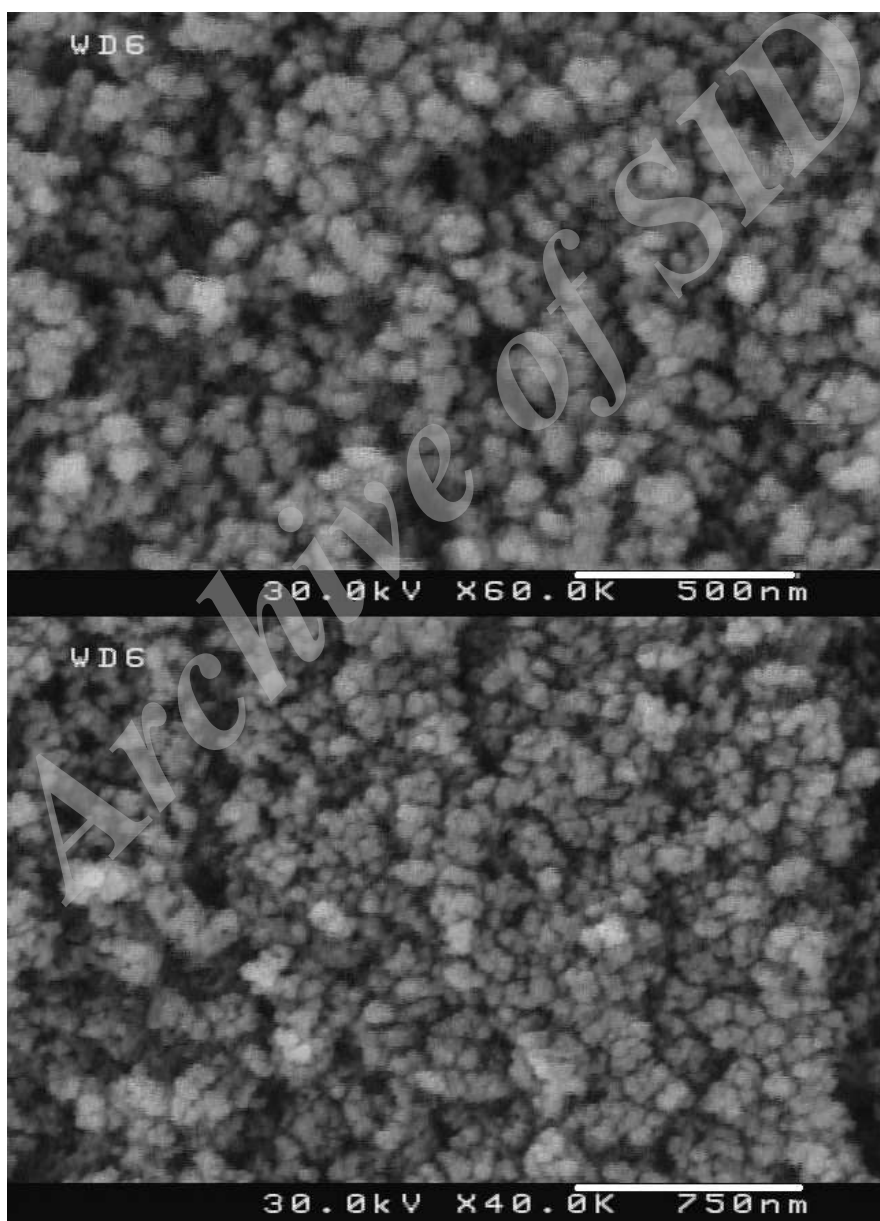


Fig. 1. SEM images of Mg(OH)₂ nanoparticles, prepared by NaOH addition

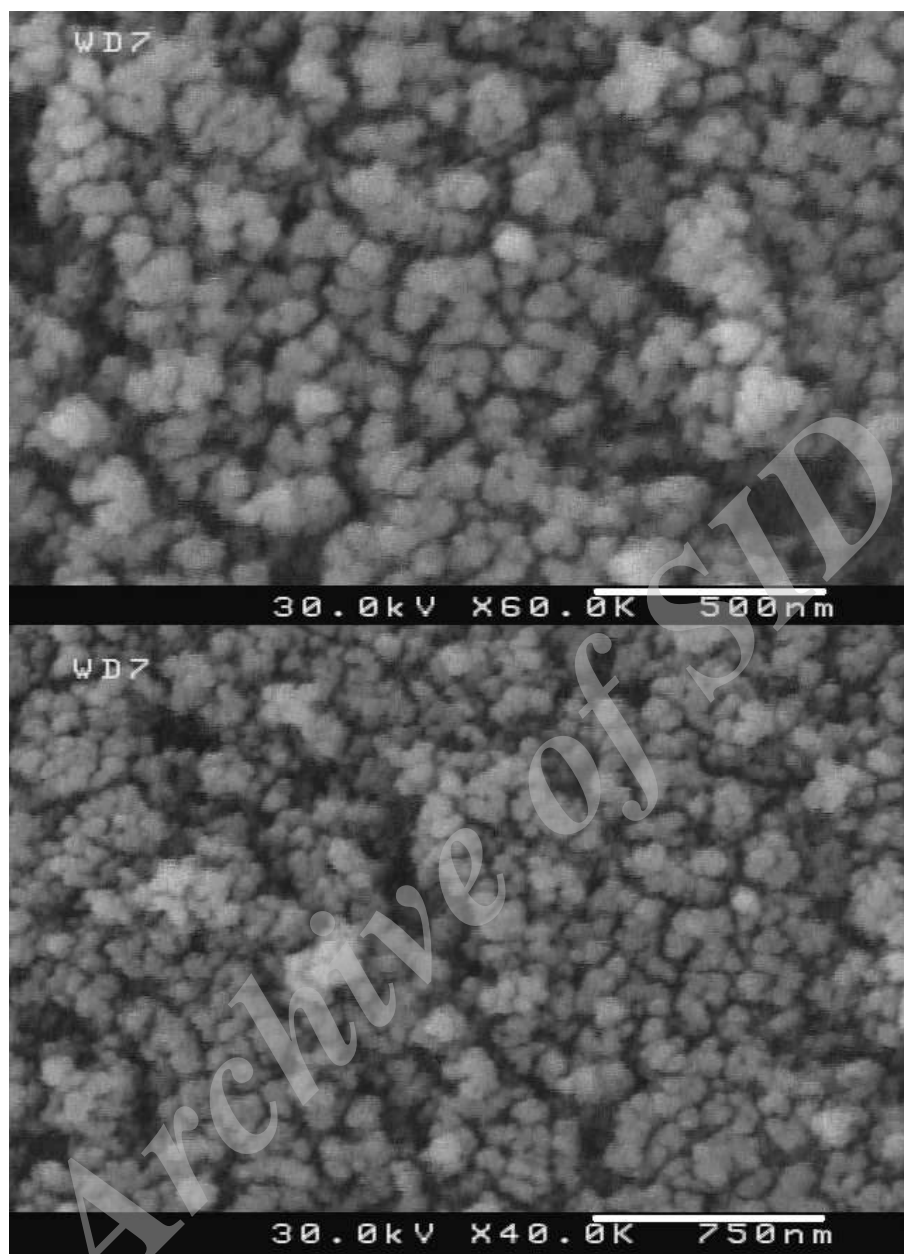


Fig. 2. SEM images of Mg(OH)₂ nanoparticles, prepared by NH₃ addition

(JCPDS card no. 44-1482, Space group: P-3m1, cell constants: a, b: 3.1441, c: 4.775 angstrom). The crystallite size evaluation was also performed using the Scherrer equation [34, 35].

$$D_c = 0.9\lambda / \beta \cos\theta \quad (4)$$

Where β is the width of the observed diffraction peak at its half maximum intensity (FWHM) and λ

is the X-ray wavelength (CuK α radiation, equals to 0.154 nm). The calculated crystallite size is about 15 nm.

FT-IR spectrum of the Mg(OH)₂ is shown in Fig. 5; the sharp absorption peak at 3695 cm⁻¹ is attributed to the O-H bond stretching vibration in the crystal structure. Absorption at 434 cm⁻¹ is assigned to the Mg-O stretching vibration in Mg(OH)₂ [36]. Broad absorption peak around

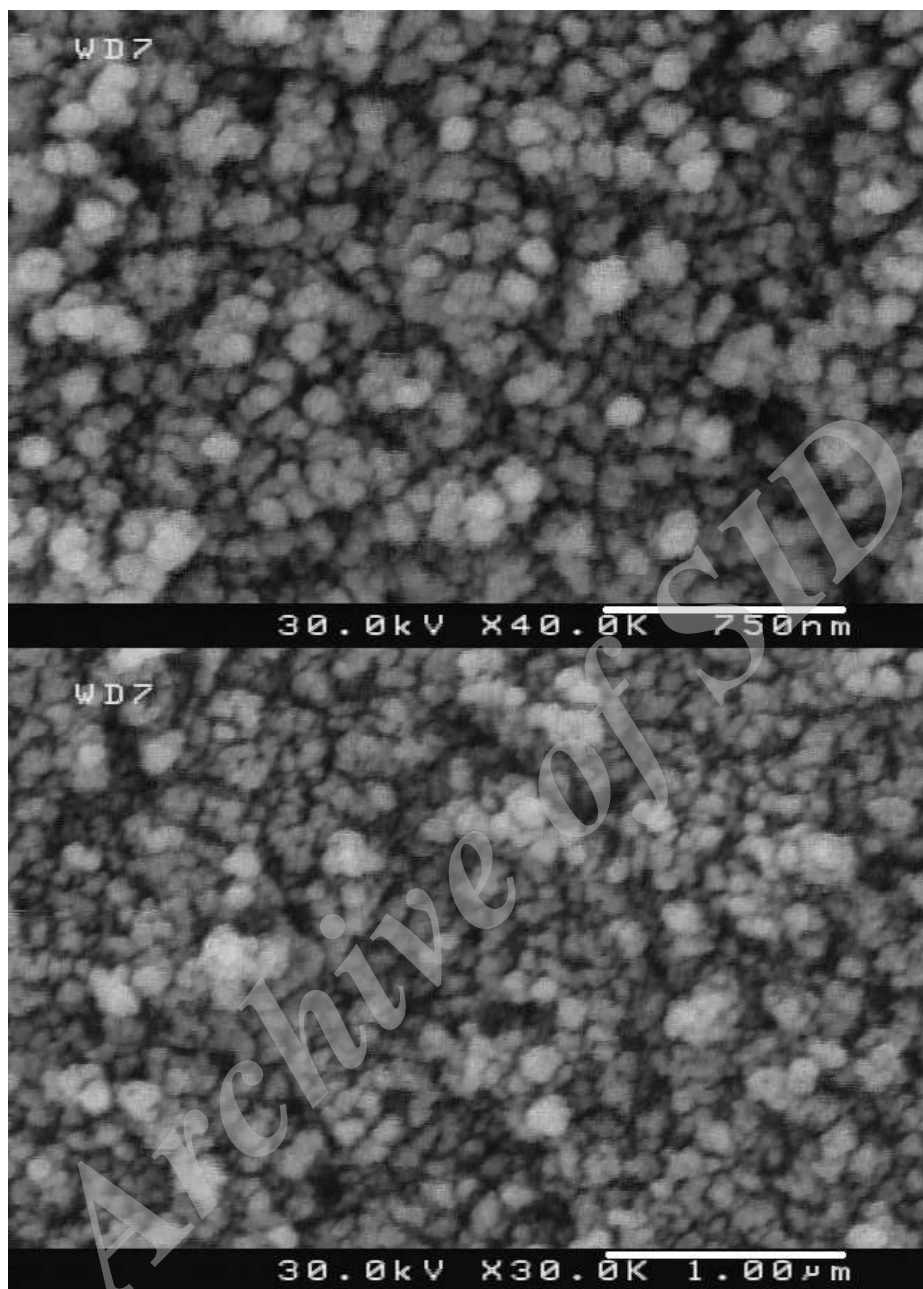


Fig. 3. SEM images of Mg(OH)₂ nanoparticles after calcination

3410 cm⁻¹ is related to O–H bonds of water and moisture that are adsorbed on the surface of nanostructures. FT-IR spectrum shows that the product does not show any intense IR-active peak correspond to impurities.

Permselectivity and transport number

The test cell used in evaluation of membrane electrochemical properties and the schematic

of nanocomposites membrane preparation are shown in Figs. 6 and 7 respectively. Also, the permselectivity and transport number of membranes are depicted in Fig. 8. The measurements were performed three times for each sample and the average values were reported in order to minimize the experimental errors.

At first, both increased with the increment of Mg(OH)₂ nanoparticle concentration to 1 wt%

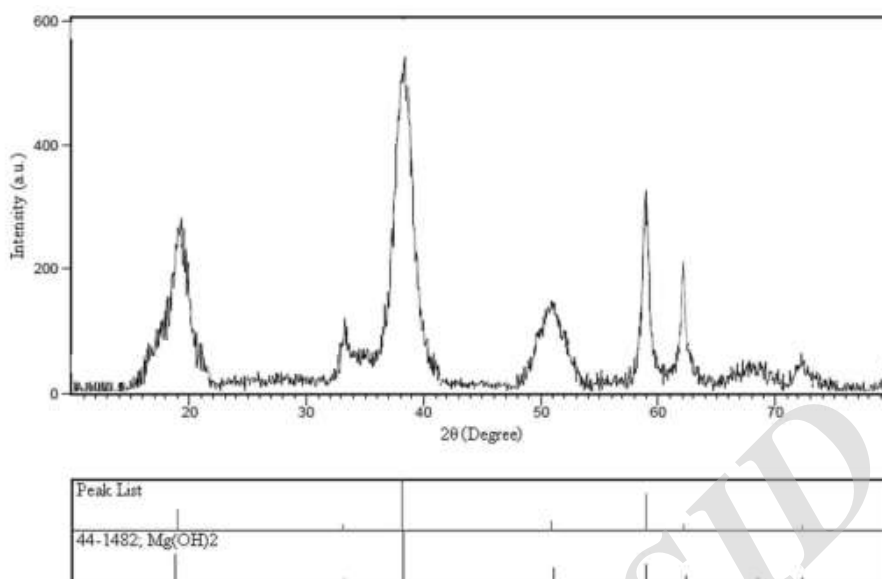


Fig. 4. XRD pattern of Mg(OH)₂ nanoparticles

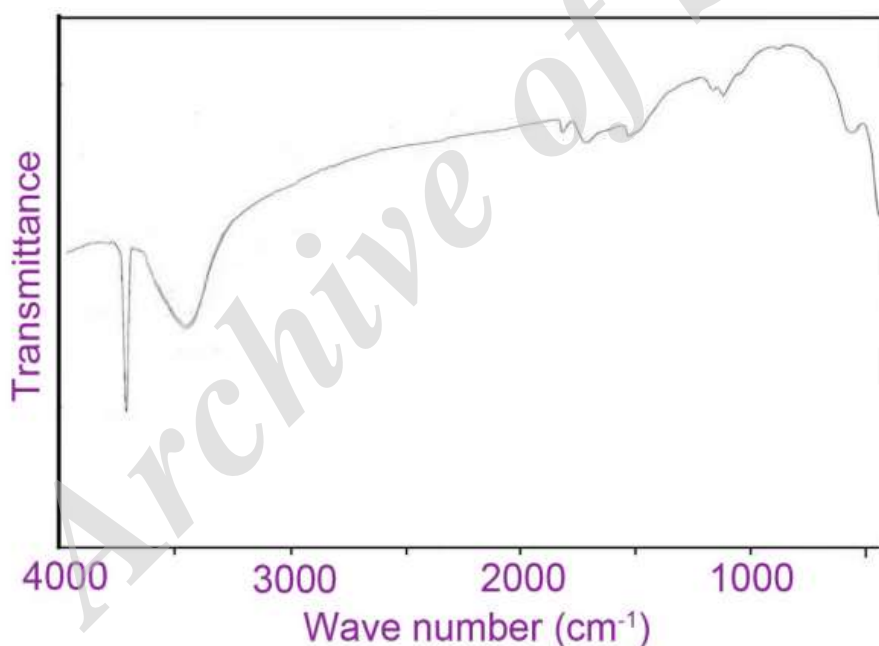


Fig. 5. FT-IR spectrum of the Mg(OH)₂ nanoparticles

(sample 2) in the casting solution. This trend can be elucidated with respect to the surface charge of the nanoparticles. Depending on the pH value of the solutions in the cell compartments the surface charge of the particles and hence the electrostatic forces can be controlled. In our experimental conditions (pH= 7.5) the surface of Mg(OH)₂ nanoparticles was negatively charged

[35]. Therefore, the existence of attractive electrostatic forces between magnesium hydroxide nanoparticles and Na⁺ ions provides higher possibility for the counter-ions transport which in turn leads to enhanced transport number of counter-ions. Also, with the increase of nanoparticle concentration (up to 1 wt%), the ionic pathways in the membrane matrix are partially

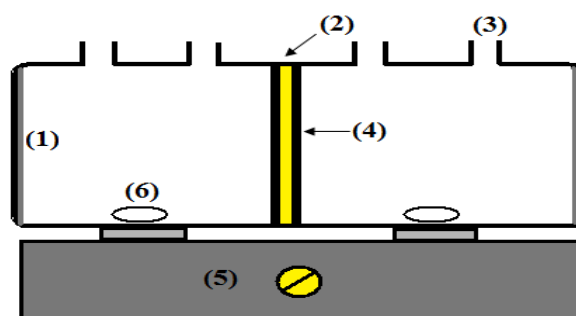


Fig. 6. Schematic diagram of test cell: (1) Pt electrode, (2) membrane, (3) orifice, (4) rubber ring, (5) stirrer, (6) magnetic bar

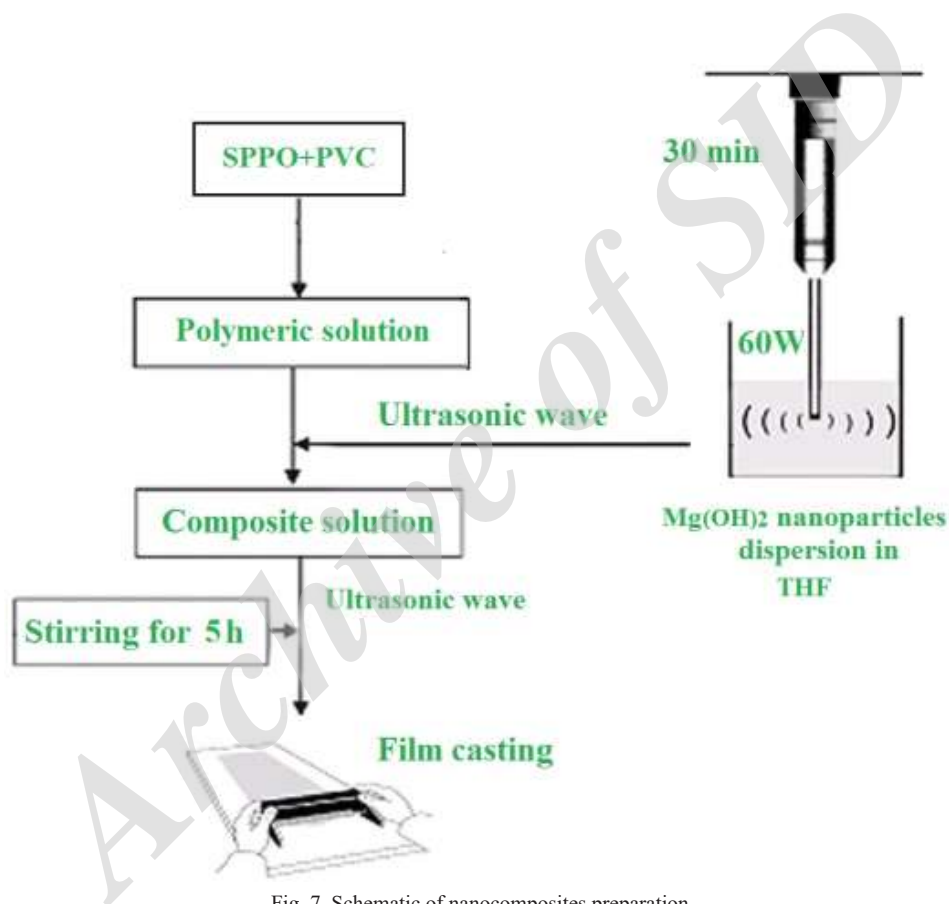


Fig. 7. Schematic of nanocomposites preparation

filled with Mg(OH)₂ nanoparticles and so passages are narrowed by them as space limiting factors. This enhances the membrane permselectivity. The permselectivity and transport number were diminished again with more additive loading from 1 to 3 wt%. More increment in filler loading (Mg(OH)₂) reduces the membrane selectivity due to enhancement of particles density in the casting solution which leads to discontinuity of polymer chains binder [31].

Ionic permeability and flux

According to occurred reactions in the cathodic and anodic compartments, the number of transported sodium ions through the membrane to cathodic part is equal to the generated OH⁻ ions in the cathodic compartment. Therefore, the results of ionic permeability and flux were deduced from pH changes in cathodic region. The schematic of ions transport through ion exchange membranes is shown in Fig. 9.

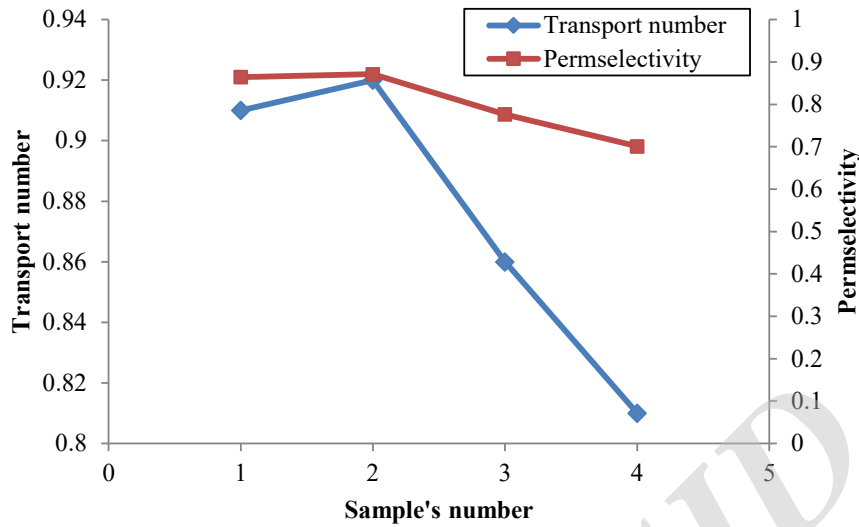


Fig. 8. The permselectivity and transport number of prepared membranes

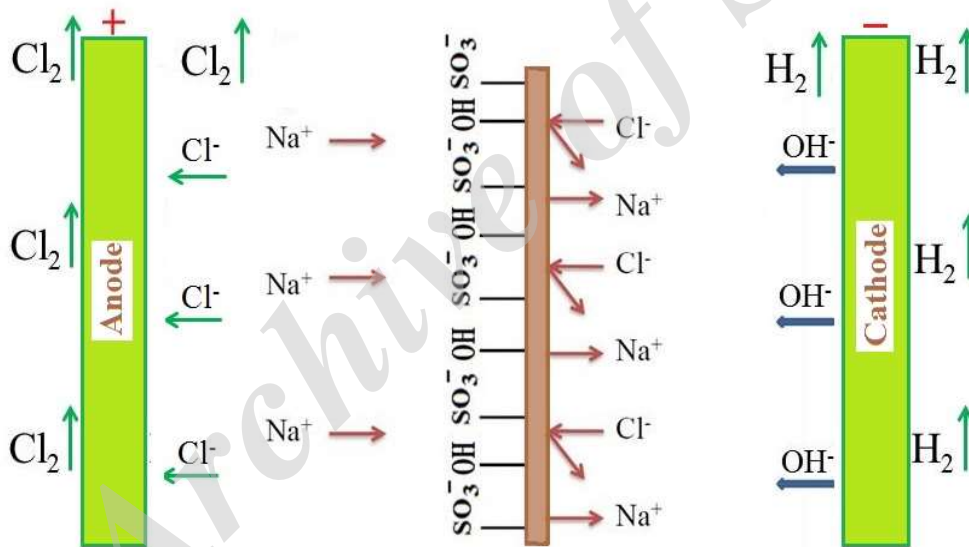


Fig. 9. Schematic diagram of ions transport through ion exchange membranes

Results (Fig. 10) showed that the ionic permeability and flux were firstly increased with increment in nanoparticles loading up to 2 wt.% (sample 3) in the casting solution. This is essentially because of the increased water channels and porosity in the membrane by addition of inorganic nanoparticles, which simplifies the migration of ions [12]. When more filler was added in the polymer matrix (sample 4), the nanoparticles tended to aggregate and thus tortuosity was increased due to the more

polymer–filler aggregates interaction. Therefore, the ionic permeability and flux of sample 4 are lower than other samples in this study.

Membrane oxidative stability

The oxidative stabilities are presented in Table 2. The results indicated that the oxidative stability of membranes decreased with increasing the filler loadings in the casting solution. The increasing of water diffusion leads to higher oxidant's diffusion in the membranes network and more weight loss

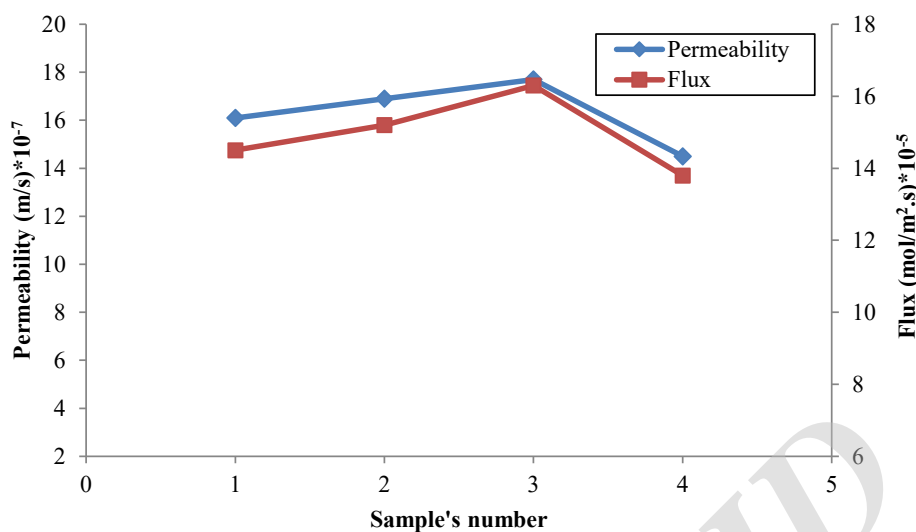


Fig. 10. The ionic permeability and flux of prepared cation exchange membranes

Table 2. The effect of additive loading on weight loss in the oxidative stability test.

Sample's number	Weight loss (%)
1	14.33
2	15.21
3	16.14
4	16.78

during the experiment.

CONCLUSION

A new type of ion exchange membranes containing magnesium hydroxide nanoparticles was successfully prepared. The existence of Mg(OH)₂ nanoparticles had a considerable effect on the structure and properties of the ion exchange membranes, which in turn influenced the overall membranes performance. It was found that Mg(OH)₂ nanoparticles could affect overall electrochemical and physicochemical properties including transport number, permselectivity, ionic permeability, flux of ions and membrane oxidative stability. In general, the content and type of inorganic nanoparticles strongly influenced the

structure and properties of the nanocomposite membranes. The membrane with 1 wt% Mg(OH)₂ nanoparticles exhibited higher permselectivity and transport number in comparison with other prepared membranes in this research.

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CONFLICT OF INTEREST

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

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