



Electrochemical characterization of mixed matrix heterogeneous cation exchange membrane modified by aluminum oxide nanoparticles: Mono/bivalent ionic transportation



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ABSTRACT

Polyvinylchloride/aluminum oxide nanoparticles mixed matrix heterogeneous cation exchange membranes were prepared by solution casting technique. The effect of Al₂O₃ nanoparticles concentration on membrane electrochemical properties was studied. It was found that membrane water content was enhanced by increase of additive concentration in prepared membrane. Increase of aluminum oxide nanoparticles loading ratio in casting solution also caused to decrease in ion exchange capacity. Obtained results revealed that membrane permselectivity and transport number were improved initially in sodium chloride and barium chloride ionic solutions by increase of aluminum oxide nanoparticles concentration up to 2 wt% in prepared membranes and then showed decreasing trend by more increase in additive content. Also membranes exhibited lower selectivity and transport number for bivalent ions in comparison with monovalent ones. Furthermore, membrane ionic permeability and flux were decreased initially by increase in aluminum oxide nanoparticles loading ratio up to 2 wt% and then increased again by more additives concentration. Opposite trend was found for membrane areal electrical resistance. Results showed that modified membranes in this study are comparable with that of other commercial ones.

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1. Introduction

In the filed of separation science and technology, membranes have obtained much attention in diverse industries and also daily humans' life [1–3]. Among these, ion exchange membranes are widely utilized as active separators in different electrically driven processes such as electrodialysis for desalting brackish waters, reconcentrating brine from seawater and production of table salt. Moreover, IEMs are efficient tools in resource recovery, food and pharmacy processing and environmental protections such as treating industrial and biological effluents processes [2–14]. In ion exchange membranes charged groups are attached to polymer backbone and freely permeable to opposite sign ions under an electrical field influence [1,4,15]. In these processes, ion interactions with membrane, water, and with each other occur in complex fashions. Knowledge of the electrochemical properties of ion exchange membranes is important factor

behind decisions about their applicability in separation processes [11,16–19].

Preparing IEMs with special physico/chemical characteristics may be as vital step in future chemical and waste treatment application [3,11,20–22]. Variation of functional groups, selection of different polymeric matrices, polymers blending, use of various additives, alteration of cross-link density and surface modifications are some important ways to obtain superior IEMs [1,6,11,13,15,18,23–31].

Preparing heterogeneous cation exchange membranes with appropriate electrochemical properties for the application in electrodialysis processes related to water recovery and treatment was the primary target of the current research. For the purpose, *polyvinylchloride/aluminum oxide nanoparticles* mixed matrix heterogeneous cation exchange membranes were prepared by solution casting techniques using cation exchange resin powder as functional groups agent and tetrahydrofuran as solvent. PVC is a durable polymer with suitable biological and chemical resistance [32–34]. Also, aluminum oxide nanoparticle was employed as inorganic filler additive in membrane fabrication in order to improve the IEMs physico/chemical properties. Utilizing of inorganic particles or fillers especially nano materials into polymeric materials has been

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examined in many applications to enhance the mechanical, thermal and chemical stabilities of polymeric matrixes in severe conditions such as high temperature and strongly oxidizing environment and also to improve the separation properties of membranes based on the synergism between the organic–inorganic components properties [1,10,24,29–31,35–37].

Al₂O₃ nanoparticle is also a new class of advanced and stable inorganic materials with very interesting features and capacity such as hydrophilic characteristic, non-toxic, highly abrasive and resistant and inexpensive which has been utilized in UF membrane for water treatment [29–31].

Currently no research has been considered incorporating aluminum oxide nanoparticles into ion exchange membranes and the literature is silent on characteristics and functionality of electro-dialysis IEMs prepared using Al₂O₃ nanoparticles.

The effects of Al₂O₃ nanoparticles concentration in the casting solution on electrochemical characteristics of prepared heterogeneous cation exchange membranes were studied. During this experiment, sodium chloride and barium chloride were employed as monovalent and bivalent ionic solutions for the membranes characterization. The results are valuable for electro-membrane processes especially electro-dialysis process for water recovery and waste water treatment. Moreover, sonication was employed in membrane preparation to achieve better homogeneity in membrane matrix and to obtain the balance between electrochemical properties and mechanical integrity.

2. Materials and methods

2.1. Materials

Polyvinylchloride (PVC, grade S-7054, density: 490 g/l, viscosity number: 105 cm³/g) supplied by Bandar Imam Petrochemical Company (BIPC), Iran, was used as binder. Tetrahydrofuran (THF, molar mass: 72.11 g/mol, density: 0.89 g/cm³) was employed as solvent. Aluminum oxide nanoparticle (Al₂O₃, high purity) with particle size of 80 nm was provided from US Research Nanomaterials, USA. Cation exchange resin (Ion Exchanger Amberlyst[®] 15, strongly acidic cation exchanger, H⁺ form – 4.7 mequiv./g dry weight) by Supelco was used in membrane preparation. All other chemicals were supplied by Merck. Throughout the experiment, distilled water was used.

2.2. Preparation of homemade membrane

Heterogeneous cation exchange membranes were prepared by casting solution technique. In order to undertake the membrane preparation, resin particles were dried in oven at 30 °C for 48 h and then pulverized into fine particles in a ball mill and sieved to the desired mesh size. The ion exchange resin with desired particles size (–300 +400 mesh) was used in membranes fabrication. The preparation proceeded by dissolving the polymer binder into THF solvent in a glass reactor equipped with a mechanical stirrer (Model: Velp Scientifica Multi 6 stirrer) for more than 5 h. This was followed by dispersing a specific quantity of grinded resin particle as functional groups agents and aluminum oxide nanoparticles as additive in polymeric solution, respectively. The mixture was mixed vigorously at room temperature to obtain uniform particle distribution in the polymeric solution. In addition, for better dispersion of particles and breaking up their aggregates, the solution was sonicated for 1 h using an ultrasonic instrument. Sonication of solid particles increases the viscosity of casting solution by reducing aggregation and sedimentation [38] and reduces the evaporation rate of casting solvent. This improves the polymer chains relaxation as well as its conformation with particles surfaces and promotes the compatibility of particles

and binder [1,38,39]. This also reduces the amount of cracks and fissures between polymer binder and particles and so improves the membrane selectivity. Moreover, excessive homogeneity and uniform distribution of particles on membrane surface and in the bulk of membrane matrix provide superior conducting regions in membrane and generate easy flow channels for counter-ions transportation. Presence of more conducting region can strengthen the intensity of uniform electrical field around the membrane and decrease the concentration polarization phenomenon [40].

The mixing process was repeated for another 30 min using the mechanical stirrer. The mixture was then cast onto a clean and dry glass plate at 25 °C. The membranes were dried at ambient temperature (25 °C) for 30 min and immersed in distilled water. As the final stage, membranes were pretreated by immersing in 0.1 M HCl (for 12 h) and 0.5 M NaCl (for 48 h) solutions respectively. The thickness of the membrane was maintained between 80 and 100 μm (measured using digital caliper, Electronic outside Micrometer, IP54 model OLR). The summary of membrane preparation procedure and composition of casting solution are given in Tables 1 and 2 respectively.

2.3. Test cell

The electrochemical properties measurements for the prepared membranes were carried out using the test cell as reported earlier [1,23,24]. The cell consists of two cylindrical compartments made of Pyrex glass which are separated by membrane. One side of each vessel was closed by Pt electrode and the other side by membrane. The top of each compartment contained two orifices for feeding and sampling purposes. In order to minimize the effect of boundary layer during experiments and to establish the concentration polarization on the vicinity of membrane's surface, both sections were stirred vigorously by magnetic stirrers.

2.4. Membrane characterization

2.4.1. Water content

The water content was measured as weight difference between the dried and swollen membrane. The wet membrane was

Table 1
Flowsheet of membrane preparation procedure.

The procedure for IEMs preparation	
Step 1	Resin particles drying (at 30 °C for 48 h)
Step 2	Resin particles pulverizing (–300 +400 mesh)
Step 3	Polymer dissolving into solvent (for 5 h)
Step 4	Resin particles and additive dispersing in polymeric solution
Step 5	Sonication of polymeric solution (for 1 h)
Step 6	Mixing of polymeric solution (for another 30 min)
Step 7	Casting (at 25 °C)
Step 8	Film drying (at 25 °C for 30 min) and immersing in water
Step 9	Membranes pretreatment by HCl and NaCl solutions

Table 2
Composition of casting solution used in preparation of mixed matrix cation exchange membranes.^a

Membrane	Aluminum oxide nanoparticles (Al ₂ O ₃ nanoparticle:(PVC + resin)), (w/w)
Sample 1 (S1)	0.0:100
Sample 2 (S2)	2.0:100
Sample 3 (S3)	4.0:100
Sample 4 (S4)	8.0:100
Sample 5 (S5)	16.0:100

^a Solvent to polymer binder (THF:PVC) (v/w), (20:1); resin particles (resin:polymer binder) (w/w), (1:1).

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