



Prevention of bacterial adhesion on polyamide reverse osmosis membranes via electrostatic interactions using a cationic phosphorylcholine polymer coating



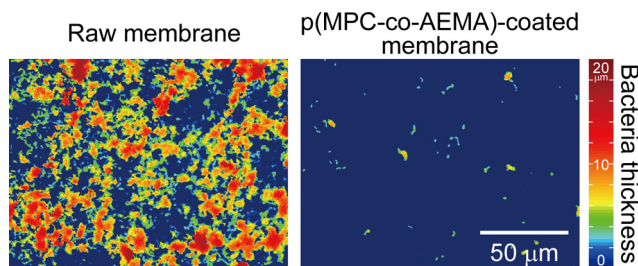
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HIGHLIGHTS

- Polyamide reverse osmosis membranes were coated with cationic phosphorylcholine.
- The cationic phosphorylcholine polymer adsorbed on the anionic membrane surface.
- The polymer was stably adsorbed under high ionic strength conditions.
- The coated membranes had high resistance to bacterial adhesion.

GRAPHICAL ABSTRACT



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ABSTRACT

A simple and easy anti-adhesive coating method against bacteria via electrostatic interaction was developed for polyamide reverse osmosis (RO) membranes using a cationic phosphorylcholine polymer. A commercial polyamide RO membrane was immersed into an aqueous solution of phosphorylcholine polymer containing cationic amino groups, poly[2-methacryloyloxyethyl phosphorylcholine (MPC)-co-2-aminoethylmethacrylate (AEMA)] (p(MPC-co-AEMA)). From the results of contact angle and surface potential measurements, the surface of the coated RO membrane became more hydrophilic than that of raw membranes and had a neutral charge. Conversely, the surface of an RO membrane immersed in an aqueous solution of MPC homopolymer without AEMA groups was not coated by the polymer. Therefore, p(MPC-co-AEMA) was adsorbed via electrostatic interaction between the cationic amino groups of AEMA and anionic carboxylic groups on the polyamide RO membrane. X-ray photoelectron spectroscopy showed the existence of phosphorylcholine groups from p(MPC-co-AEMA) on the coated membranes. The result of quartz crystal microbalance with dissipation monitoring measurements showed that adsorbed p(MPC-co-AEMA) was hardly desorbed from the polyamide surface in a high ionic strength solution at least for one day. The coated RO membrane had high resistance to bacterial adhesion and retained its original rejection performance.

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1. Introduction

Water treatment using reverse osmosis (RO) membranes saves energy and space compared with other processes and has therefore been widely applied to various systems, such as desalination and

waste water recycle. Commercially available RO membranes are mainly polyamide composite membranes composed of a polyamide rejection layer and polysulfone support membrane [1], which have advantages of high water permeability and good rejection performance. The use of RO membranes enables treatment of seawater and industrial waste water containing various organic matters, including bacteria [2–4], proteins and polysaccharides [5–8]. Organic matters cause a decrease in membrane performance by adhesion onto the membrane surface and blocking of membrane

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pores. In particular, bacteria grow onto the membrane surface and form biofilms containing extracellular polymeric substances (EPS) such as proteins and polysaccharides [3,9]. This phenomenon is called “biofouling”. Adhered organic matters are generally removed by chemical treatment, including chlorine [10]. However, amide bonds in the polyamide membranes are cleaved by chlorine treatment, causing membrane performance to decrease [11]. Prevention of bacterial adhesion is important for maintaining RO membranes and long-term operation.

Various approaches for preventing bacterial adhesion onto membrane surfaces have been reported [12]. Surface modification with polyethylene glycol (PEG) prevents the adhesion of biological materials such as proteins [13,14], cells [15], and bacteria [16] because of PEG’s high hydrophilicity and large extruded volume [17]. PEG has already been applied as a surface modifier to prevent adhesion of organic materials such as surfactants and proteins [18–20]. Recently, zwitterionic polymers such as sulfobetaine polymers and phosphorylcholine polymers have also been reported to effectively prevent adhesion of biological materials [21–24]. Zwitterionic polymers have a similar structure to biological membranes and interact with water molecules via electrostatic interaction more strongly than PEG, which interacts via hydrogen bond formation [25]. Modification with zwitterionic polymers is effective for prevention of bacterial adhesion onto membrane surfaces [26,27].

Surface modification with these polymers is generally carried out using a covalent grafting method [13,16,18–20,22,23] or coating method [21,24,28,29]. Although grafting methods have the advantage of long-term stability, the modification process causes complexity in the overall membrane fabrication process. On the other hand, coating methods, such as the dip coating method and spin coating method, can immobilize target molecules much more simply. In addition, modification of a target surface is not required. Ishigami et al. reported that RO membranes multilayer-coated using oppositely charged polyelectrolytes via electrostatic interaction had resistance against adsorption of proteins, although their water permeability was decreased [30]. For water treatment applications, surface modification is required to retain the water permeability of the original membranes.

In this study, we developed a simple and easy modification method for coating phosphorylcholine polymer onto a RO membrane via electrostatic interaction to prevent bacterial adhesion. We used poly[2-methacryloyloxyethyl phosphorylcholine (MPC)-co-2-aminoethylmethacrylate (AEMA)] (p(MPC-co-AEMA)), as a cationic phosphorylcholine polymer. Cationic phosphorylcholine polymer has been reported as DNA carriers for drug delivery [31–33]. A commercial polyamide RO membrane was immersed into an aqueous solution of p(MPC-co-AEMA), and coated via electrostatic interaction between the cationic amino groups of p(MPC-co-AEMA) and the anionic carboxyl groups on the RO membrane. The surface properties of the coated RO membranes were evaluated by contact angle, surface potential, and X-ray photoelectron spectroscopy (XPS). The coating behavior and coating stability of p(MPC-co-AEMA) on the RO membrane was characterized by a quartz crystal microbalance with dissipation (QCM-D) measurement. The water permeability, salt rejection, and anti-adhesive properties against bacteria were evaluated for membrane performance.

2. Methods

2.1. Materials

All chemicals, if not otherwise specified, were obtained from Wako Pure Chemical Industries (Osaka, Japan) and were used without further purification. All aqueous solutions were

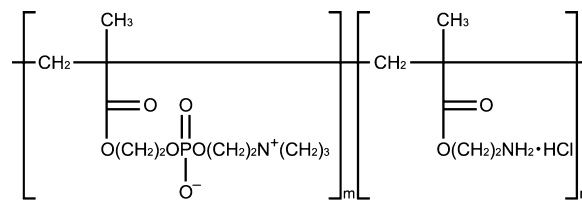


Fig. 1. Chemical structure of p(MPC-co-AEMA), $m:n=9:1$.

prepared with Milli-Q water. A commercial polyamide RO membrane, ES20, was obtained from Nitto Denko Corporation (Osaka, Japan). MPC homopolymer and p(MPC-co-AEMA) (MPC:AEMA=9:1, random copolymer; Fig. 1), kindly provided by NOF Corporation (Tokyo, Japan), were used as phosphorylcholine polymers. The weight-average molecular weight of each phosphorylcholine polymer was evaluated by gel permeation chromatography (GPC) using a refractive index detector (RID-10A; Shimadzu Corporation, Kyoto, Japan) and Shodex SB-805HQ column (Showa Denko, Tokyo, Japan) at 40 °C. A mixed solvent comprising 0.1 M NaNO₃ aqueous solution and acetonitrile (8/2, v/v) was used as the eluent. The weight-average molecular weights of MPC homopolymer and p(MPC-co-AEMA) were 4.6×10^5 and 9.7×10^5 , respectively.

2.2. Membrane coating using cationic phosphorylcholine polymer

A polyamide RO membrane was coated by phosphorylcholine polymer via electrostatic interaction. A commercial polyamide RO membrane was immersed into an aqueous solution of 0.1 wt% phosphorylcholine polymer for 3 h in a refrigerator and washed by gentle shaking twice in an aqueous solution of 3.5 wt% NaCl for 1 h to remove non-specifically adsorbed polymers.

2.3. Characterization of surface properties

We evaluated surface hydrophilicity, surface potential, and elemental composition of the membrane surface. To evaluate surface hydrophilicity of the membranes, the water contact angle was measured using a contact angle meter (DM-300; Kyowa Interface Science, Saitama, Japan). To evaluate the surface potential of the membranes, the zeta-potential (ζ -potential) was measured with an electrophoretic light-scattering apparatus (ELS-4000K; Otsuka Electronics, Osaka, Japan) in 10 mmol/L NaCl aqueous solution at pH 7.0. The chemical composition of the membrane surface was analyzed using an XPS instrument (JPS-9010MC, JEOL, Tokyo, Japan). The membrane morphology was observed using a field emission scanning electron microscope (FE-SEM; JSF-7500F; JEOL), the same as used in our previous study [34].

2.4. Characterization of coating behavior and coating stability

The coating behavior and coating stability of p(MPC-co-AEMA) on the RO membrane were characterized using a QCM-D instrument (Q-sense E1; BiolinScientific, Västra Frölunda, Sweden). A polyamide-coated quartz sensor was prepared using an interfacial polymerization method with trimesoyl chloride (TMC; Sigma-Aldrich Corp., St. Louis, MO) and *m*-phenylenediamine (MPD), commonly used to fabricate polyamide RO membranes [11,35]. Steiner et al. formed a polyamide layer on a gold surface, producing a surface similar to of a polyamide RO membrane [36]. We modified their protocol for a QCM-D sensor, as shown in Fig. 2. A gold-coated quartz sensor (QSX 301; BiolinScientific) was immersed into an ethanol solution of 1 mmol/L 2-aminoethanethiol overnight to aminate the sensor surface. The aminated sensor was washed with ethanol twice, dried, and washed with

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