Kraton based ionic polymer metal composite (IPMC) actuator

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ABSTRACT

In this study, the tip displacement, proton conductivity, current density, water uptake and ion exchange capacity of kraton non-perfluorinated IPMCs are examined and the results are compared with nafion based IPMCs membranes. The water holding capacity of kraton membrane and nafion film was found to be 308.69% and 16.20%, at 65 °C and 45 °C within 10 h of immersion time, respectively. The kraton membrane and nafion film have the ion exchange capacity of 1.9 and 0.75 meq g^{-1} of dry membrane, respectively. SEM studies revealed that the morphology of nafion membrane was negligibly affected after performing the action experiment while in kraton membrane ruptures and notable spaces at joint were observed. The electrical properties revealed better actuation performance of kraton membrane. The tip displacement for nafion and kraton membrane was also carried out at 3 V DC electrical voltages. Kraton film showed larger displacement and therefore actuation as compared to that of nafion film.

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1. Introduction

Ionic polymer metal composites (IPMCs) have been considered as a promising candidate for electric stimuli sensitive materials in actuators, bioinspired artificial muscles and sensors because of their advantageous properties like operation at low electric voltage (1–5 V), large dynamic deformation, lighter weight, flexibility and precise sensing ability [1–9]. Typically, an IPMC comprises of a neutralized semi permeable ion exchange polymer membrane coated with metal Pt or Au as electrode at both sides and water as inner medium for the dissociation for metal cation. When an electric field is applied the metal cations move towards the negative electrode along with water molecules. Therefore, the polymer membrane swells near the cathode which causes a strain near the cation rich region in IPMC membrane, resulting in bending motion in the film towards the anode [10]. Under dry conditions the cross-linked cations are not free to move. However, in wet condition (on hydration) cations are surrounded by water molecules to make the whole film mobile. Thus, transduction in the IPMCs film is because of the movement of cations with the water molecules [11]. Typically, per-fluorinated IPMC membranes with the trade name of nafion are being used as actuators and dynamic sensors because of its useful properties like high proton exchange capacity, thermal, mechanical and chemical stabilities [12–23]. However, its high cost as well as high evaporation of water molecules under applied voltage limits its further application. Therefore, researchers are working towards developing low cost non-perfluorinated membranes having high water holding capacity even at high temperatures, to replace the commercial nafion membranes [24–27,19]. In this study, non-perfluorinated kraton polymer based ionic polymer metal composite membranes were prepared and characterized for bending actuation. The results are compared with that of nafion membrane.

2. Experimental

2.1. Material

A nafion membrane (0.05 mm thick, NRE-212) (Sigma Aldrich), tetraamineplatinum(II) chloride monohydrate [Pt(NH$_3$)4Cl$_2$ ·H$_2$O (Crystalline)] (Alfa Aesar, USA) and a non-perfluorinated kraton [pentablock copolymer poly((t-butyl-styrene)-b-(ethylene-r-propylene)-b-(styrene-r-styrene sufonate)-b-(ethylene-r-propylene)-b-(t-butyl-styrene) (tBS-EP-SS-EP-tBS))](MD9200) (Nexar Polymer, USA), sodium borohydride and hydrochloric acid (35%) (Thomas Baker Pvt. Ltd., India) and ammonium hydroxide (25%) (Merk Specialties Pvt. Ltd., India) were used as received without any purification.

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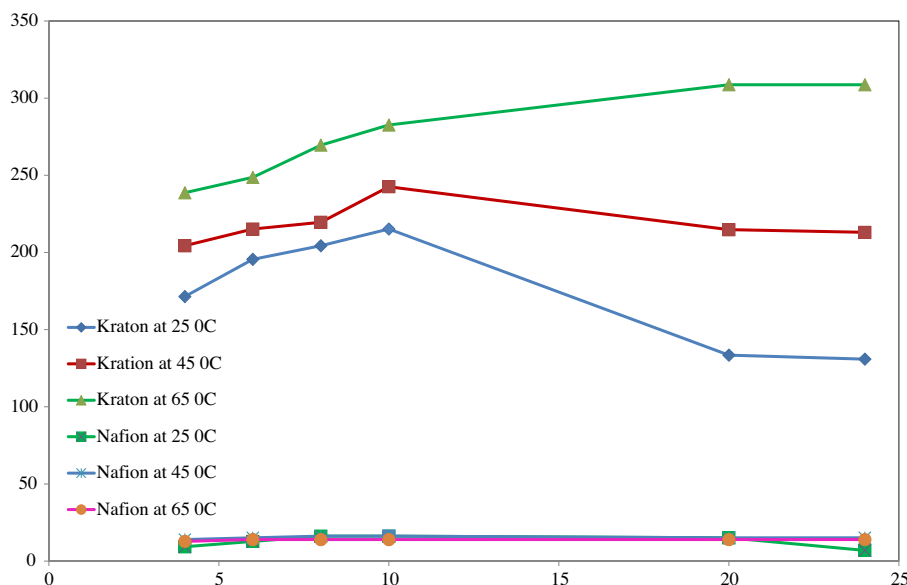
Fig. 1. Water uptake of kration and nafion based IPMCs films at room temperature (25 ± 3 °C), 45 °C and 65 °C for a time period of 4 h, 6 h, 8 h, 10 h, 20 h and 24 h.

2.2. Membrane preparation

An ionic polymer metal composite (IPMC) film of kraton polymer was prepared by casting 5 ml of kraton in a petri dish (50 mm × 17 mm) with the help of a pipette. After casting the polymer solution, the petri dish was covered with Whatman filter paper (No. 1) for slow evaporation of the solvents at room temperature (25 ± 3 °C) for 5 h. However, evaporating the solvent in a thermostated oven at and above 30 °C solidifies kraton film with visible surface cracks. After drying, the film was removed from the petri dish with the help of a foreship and spatula. The film was stored in a desiccator to carry out further studies.

2.3. Water uptake

To find out the water uptake capacity of nafion and kraton polymer films were kept in demineralized water. A series of experiments for water uptake at different time duration and temperatures were performed. The experiments were performed at room temperature (25 ± 3 °C), 45 °C and 65 °C, for different times 4 h, 6 h, 8 h, 20 h and 24 h as shown in Fig. 1.

2.4. Ion exchange capacity

The films were converted into H^+ ion form by placing in 1.0 M HNO_3 for 24 h followed by neutralization with demineralized water and dried at 45 °C. A 0.25 g of polymer film was cut into small pieces and packed into a glass column. The acidic form of the film was converted into Na^+ form by passing 1 M $NaNO_3$ through the column to elute the H^+ ions keeping a slow flow rate (~0.5 ml min^{-1}). The effluent was titrated against 0.1 M NaOH solution using phenolphthalein indicator. The ion exchange capacity of polymer film in meq g^{-1} of dry film was obtained using following formula (1) and given in Table 1.

Ion exchange capacity

$$= \frac{\text{Volume of NaOH consumed} \times \text{Molarity of NaOH}}{\text{Weight of the dry membrane}} \quad (1)$$

2.5. Electroless plating

The fabrication of ionic polymer metal composite (IPMC) membranes was started with surface roughening of membranes on both sides which was carried out by using mild sandpaper and followed by cleaning ultrasonically for 30 min, i.e., electroless plating method described in the literature [28,29].

After cleaning, the membranes were treated with an aqueous solution of 2.0 N HCl in hot air oven at 45 °C followed by washing with demineralized water for 1 h. The membranes were treated with 4.5 ml of aqueous solution of 0.04 M tetraammineplatinum(II) chloride monohydrate and 0.1 ml of 5.0% aqueous solution of NH_4OH, with constant stirring up to 5 h at room temperature. After the exchange of protons of membranes with platinum ions, the membranes were stirrer in distilled water for 15 min to remove excess platinum absorbed at the surface of the membranes and then transferred to another flask. A 0.5 ml of 5% aqueous solution of $NaBH_4$ was added after every 30 min for reduction of platinum ions into Pt metals. Further, 5 ml of $NaBH_4$ solution was added followed by stirring for 1 h at room temperature. The membranes were washed with distilled water for termination of reduction reaction. Finally, membranes were converted into acidic form by placing membranes in 0.1 M HCl solutions at room temperature up to 1.5 h.

2.6. Proton conductivity

The proton exchange capacity of the ionic polymer metal composite (IPMC) membrane is one of the most important features to achieve bending which occurs because of forming hydronium ions by the existing protons at the IPMC membrane in hydrated state. The bending movement of IPMC membrane is because of movement of hydrated cation towards the cathode as shown in Scheme 1. The proton conductivity of hydrated IPMC film (1 cm × 3 cm) was

Table 1
Ion exchange capacity and proton conductivity of kraton and nafion based IPMCs films.

Material	Ion exchange capacity (meq g^{-1} of dry membrane)	Proton conductivity (σ) (S cm^{-1})
Nafion membrane	0.75	4.026
Kraton membrane	1.9	17.15
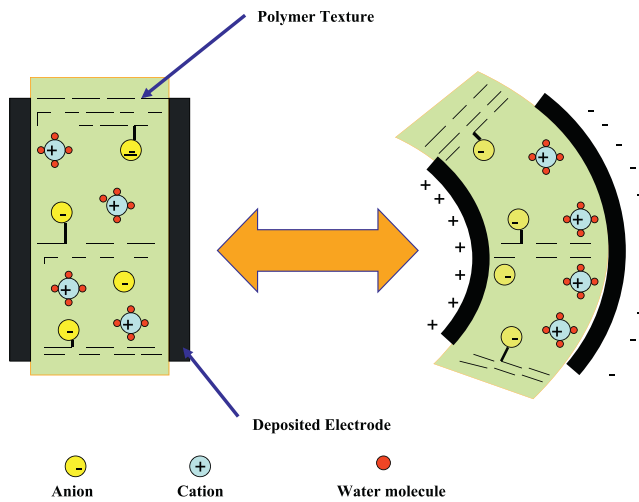
Scheme 1. Graphical representation of bending mechanism for IPMC membrane.

determined by an impedance analyzer (FRA32M.X), connected with Autolab 302N modular potentiostat/galvanostat used over a frequency of 100 kHz and an AC perturbation of 10 mV was applied to the cell. Before performing the experiment, the membrane was immersed in demineralized water for 12 h. The whole experiment was performed in water at room temperature ($25 \pm 3\,^{\circ}$C) and the proton conductivity (σ) was calculated using Eq. (2) as follows:

$$\sigma = \frac{L}{R \times A} \qquad (2)$$

where σ is proton conductivity in (S cm^{-1}), L is the thickness of membrane in (cm), A is cross-sectional area of polymer membrane (cm^2) and R is the resistance (Ohm) [30].

3. Results and discussion

3.1. Water uptake

Type of polymer, solvent, counter ion, immovable ion and solvent uptake are the fundamental causes for better performance of ionic polymer metal composite (IPMC) membrane. The bending of membrane under an applied electric field is based on migration of solvated cation and water molecules towards cathode. The higher the water uptake, better will be the performance of IPMC films. It is observed from Fig. 1 that the water uptake capacity of nafion based IPMC film at room temperature ($25 \pm 3\,^{\circ}$C) increases with the increase of immersion time up to 10 h and after that saturation was found predominant. In case of kraton polymer film similar results were obtained with the water holding capacity 13 times greater than that of nafion membrane. The water holding capacity of nafion and kraton films at 45 °C and 10 h of immersion was found to be 16.20% and 242%, respectively. After increasing the temperature up to 65 °C the water holding capacity of nafion film decreases significantly. However, the water uptake capacity of kraton film increases at 65 °C up to 20 h of immersion time. The results showed that the kraton polymer membrane is having higher water uptake capacity than the nafion membrane. This may be because of the presence of more active thermally enlarged
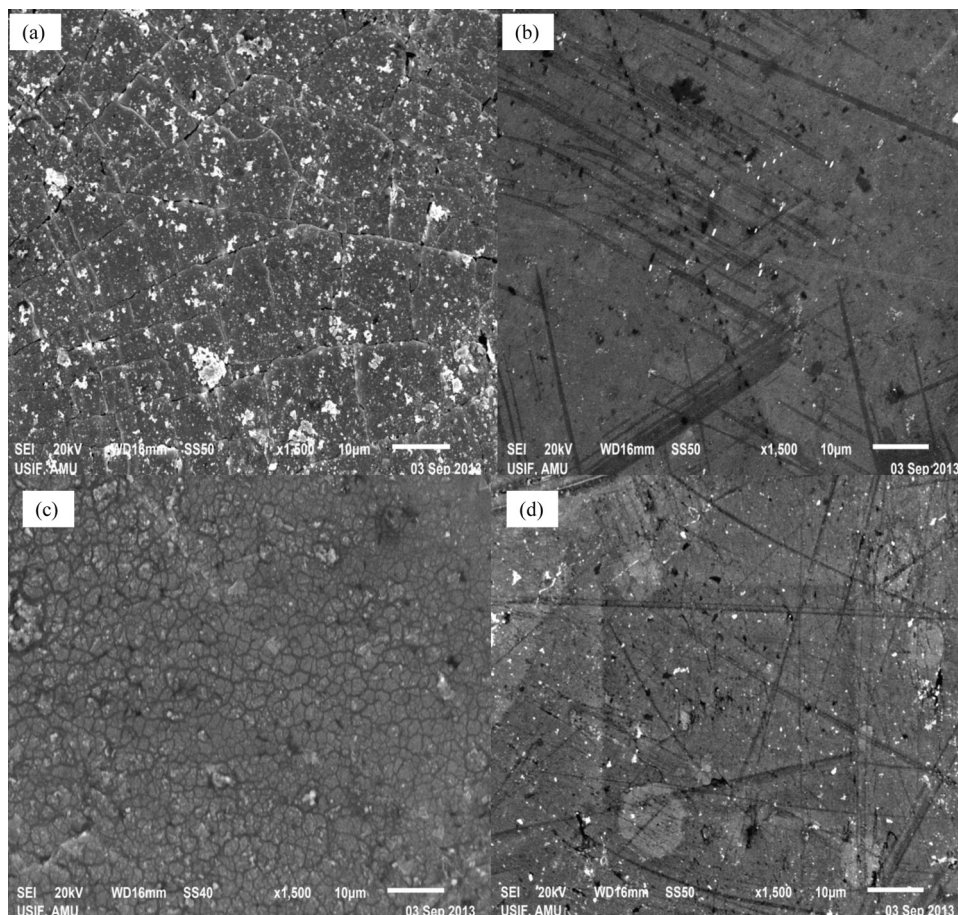
Fig. 2. SEM micrograph of IPMCs before actuation (a) kraton membrane (b) nafion membrane and after actuation (c) kraton membrane (d) nafion membrane.
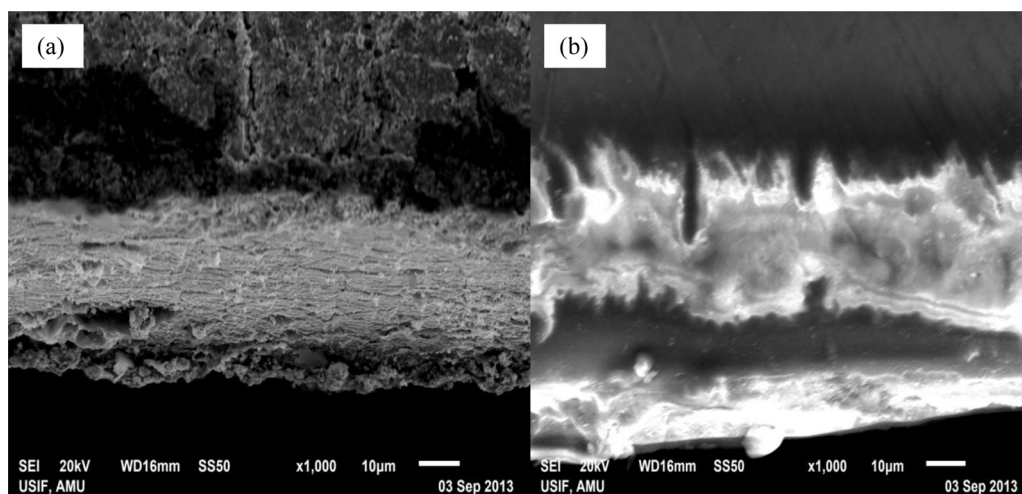
Fig. 3. Cross-sectional SEM micrographs of IPMCs of (a) kraton membrane (b) nafion membrane.

$-SO_3H$ sites at kraton film as compared to nafion film. Because of high water uptake of IPMCs films even at higher temperature, more hydrated cation can move through the membrane to actuate the IPMC films.
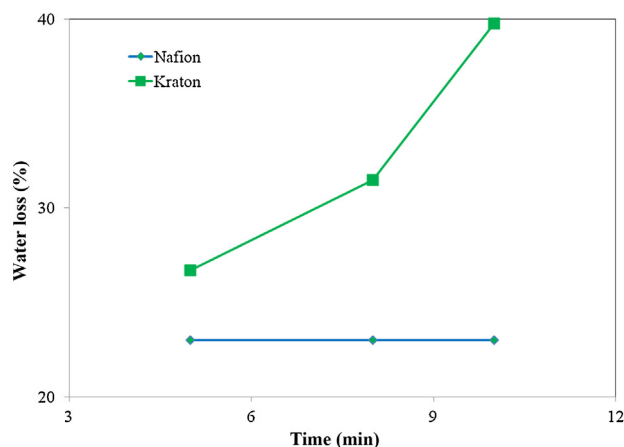

Fig. 4. Water loss from nafion and kraton based IPMC films on applying 3 V electrical voltage at 5 min, 8 min and 10 min.
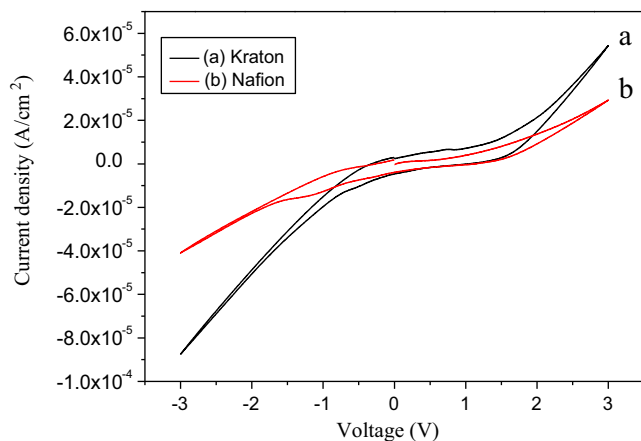
3.2. Ion-exchange capacity and proton conductivity studies

The ion exchange capacities and proton conductivities of kraton and nafion films were found to be 1.9 meq g^{-1}, 17.15 S cm^{-1} and 0.75 meq g^{-1}, 4.026 S cm^{-1}, respectively (Table 1). The high ion exchange capacity and proton conductivity of kraton polymer film will enable higher water uptake favoring the quick movement of more hydrated cations towards cathode creating a pressure towards anode, resulting in larger and faster actuation than the nafion film.

3.3. Scanning electron microscope (SEM) study

The performance and bending behavior of IPMC is directly related to migration of metal cation and electroosmosis of inner solution. The morphology of IPMC film plays an important role in the transport of ion and therefore in the conductivity behavior [31]. Fig. 2(a–d) showed the SEM images of the surface morphologies of kraton and nafion films before and after applying electrical voltage of 3 V during bending of the films.

Fig. 2(a) and (b) showed that a smooth Pt electrode surface with well interconnected large domains leaving behind negligible space at joints can be seen easily in the SEM micrographs of fresh nafion films, while spaces at joints are predominant on kraton films because of the presence of scattered $-SO_3H$ groups.

The electrode surfaces of nafion and kraton IPMCs films after performing the actuation test are damaged to some extent. Nafion

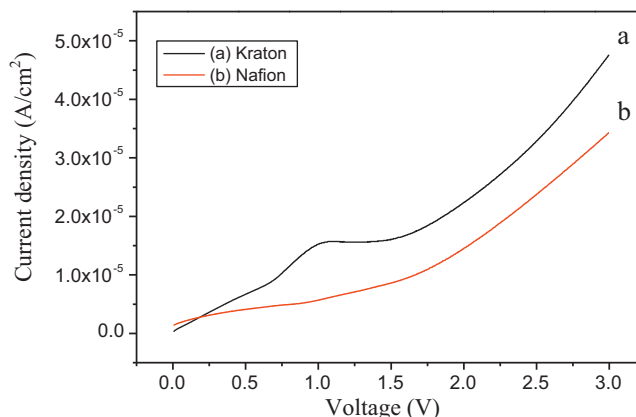**Fig. 5.** Cyclic voltammetric curves of nafion film and kraton membrane as a function of voltage obtained at ±3 V (triangle) with a scan rate of 100 mV s^{-1}.

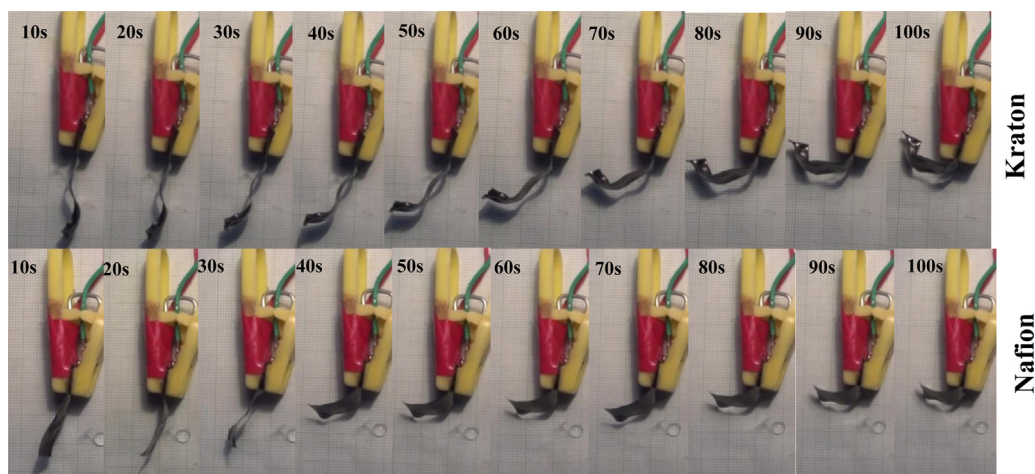**Fig. 6.** LSV of nafion membrane and kraton films IPMCs at 3 V.

Fig. 7. Displacement images of kraton film and nafion membrane at varying times under 3 V dc.

film showed few small interconnected and slightly rough domains with some ruptures while notable spaces at joints can be seen in the kraton micrograph because of the presence of scattered —SO$_3$H groups Fig. 2(c) and (d).

SEM micrographs shown in Fig. 3(a) and (b) depicts the cross-sectional images of the fresh kraton and nafion films. The Pt particles penetration in the IPMC membranes can be easily seen. It is also observed the denser aggregation of Pt particles in kraton membrane is leading to the granular damming which provides a good protection against water loss. This is because of the fact that the dense aggregation hinders in the path of flow of water molecules.

3.4. Water loss from polymer membrane

One of the important reasons for the short lifetime of IPMC film is the water loss from membrane and damage of electrode layer. The mechanisms behind loss of inner solvent include (a) evaporation of water, (b) leakage from damaged or porous surface and (c) electrolysis. Water loss of preweighed membrane was determined by applying an electric voltage of 3 V at different interval of time, i.e., 5 min, 8 min and 10 min. The results are given in Fig. 4. The results showed the water loss for nafion and kraton membranes was found to be 23% and 39%, respectively, after applying an electric voltage of 3 V for a maximum time of 10 min (Fig. 4). The water loss of kraton membrane is lower than that of nafion with respect to the water holding capacity of kraton membrane (308.69%) and nafion film (16.20%) (Fig. 1). The water loss may be because of the water leakage from damaged surface and/or electrolysis.

3.5. Electrical property

The electrical properties of IPMC membranes were explored by employing potentiostatic cyclic voltammetry. The performance of the Pt electrodes of nafion and kraton polymer membranes was confirmed by current–voltage hysteresis curves recorded under a ±3 V voltage with 100 mV/s scan rate as shown in Fig. 5. The movement of the hydrated ions because of the applied electrical voltage, with decomposition profile of water due to electrolysis reflects the shape of *I–V* hysteresis curves. It was observed the slope of the *I–V* curve for kraton membrane is significantly higher than that of the nafion membrane suggesting the fast movement of hydrated cations and slow evaporation of water in kraton membrane. It was also observed the current density of kraton membrane is significantly higher than that of nafion membrane as shown in Fig. 6.

This study proves the better actuation performance of kraton membrane.

The actuation performance of kraton and nafion membranes was evaluated by measuring the tip displacement under an electric potential of 3 V. The tip movement between two adjacent positions was measured horizontally, vertically, and diagonally based on the curvature generated from the initial position to last point of displacement. The results are shown in Fig. 7. The displacement results showed that within ca. 100 s, a displacement of ca. 40 mm was achieved for kraton membrane while nafion membrane showed a displacement of ca. 23 mm within ca. 100 s. The tip displacement from the original position to the displaced position can be clearly seen in Fig. 7. However, the back relaxation for the both of the membranes was found to be slow and is achieved within ca. 200 s for kraton membrane and ca. 320 s for nafion film.

4. Conclusion

The IPMCs membranes of kraton and nafion membranes were prepared by electroless plating method to examine their actuation performance. Both of the membranes were characterized for their water holding capacity and it was found that the kraton membrane has higher water uptake than that of nafion. Electrical properties carried out by the cyclic voltammetry also confirmed the better actuation performance of kraton membrane. The tip displacement study also revealed fast actuation of kraton membrane.

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