



# Evidence of counterion migration in ionic polymer actuators via investigation of electromechanical performance



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## ABSTRACT

Functional ionomeric polymer membranes are the backbone of a wide range of ionic devices; the mobility of ions through the ionomeric membrane is the principle of operation of these devices. Drift and diffusion of ions through ionomeric membranes strongly depend on the ionic properties of host membrane, as well as the physical and chemical properties of the ions. It is well-established that cations and anions provided via a dopant (e.g. electrolyte or ionic liquid) are mobilized under stimulation. However, in this study, we report that in addition to ions sourced by the dopant, counterions of the ionomeric membrane are also mobilized when stimulated. In particular, we have investigated the electromechanical response of ionic electroactive polymer actuators consisting of Nafion ionomeric membranes with different counterions and have demonstrated that those with cation counterions of larger Van der Waals volume exhibit stronger actuation due to motion of the larger cation counterions compared to actuators consisting of Nafion with counterion of smaller Van der Waals volumes.

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## 1. Introduction

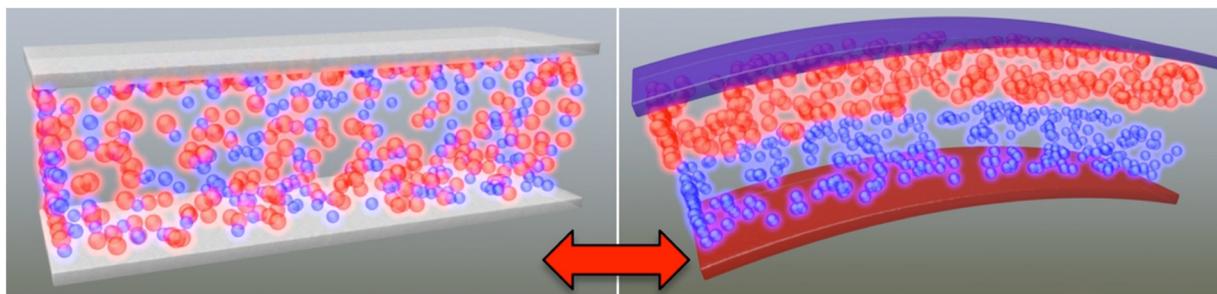
Ionic properties and ion permeability of ionomeric membranes, especially those of Nafion, have been the subject of extensive and continuous studies in the past several years [1–4]. The significance of such studies is mainly due to the increasing importance and application of ionomeric membranes in ionic/electronic devices for energy generation and storage applications. The functionality of ionic devices relies on mobility of ions through the ionomeric membrane. Ion diffusion and/or drift through ion permeable polymer membranes is the most essential requirement for operation of ionic devices; such as lithium-ion polymer batteries, fuel cells, super capacitors and ionic electroactive polymer sensors and actuators, to name a few examples [5–12]. For instance, diffusion of protons through a proton-exchange membrane is the principle of operation of hydrogen fuel cells [13], and charging of secondary cell metal-ion polymer batteries (used in most smartphones and tablets) is solely based on the ion drift through a polymer electrolyte membrane when an external electric field is applied [14]. Better understanding

of ion mobility through ionomeric membranes will provide means for development of electric/ionic devices with higher performance and efficiency.

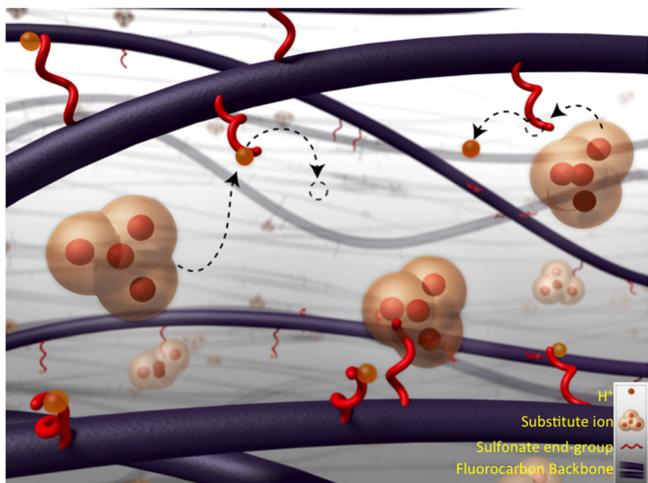
Although ion mobility, both diffusion and drift, is well utilized in commercial devices, we still lack a complete understanding of this phenomenon. It is not yet clear to the scientific community the detailed process of how ions move through the ionomeric membranes and how this process can be manipulated. The general understanding is that Nafion is a proton-exchange membrane; thus, H<sup>+</sup> can easily diffuse through it. Diffusion of H<sup>+</sup> through Nafion is well studied and applied in many conceptual applications such as fuel cells [15]. There is no doubt about H<sup>+</sup> permeability of Nafion; however, when subjected to an electrical field Nafion is also permeable to drift of other ions [16–21]. Our prior work on ionic electroactive polymer (IEAP) actuators confirmed that, when doped with ionic liquid, Nafion is permeable to both cations and anions of the ionic liquid; and, the electromechanical response of IEAP actuators is directly proportional to concentration of the ions from dopant [22,23]. The functionality of IEAP actuators is solely the result of motion of ions through the ionomeric membrane. The common understanding is that cations and anions provided by the doping of the ionomeric membrane with electrolyte are responsible for the electromechanical response of IEAP actuators. Upon application of an electric field, cations and anions are mobilized

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**Fig. 1.** Schematics of uncharged and charged doped 3-layer ionic electroactive polymer actuator. Red and blue spheres illustrate cations and anions, respectively. (Not to scale). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)



**Fig. 2.** Schematic presentation of ion-exchange process in Nafion. Proton counterions are substitute by other cations.

and move toward electrodes of opposite charge. Since cations and anions have different Van der Waals volumes, their accumulation at the cathode and anode results in a volume imbalance in the system; and thus, a mechanical deformation [24]; this phenomena is schematically presented in Fig. 1.

In this study we have altered the functionality of ionomeric membranes by exchanging the proton counterion of Nafion with larger cations; and have utilized the electromechanical response of IEAP actuators, consisting of Nafion with different counterions and dopants, as a means to study the mobility of ions through Nafion ionomeric membranes and, more specifically, investigate mobility of counterions of Nafion. Ion-exchange process is schematically demonstrated in Fig. 2. This work contributes to the knowledge of electric and ionic properties of ionic functional materials and their applications in electric and ionic devices such as sensors, actuators, fuel cells and metal-ion polymer batteries.

## 2. Experimental

### 2.1. Materials

Commercially-available Nafion membrane of 90  $\mu\text{m}$  thickness (Ion Power, Inc.) was used as the base ionomeric membrane. 1-Ethyl-3-methylimidazolium trifluoromethanesulfonate (EMI-Tf, molecular formula:  $\text{C}_7\text{H}_{11}\text{F}_3\text{N}_2\text{O}_3\text{S}$ ), triethylsulfonium bis(trifluoromethylsulfonyl)imide (TES-TFSI, molecular formula:  $\text{C}_8\text{H}_{15}\text{F}_6\text{NO}_4\text{S}_3$ ) and 1-butyl-1-methylpyrrolidinium bis(trifluoromethylsulfonyl)imide (BMP-TFSI, molecular formula:  $\text{C}_{11}\text{H}_{20}\text{F}_6\text{N}_2\text{O}_4\text{S}_2$ ) ionic liquids, and 1-ethyl-3-methylimidazolium chloride (EMI-Cl), zinc chloride and sodium chloride salts were

purchased from Sigma Aldrich and used without further modification. Transferable 24K gold leaves of 50 nm thickness were purchased from L.A. Gold Leaf and cut to desired size before using.

### 2.2. Methods

#### 2.2.1. Ion-exchange

Salt solutions were prepared at 0.5 M concentration by dissolving the proper amount of the desired salt in deionized water. The solution was then stirred overnight. Ionic membranes of the desired size ( $2.5 \times 12 \text{ cm}^2$ ) were cut out of a sheet of 90  $\mu\text{m}$  thick Nafion and boiled in diluted (1 M) sulfuric acid solution at 100  $^\circ\text{C}$  for 120 min. Water was added frequently to keep the volume of the mixture constant and to compensate for the evaporated water. The samples were then boiled in deionized water at 100  $^\circ\text{C}$  for 120 min, then dried using a wipe and cut into smaller pieces ( $2.5 \times 6 \text{ cm}^2$ ). Cut samples were then placed in ample amount of saturated salt solution in container with tightened caps, and heated to 80  $^\circ\text{C}$  for two days. The temperature was then reduced to 60  $^\circ\text{C}$  for another eight days to assure ion-exchange between the Nafion and salt solution. Considering high sensitivity of Nafion-ionic liquid systems to humidity [25,26], samples were then placed under vacuum ( $\sim -100 \text{ kPa}$ ) and heated to 115  $^\circ\text{C}$  for three days to dehydrate and were kept in desiccator or used immediately.

#### 2.2.2. Doping and assembly

Samples were then cut into smaller pieces ( $2.5 \times 2.5 \text{ cm}^2$ ), weighed and soaked in the desired ionic liquid to uptake  $\sim 40 \text{ wt}\%$  of their dry weight. Eq. (1) was used to calculate the electrolyte uptake, where  $W_e(\%)$  is the weight-percent of the electrolyte; and,  $W_d$  and  $W_f$  are the weights of dry and doped samples, respectively.

$$W_e(\%) = \frac{W_f - W_d}{W_d} \times 100 \quad (1)$$

Gold leaves were hot-pressed at 95  $^\circ\text{C}$ , under 4500 N for 25 s on both sides of the ionic liquid-doped samples to fabricate ionic electroactive actuators.

#### 2.2.3. Electromechanical characterization

Actuators were cut into approximately  $1.5 \times 15 \text{ mm}^2$  pieces and tested under application of a 4V applied potential. Electromechanical response of the actuators was monitored and recorded using a charge-coupled device (CCD) video camera, mounted to an in-house fabricated micro-probe station, at 30 frames per second. Individual frames were then analyzed to measure the radius of curvature ( $r$ ) as a function of time and to calculate curvature ( $Q$ ) from Eq. (2).

$$Q(t) = \frac{1}{r(t)} \quad (2)$$

For actuators with small tip displacement, strain can be calculated from free length, thickness and tip displacement of the

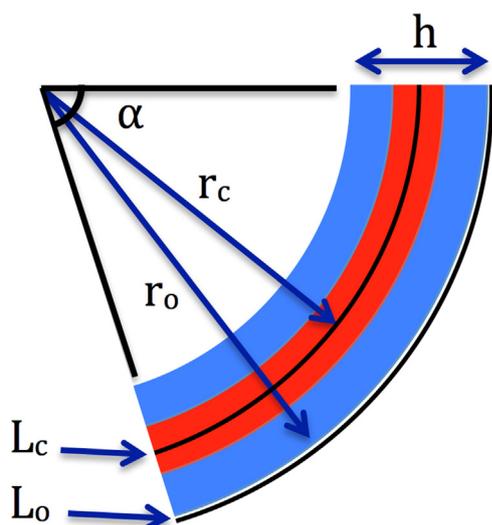


Fig. 3. Schematic representation of ionic electroactive polymer actuator with geometrical components used in calculation of strain. (Not to scale).

actuator [27]; however, for actuators with more extensive bending, the radius of curvature must be taken into account. Strain ( $\varepsilon\%$ ) was then calculated based on the thickness ( $h$ ) and radius of curvature of each actuator, using Eq. (7), which is derived from the ratio of the change in the free length of actuator between center and surface of the actuator, to the initial free length. Schematic presented in Fig. 3 and following calculations demonstrate deriving of Eq. (7); where  $L_c$  is actuator's length at the center (which is equal to actuator's free length),  $L_o$  is actuator's length at the expanded surface,  $r_c$  and  $r_o$  are radius to the center and expanded surface of actuator, respectively; and,  $\alpha$  is the angle between the mounted and free end of the actuator.

$$L_c = \alpha r_c \quad (3)$$

$$L_o = \alpha r_o = \alpha(r_c + h/2) \quad (4)$$

$$\varepsilon\% = \frac{L_o - L_c}{L_c} \times 100 \quad (5)$$

substituting Eqs. (3) and (4) in Eq. (5), we will get:

$$\varepsilon\% = \frac{\alpha(r_c + h/2) - \alpha r_c}{\alpha r_c} \times 100 \quad (6)$$

simplifying Eq. (6) and including time dependency, we will have:

$$\varepsilon\%(t) = \frac{h}{2r_c(t)} \times 100 \quad (7)$$

To obtain strain data, each set of experiments was repeated at least three times to confirm reproducibility. Where appropriate, data were averaged; otherwise the most common behavior was used.

### 3. Results and discussion

#### 3.1. Van der Waals radius of counterions

Four sets of samples were fabricated consisting of Nafion films with different counterions, doped with EMI-Tf ionic liquid. Nafion in its proton form ( $H^+$  counterion), and Nafion ion-exchanged with  $Zn^{2+}$ ,  $Na^+$  and  $EMI^+$  were studied for the cationic portion of their electromechanical response. Table 1 summarizes Van der Waals properties of the investigated counterions.

The Van der Waals volumes of the atomic ions were calculated directly from the Van der Waals radius of each atomic ion; the Van der Waals volume of the EMI molecular ion was calculated based on

Table 1

Van der Waals properties of the counterions of Nafion membrane and anions and cations of ionic liquid dopants.

Counter-ion	Van der Waals radius (pm)	Van der Waals volume ( $\text{\AA}^3$ )
$H^+$	120.0	7.2
$Zn^{2+}$	139.0	11.2
$Na^+$	227.0	48.9
EMI <sup>+</sup>	294.6	107.1
TES <sup>+</sup>	316.0	132.2
BMP <sup>+</sup>	339.7	164.2
Tf <sup>-</sup>	275.5	87.6
TFSI <sup>-</sup>	337.8	161.4

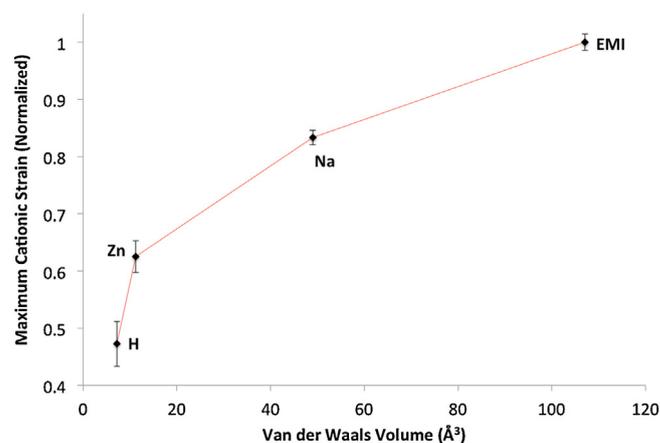


Fig. 4. Magnitude (arbitrary units) of maximum cationic strain of IEAP actuators consisting of Nafion with different counterions as a function of Van der Waals volume of counterions. Actuators consisting of Nafion with larger counterions exhibit enhanced cationic strain.

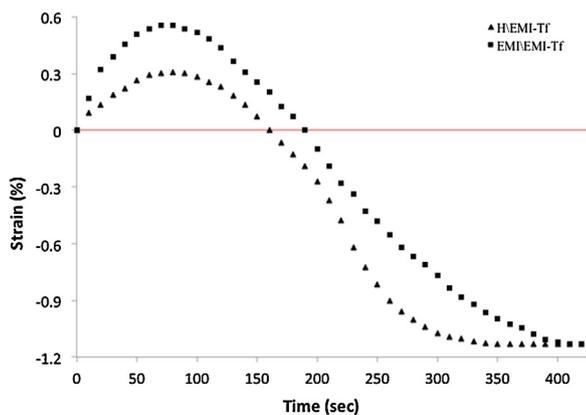
the number of bonds, aromatic and nonaromatic rings, as described by Zhao et al. [28].

#### 3.1.1. Influence of counterions on cationic electromechanical response

Presented in Fig. 4 is the magnitude of the electromechanical response of the actuators as a function of the Van der Waals volume of the counterions. The actuator consisting of Nafion with  $H^+$  counterion exhibits significantly smaller cationic strain compared to the actuators consisting of Nafion with larger counterions. The increase in the magnitude of cationic strain is more significant between atomic counterions and the molecular counterion. This is most probably due to the complex 3-dimensional structure of EMI<sup>+</sup> multi-atom ion compared to the simpler spherical structure of single-atom ions. Interestingly, actuators containing different counterions reached the steady state at approximately the same time ( $93 \pm 2$  s), implying that the ions move at approximately the same speed and that the ion mobility is drift dominated rather than diffusion dominated. Back relaxation was not observed in any one of the systems.

#### 3.2. Electromechanical response as a function of dopant

We further investigated the influence of the counterions on electromechanical response by comparing the full (cationic and anionic) electromechanical response of IEAP actuators consisting of Nafion with  $H^+$  and EMI<sup>+</sup> counter-ions (the two extreme cases in this study), doped with three different types of ionic liquids. The samples investigated in this section are named by the following format: (Counterion\Cation-Anion) where cation and anion are those from the ionic liquid.

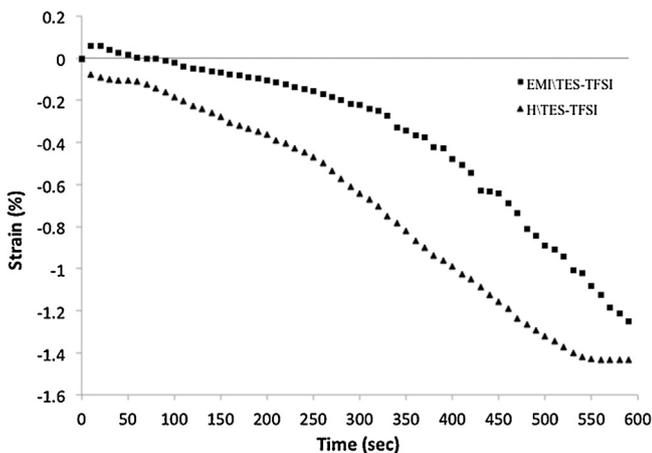


**Fig. 5.** Electromechanical responses of IEAP actuators doped with EMI-Tf ionic liquid consisting of Nafion membranes with  $H^+$  and  $EMI^+$  counterions. Cationic strain is enhanced with larger counterion.

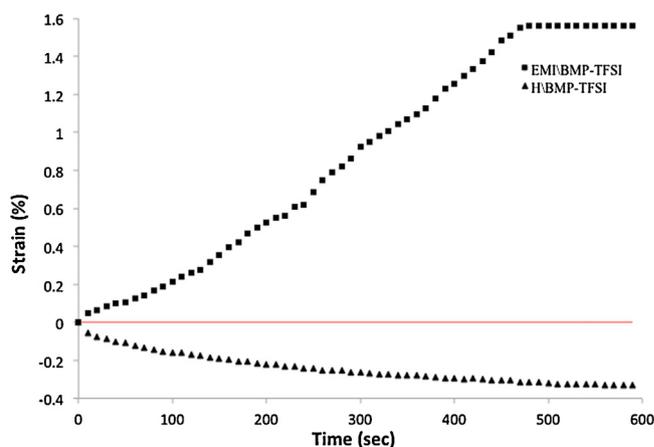
As we described previously [22], the cationic and anionic strains are the result of the out-of-phase motion of cations and anions in Nafion. For instance, in the case of IEAP actuators doped with EMI-Tf ionic liquid, the drift velocity of  $EMI^+$  cations is faster than that of  $Tf^-$  anions. As a result, when the voltage is applied, a bending toward the anode is observed first, that is due to the fast accumulation of cations at the cathode, followed by a dominating anionic motion that is due to accumulation of anions at the anode. In the case of EMI-Tf ionic liquid, both cationic and anionic strains are significant and distinguishable, especially over long path-lengths (i.e. thick Nafion); and, anionic motion is dominant due to the higher effectiveness of anions (or anionic clusters) [29] in generating strain compare to cations. This behavior may be different or even reversed, depending on the physical and electrochemical properties of the electrolyte used in doping of the Nafion.

As presented in Fig. 5, IEAP actuators consisting of Nafion with  $H^+$  and  $EMI^+$  counterions, doped with EMI-Tf ionic liquid exhibit both cationic (in the plots showed as positive strain (%)) and anionic (in the plots showed as negative strain (%)) strain, with the anionic strain ultimately dominating the response. An interesting observation is that when the  $H^+$  counterions are exchanged with  $EMI^+$ , the entire response curve of the IEAP actuator is almost uniformly shifted toward cationic strain, suggesting contribution of the  $EMI^+$  counterions toward cationic strain.

Similar behavior was observed when other ionic liquids were used as dopants. As shown in Figs. 6 and 7, IEAP actuators



**Fig. 6.** Electromechanical responses of IEAP actuators doped with TES-TFSI ionic liquid consisting of Nafion membranes with  $H^+$  and  $EMI^+$  counterions. The response is shifted toward cationic strain with larger counterion.



**Fig. 7.** Electromechanical responses of IEAP actuators doped with BMP-TFSI ionic liquid consisting of Nafion membranes with  $H^+$  and  $EMI^+$  counterions. The electromechanical response is completely reversed from fully anionic to fully cationic with larger counterion.

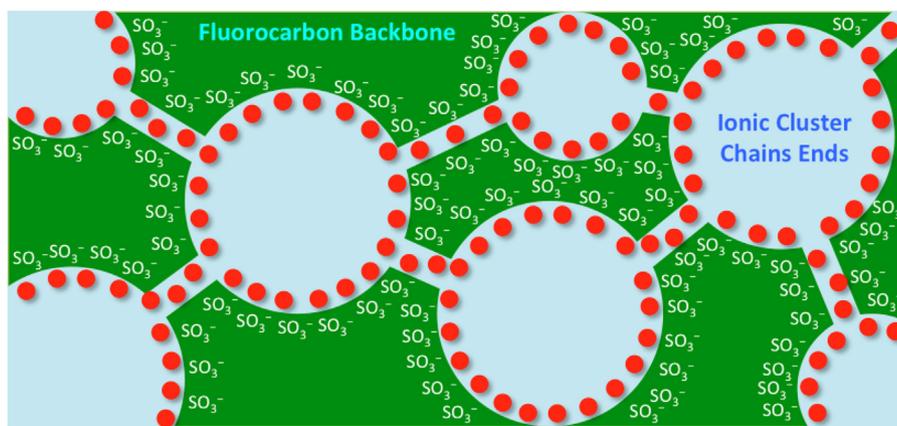
consisting of TES-TFSI and BMP-TFSI in Nafion with  $H^+$  counterion have dominating anionic strain, which prevents observation of any cationic strain even at the beginning of actuation, suggesting that unlike  $Tf^-$  anions in EMI-Tf,  $TFSI^-$  anions in TES-TFSI and BMP-TFSI are quickly mobilized upon application of the potential difference. Hence,  $TES^+$  and  $BMP^+$  cations are not allowed the time required to generate a temporary dominating cationic strain, or both types of ions are mobilized simultaneously yet the effectiveness of the  $TFSI^-$  is dominant.

In both cases, when  $H^+$  are exchanged with  $EMI^+$  ions, the overall response is shifted toward cationic strain. In the case of the EMI\TES-TFSI IEAP actuator, a small cationic motion is observed in the first tens of seconds, yet quickly canceled by the anionic strain. However, the overall electromechanical response exhibits a shift toward cationic strain, again suggesting the contribution of the  $EMI^+$  counterions to the net strain. In the case of the EMI\BMP-TFSI IEAP actuator the contribution of  $EMI^+$  is more significant. The electromechanical response is fully reversed from an anionic-only to a cationic-only strain, suggesting significant influence and contribution of  $EMI^+$  toward the electromechanical response of the IEAP actuators or, in more general terms, influence on the ionic properties of Nafion ionomeric membranes.

### 3.3. Discussion

Our experiments and observations suggest that the counterions of the ionomeric membrane, Nafion in this case, are mobilized upon exposure to an external electric field and thus have significant influence on the ionic response of the membrane and do contribute to the electromechanical response of the IEAP actuators. Considering the standard cluster-network model to explain the morphology of Nafion (see Fig. 8), two possible hypotheses may be developed to explain the contribution of counterions to ion permeability of Nafion:

- A) Counterions with larger Van der Waals volume expand the narrow channels between the interconnected clusters. These narrow channels in Nafion with  $H^+$  counterions have an approximate diameter of  $10 \text{ \AA}$ , which is considerably larger than the Van der Waals diameter of  $H^+$  ( $2.4 \text{ \AA}$ , see Table 1). When larger cations (e.g.  $EMI^+$ ) are introduced to the network, ionic interactions between the cations and sulfonate end-groups force the cations into the channels, and to compensate for repulsion between neighboring cations the channels expand; in presence



**Fig. 8.** Schematic cluster-network morphology of Nafion. It is anticipated that counterions (red dots) are mobilized when exposed to an external electric field. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

of an electric field, the expanded channels allow motion of cations ( $\text{EMI}^+$  in this case) through the network. These expanded channels provide means for higher mobility of the ions throughout the Nafion membrane.

B) Counterions in Nafion are always mobile and contribute toward cationic strain; yet, due to the small Van der Waals volume of  $\text{H}^+$ , this contribution is less significant. When a larger cation is introduced, the contribution toward cationic strain is more significant and thus observable.

Although it is very difficult to explain and construct an accurate model for mobility of ions through Nafion, our experimental results and observations suggest that while both hypotheses above may be, to some extent, correct, hypothesis B explains the behavior of IEAP actuators more consistently and is responsible for the observation of an enhanced cationic strain in the presence of larger counterions.

When an electric field is applied, it breaks the electrostatic bonds between the counterions and sulfonate end-groups, mobilizing the counterions. Along with the cations from the doping electrolyte, the mobilized counterions (red dots in Fig. 8) are attracted to the cathode while the anions are attracted to the anode. Depending on the Van der Waals volume of the counterions, their contribution toward cationic strain varies. In the case of  $\text{H}^+$  counterions, the contribution is minimal due to the small Van der Waals volume, while it is more significant in case of larger counterions such as  $\text{EMI}^+$ .

#### 4. Conclusion

We investigated ion mobility through Nafion ionomeric membrane via the electromechanical response of IEAP actuators fabricated using Nafion with a variety of counterions and ionic liquid dopants. It was observed that exchange of the  $\text{H}^+$  counterion of Nafion with a cation of larger Van der Waals volume results in the generation of enhanced cationic strain. Experiments were performed with four types of counterions and three different types of ionic liquids as dopants, and in all cases the enhancement was observed. The results of this study suggest that in the presence of an electric field, in addition to cations and anions from the dopant that drift through the Nafion, the counterions of Nafion are also mobilized and drift through the interconnected channels of the polymeric backbone structure and accumulate at the cathode to contribute toward cationic strain. Further investigations are expected to contribute toward more efficient actuators, sensors, metal-ion polymer batteries, and other ionic devices.

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## Biographies



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