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PEDOT based Conducting IPN Actuators.

Effects of electrolyte on actuation

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Abstract

Actuators from conducting IPNs architecture are described. The electroactive materials are based on several PEO/elastomer IPNs as solid polymer electrolytes in which the conducting polymer Poly(3,4-ethylenedioxythiophene) (PEDOT) is gradually dispersed, i.e. its content decreases from the outside towards the centre of the film. Influence of the electrolyte on the actuation properties has been studied. Moreover, the effect of actuator thickness has been investigated. It is shown that the decrease of the thickness leads to fast response systems presenting large deformations at frequencies above 100 Hz.

Introduction

Electronic conducting polymers (ECPs) have attracted attention notably because of possible dimensional changes due to the counter-ion (and solvent molecule) expulsion/incorporation during oxidation or reduction processes. Providing an ionic conducting medium is available, ECPs can be used as the active component in actuators or artificial muscles and lead to interesting potential applications (robotics, prosthetics, microvalves, etc.). [1-5]. ECPs such as polyaniline, polythiophene and their derivatives have been studied; nevertheless polypyrrole (PPy) is the most widely studied ECP as active component in actuators [6]. Consequently many studies on actuators are based on self-standing PPy film or PPY film/ no volume change film (PPY/ NVC) [7] bilayer structure for which the PPy film is connecting as a working-electrode in a classical 3 electrochemical cell. The first actuators capable of a reversible bending movement in air were also made of PPy [1]. These actuators are constructed using two PPY film electrodes and a solid polymer electrolyte (SPE) film in a (PPy//SPE//PPy) sandwich configuration. These trilayered actuators opened the possibility of getting ECP based actuators working out of a liquid electrolyte. However a delamination process was reported that limits the actuator's lifetime [8,9]. To overcome the delamination process Spinks et al. used a porous membrane as the SPE. The membrane acts a reservoir for the ionic electrolyte and provides improved adhesion to the ECP layers that are deposited on the membrane. The adhesion improvement is attributed to the rough nature of the porous membrane.

PEDOT has received particular attention in recent years. PEDOT displays a high structural regularity due to the exclusive 2–5 linkage resulting from the presence of the 3,4-ethylenedioxy substituent. It has also been described as a chemically and thermally [10] stable polymer exhibiting a particularly high electrochemical stability [11-13] in the doped state. Moreover the donor

properties of alkylendioxy substituent as well as the rigid and planar 3,4-ethylenedioxythiophene (EDOT) conjugated rings lead to a low-band-gap polymer ($E_g \sim 1.6$ eV). Indeed, PEDOT can be easily oxidized [14-16] and it presents high visible contrasts upon electrochemical switching, being transparent light blue in its p-doped state and dark blue in its dedoped one [17-19]. As an ECP that undergoes dimensional changes resulting from redox processes, PEDOT is potentially attractive for the design of electrochemical actuators [20]. Nevertheless unlike PPy, PEDOT based actuators have not received particularly high attention. Only few groups are currently dealing with PEDOT based actuators [21,22]. Likewise, fundamental studies on actuation properties have been hardly carried out because PEDOT can not form a self-standing film. Several years ago an original polymer architecture was proposed by our laboratory by developing an actuator based both on Interpenetrating Polymer Networks and on PEDOT [22]. The linear electronic conducting polymer is then entangled within an interpenetrating polymer network (IPN) SPE matrix and its controlled inhomogeneous mass distribution mimics the sandwich like configuration. The IPN structure ensures a good compatibility between PEDOT and the SPE matrix and totally prevents delamination. Conducting IPN actuator is thus similar to a layered one but for the fact that it is a one-piece device instead of being a trilayered device. As for conventional PPy based actuators, the ion movements are principally responsible for the volume change that produces an actuation response. The extent and speed of actuation is influenced by the nature of ions that move during the redox reaction as well as the speed at which the ion move. The behavior of conducting IPN actuators is then sensitive to the electrolyte nature and to the SPE matrix characteristics. Their influences are described in the present paper.

Experimental

Materials

Polytetrahydrofuran (PTHF, Terathane® $M_w = 2900$ g mol⁻¹), polybutadiene – hydroxyl functionalized (HTPB, $M_w = 2800$ g.mol⁻¹), methoxy poly(ethylene glycol) methacrylate (PEGM475 $M_w = 475$ g mol⁻¹), poly(ethylene glycol) dimethacrylate (PEGDM750, $M_w = 750$ g mol⁻¹; dibutyltin dilaurate (DBTDL 95%), obtained from Aldrich were used without further purification. Nitrile butadiene rubber with 44 wt % acrylonitrile content (NBR, $M_w = 230\ 000$ g mol⁻¹, Perbunan 4456F) was provided by Lanxess. 3,4-ethylenedioxythiophene (EDOT) (Bayer) was distilled under reduced pressure at 90°C prior to use. Dichloromethane (VWR), toluene (VWR), methanol (VWR), lithium perchlorate (Aldrich), anhydrous iron III chloride (Acros) and Desmodur® N3300 (5.2×10^{-3} moles of NCO for 1 g of Desmodur®) (Bayer) were used without further purification. Dicyclohexylperoxidicarbonate (DCPD) (Groupe Arnaud) and 1-ethyl-3-methylimidazolium bis-(trifluoromethylsulfonyl)-imide (EMITFSI) (Solvionic) was used as received.

Synthesis of IPNs, conducting IPNs and actuators.

Solid Polymer Electrolytes (SPE) (PEO/PB, PEO/PTHF, PEO/NBR IPNs) have been synthesized according to procedures described elsewhere [23-25]. Conducting IPNs have been synthesized by chemical oxidative polymerization of EDOT according to procedure described elsewhere [22,24,26]. The edges of the conducting IPNs were cut off in order to avoid short-circuit between the two faces of the device. The conducting IPNs were immersed in electrolyte (salt/solvent or neat EMITFSI ionic liquid) for a swelling step. In the case of salt/solvent electrolytes, the actuators are tested immediately after the desired amount of electrolyte has been incorporated. In the case of EMITFSI ionic liquid, the actuator is kept in air without further precaution.

Actuator characterizations.

The conducting IPNs were maintained horizontal with steel clamps in order to ensure electrical contact with the VMP Bio-logic (Science Instrument) potentiostat. Electrochemical studies were carried out by connecting the counter electrode and the reference electrode together on one side of

the actuator and by connecting the working electrode to the other side. The displacement response was stimulated by applying a square or triangle wave potential from ± 2 to ± 5 V and recorded with a numeric camera and a laser displacement sensor (ILD 1401-5, Micro-Epsilon) fitted to a computer. The actuator position was characterized by calculating the curvature ρ (cm^{-1}) as described elsewhere [27]. The strain ε of the device was calculated using equation (1):

$$\varepsilon = \frac{Dw}{(D^2 + L^2)} \times 100 \quad (1)$$

where D is the displacement from the unactivated position to the activated position, L the length of the sample and w the thickness of the sample.

Scanning electron microscopy and energy dispersion spectroscopy (EDS).

Scanning electron microscopy (SEM) was performed using a Carl Zeiss AG-ULTRA 55 by GEMINI® with a field emission gun at 2–10 kV. The SEM is equipped with an energy dispersion spectrometer allowing a sulfur mapping on dry conducting IPN (HV = 10 kV).

Results and Discussion

Conducting IPN synthesis

The preparation of PEO based IPNs is among the most efficient approaches in Solid Polymer Electrolyte (SPE) design suppressing PEO crystallinity at ambient temperature and ensuring polymer flexibility. Moreover in the chosen PEDOT-based materials the presence of ethylene oxide units in both the PEO network and the ethylenedioxy function of PEDOT will ensure a compatibilizing effect [22]. In order to further improve the ionic conductivity, short PEO side chains can be introduced in the structure of the IPNs as dangling chains leading to the design of a branched PEO network [28]. The flexibility of the chains is thus expected to be less restricted than in an unbranched network and the amount of volume available for the migration of ions would be much higher. In the present paper the PEO based IPN SPE matrix is composed either of a polybutadiene (PB) or Nitrile Butadiene Rubber (NBR) or polytetrahydrofuran (PTHF) partner according to the nature of the desired application. The synthesis and the characterization of these PEO based IPN matrices have been described elsewhere. A series of PEO based IPNs were prepared, namely the 80/20 (w/w) PEO/PB, the 60/40 (w/w) PEO/NBR and the 50/50 (w/w) PEO/PTHF.

The conducting IPNs are obtained from the above described SPE. The PEO-based IPN film is swollen with EDOT which is subsequently polymerized to PEDOT. The EDOT-swollen IPNs are immersed in a 1.5 mol L^{-1} FeCl_3 aqueous solution for various lengths of time leading to the formation of PEDOT inside the IPN. The preferential PEDOT distribution as a thin layer towards the surface of the film has been evidenced from EDS. For polymerization duration of 18 h, the quantity in the central part of the film is low enough to ensure that no short-circuiting occurs. The general procedure for the synthesis of conducting IPN is described Figure 1.

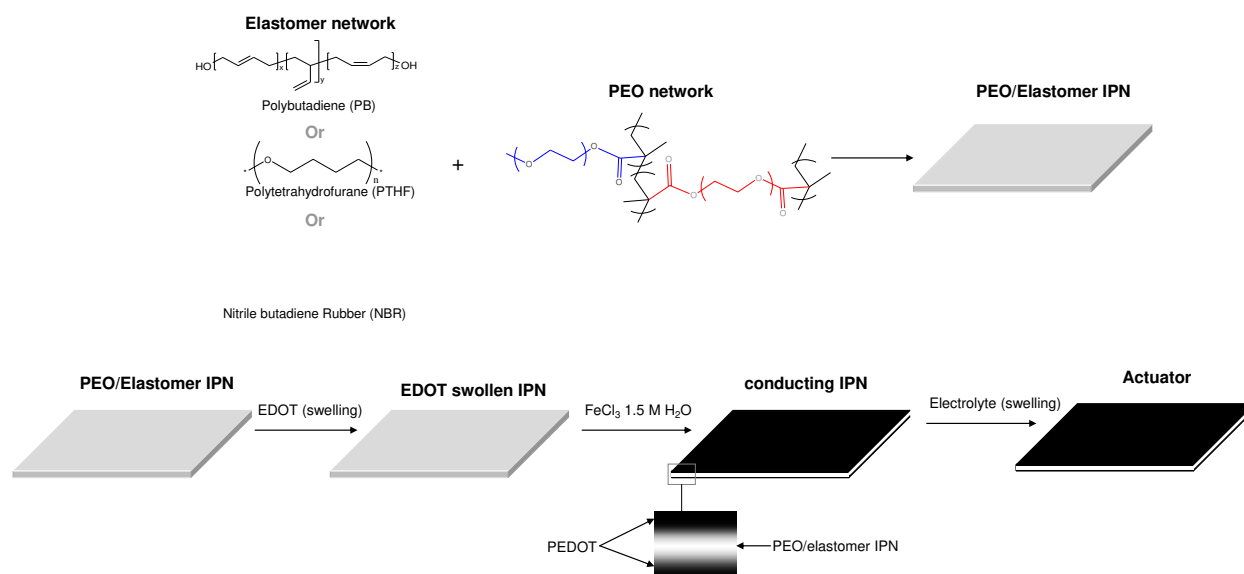


Figure 1 : Schematic procedure for the synthesis of conducting IPN actuators

Effects of the salt and solvent

A series of conducting IPNs were prepared according to the procedure described above. As synthesized via chemical oxidation of the monomer, PEDOT is present in the IPN in the charged conducting state. The FeClO_4^- dopant anion is fixed during the oxidative polymerization, but may be exchanged during the electrolyte swelling process. Therefore before actuation experiments, dry conducting IPNs were dipped in different salt/solvent combination solutions ($10^{-1} \text{ mol L}^{-1}$). The mass quantity of electrolyte in the conducting IPN was controlled by the time duration of immersion in the salt/solvent solution.

After salt uptake in $\text{LiClO}_4/\text{acetonitrile}$, a strip of conducting IPN ($10 \times 6 \times 0.25 \text{ mm}^3$) was subjected to a constant voltage of 2 V applied between the two sides of the actuator film. The actuator bends and reach a positive curvature value of 0.4 cm^{-1} in 60s (Figure 2-A). The actuation direction corresponds to an increase in volume at the anode and a decrease in volume at the cathode indicating that a positive charge on the PEDOT backbone is balanced by the incorporation of ClO_4^- . This mechanism with ClO_4^- incorporation /expulsion has been already described in PPy actuators [5,29] but it is shown here that a different behavior is achieved on PEDOT based actuators. Skaarup et al. [29] have found that for PPy actuators in $\text{LiClO}_4/\text{acetonitrile}$ electrolyte, ClO_4^- is a non-mobile specie. The mobile anion mechanism is further confirmed when the conducting IPN actuator is dipped in $\text{NBu}_4\text{ClO}_4 / \text{acetonitrile}$ electrolyte. Indeed NBu_4^+ cation is much more bulky than Li^+ and should be immobilized. As show in Figure 2, the actuator bends in the same direction with similar capacity which confirms that the mechanism is governed by an anion exchange. In the range of 5-15wt% salt, the electrolyte content has only a weak influence on the rate and strain when a voltage of 2V is applied.

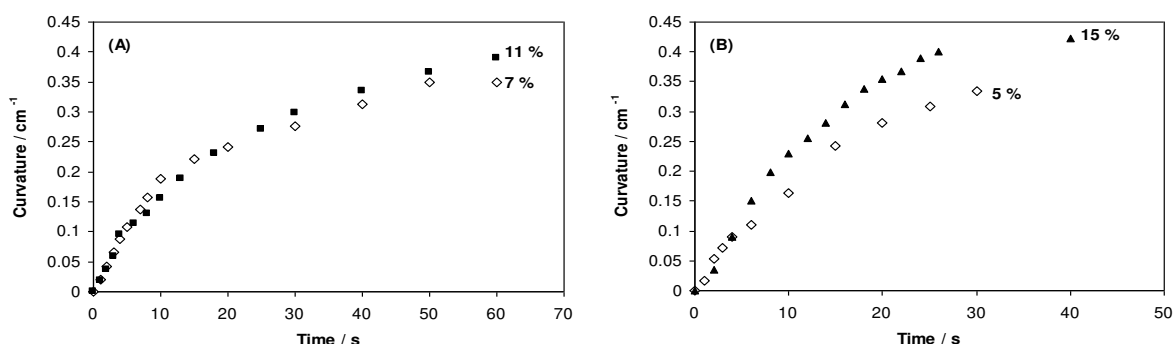


Figure 2: Curvature as a function of time for a PEO/PB/PEDOT conducting IPN actuator under 2 V. electrolyte: (A) $\text{LiClO}_4/\text{acetonitrile}$ and (B) $\text{tBuNClO}_4/\text{acetonitrile}$

Different solvents can be used for the electrolyte and could have an impact on the actuation behavior since interactions between the SPE, the PEDOT and the solvent can be modified. Conducting IPN actuators are now swollen in $\text{LiClO}_4 / \text{H}_2\text{O}$ electrolyte. This system shows more complex behavior as illustrated in Figure 3. Depending on the amount of loaded LiClO_4 , significantly different results are obtained.

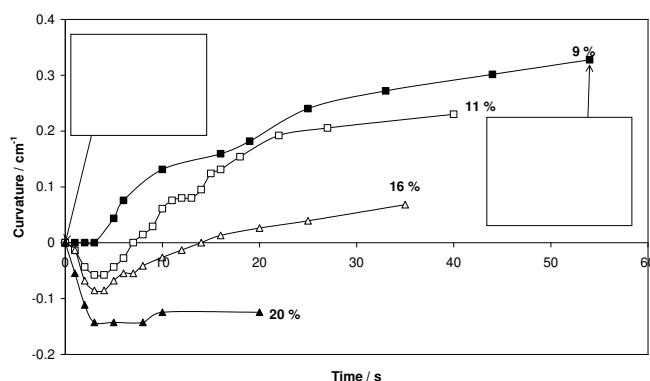


Figure 3 : Curvature as a function of the time for a PEO/PB/PEDOT conducting IPN actuator swollen by $\text{LiClO}_4/\text{H}_2\text{O}$ under 2 V. Amount of loaded LiClO_4 : (■) 9 %, (□) 11 %, (△) 16 %, (▲) 20 %.

The amount of LiClO_4 loaded in the actuator seems to have a major and unexpected role on the curvature. For a content of 9wt% LiClO_4 , the actuator remains motionless during the first 3 seconds while applying a voltage of +2V. Then, the actuator bends and reaches a positive curvature value of 0.35 cm^{-1} after 40s. Therefore, since PEDOT electrode volume expansion and contraction are observed during oxidation and reduction respectively, the actuation mechanism seems to be mainly governed by an anion movement. In the case of actuators containing 11 and 16wt% LiClO_4 , a particular behavior is observed while applying +2V. Indeed, during the first 3 seconds the devices bend in the opposite direction to the previous case and after this period, the bending direction is reversed. The curvature values at 30s decrease when the amount of LiClO_4 increases. Finally, for the sample containing 20wt% LiClO_4 , only the opposite movement is observed. The actuator bends in the first 3 seconds and hold a constant position (-0.12 cm^{-1}). Likewise, Li^+ cation is not immobile anymore and a competition mechanism seems to be involved. Indeed in the case of an electrolyte comprising two mobile ions, a competition mechanism could take place, leading to the successive deformation of the actuator in one direction then in the other. In the case of conducting IPN actuator based on PEDOT, the three behaviors, namely mechanism of anion or cation movement or both successively, seem to be observed as function of loaded LiClO_4 . All the above experiments show the complexity of the processes involved in conducting actuators. To our best knowledge such behavior as a function of salt concentration has not been described before.

Different solvents and salts have been tested as electrolyte. Finally the best actuation performances are obtained with the following electrolytes i.e. $\text{LiClO}_4 / \text{propylene carbonate (PC)}$ and EMITFSI. Conducting IPN is actuated in $\text{LiClO}_4 / \text{PC}$ and the optimum performances of 1.1 cm^{-1} curvature at a rate of $0.5 \text{ cm}^{-1}/\text{s}$ is achieved. This electrolyte allows the largest bending as shown in Figure 4 and the direction as well as the amplitude of the bending movement indicates that the mechanism is mainly governed by the anion movement. Likewise, PC can not only swell the PEO and the elastomer partners but has also the ability to diffuse in the PEDOT partner. Therefore PC electrolyte can enable plasticizing and softening all the partners of the conducting IPN which result in higher strain. However the main drawback of $\text{LiClO}_4 / \text{PC}$ electrolyte is the solvent evaporation. For instance conducting IPN actuator can be cycled over 3h before the bending deformation stops due to PC evaporation. After being immersed again for a few hours in $\text{LiClO}_4 / \text{PC}$ solution the

actuator fully recovers its working capacity [30]. In order to prevent the evaporation process ionic liquid (IL) has been used as electrolyte. Indeed ILs are non-volatile compounds and have been successfully used for electromechanical actuators [31-33]. In the presence of ethyl-3-methylimidazolium bis-(trifluoromethylsulfonyl)imide (EMITFSI) as IL, the actuator can be operated in open air over one month and be stored over a period of 6 months at least without any precaution and be operated again immediately. In the presence of EMITFSI, the actuation is dominated by cation movement [34] giving an actuation curvature of -0.4 cm^{-1} at quite low rate (Figure 4). Compared with $\text{LiClO}_4 / \text{PC}$ electrolyte, conducting IPN actuators are less efficient in the presence of EMITFSI, however it should point out that the major advantage obtained from EMITFSI is the extended cycling. For instance EMITFSI allows the conducting IPN actuator to be operated in air for one month without drying. In this configuration an actuator film has been subjected to 7×10^6 bending motions ($f = 10 \text{ Hz}$) without degradation and/or delamination [30].

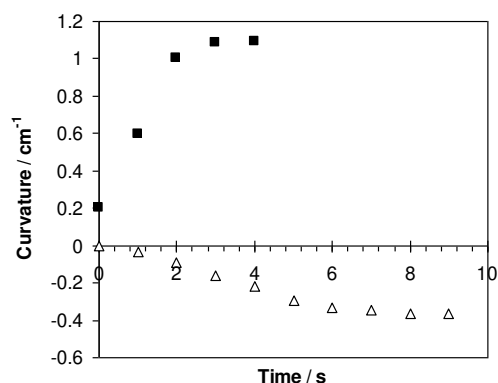


Figure 4: Curvature as a function of the time for a PEO/PB/PEDOT conducting IPN actuator swollen by (■) LiClO_4/PC or (Δ) EMITFSI.

Conducting Polymer actuators with fast response

In order to investigate the response speed of conducting IPN actuators, thin IPN based SPEs have been elaborated and have been used as starting material for the conducting IPN synthesis. In principle, ionic EAPs, such as conducting polymer actuators, present slow responses since they are based on ion motions inside polymer membrane. In the case of ECP actuators, it is commonly accepted that actuation mainly results from the redox process of the conducting polymer which is limited by ion diffusion. However, several examples of ionic EAPs presenting high actuation frequency have been described. For instance, Spinks et al [35] have described a fast-response actuator, based on polypyrrole, which displays a resonance peak as high as 90 Hz depending on the sample size. Recently Asaka et al [36] have reported a beam shaped actuator consisting of single-walled carbon nanotubes (SWNTs), ionic liquid, PVDF and polyaniline as an additive. Bending deformation was measured in the frequency range between 5 mHz and 100 Hz. Nevertheless the strains are low at high frequencies,

Since the actuation mechanism is dominated by mass transfer, the actuation is determined by the diffusion rates and distances [4,35]. Therefore, conducting polymer actuators with fast response can be obtained by decreasing the thickness of the SPE. Then, the previously described IPN SPEs have been synthesized as thin films. For instance 50/50 PEO/PTHF or 50/50 PEO/NBR IPNs present a final thickness of 10 and 30 μm respectively.

Thin actuators were prepared from PEO/elastomer and EDOT according to the procedure described elsewhere [22,24]. As shown in Figure 5, by the control of the polymerization condition (EDOT content, temperature, FeCl_3 concentration and time), trilayer configuration can still be obtained despite the use of very thin membranes.

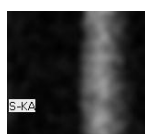


Figure 5: SEM images of the cross-section of the conducting IPNs: (A) PEO/NBR/PEDOT and (B) PEO/PTHF/PEDOT. Energy Dispersive Spectroscopy with sulfur atom mapping (light grey) of the conducting IPNs: (C) PEO/NBR/PEDOT and (D) PEO/PTHF/PEDOT.

Afterward PEO/PTHF/PEDOT or PEO/NBR/PEDOT conducting IPNs are swollen with EMITFSI (140wt%). The study of the electrochemical behaviour of these systems has been performed by cyclic voltametry at different scan rates. Figure 6 presents the cyclic voltametry of PEO/PTHF/PEDOT beam (2.6 mm×1.2 mm×0.020 mm) between -2 and 2 V in open air (two-electrode system, scan rate of 50 mV s^{-1}). The actuator exhibits a symmetric voltamogram with two distinct oxidation peaks at -0.5 and 0.5 V and two distinct reduction peaks at 0.5 and -0.6 V. This behaviour has been already observed in the case of electropolymerized PEDOT studied in EMITFSI [37]. Since EMI^+ cation is the involved ion in the redox process [37], this electrochemical behaviour has been attributed to the presence of two coexisting zones corresponding to two different PEDOT morphologies in which cation diffusion is different.

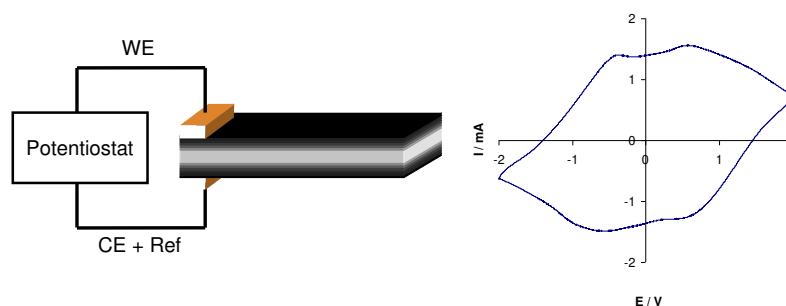


Figure 6: Cyclic voltametry of PEO/PTHF/PEDOT conducting IPN swollen with EMITFSI in a 2 electrode configuration. Scan rate: 50 mV.s^{-1} .

Figure 7-A presents voltamperograms of this actuator in a 2 electrode setup for increasing scan rates between 50 and 1.10^5 mV.s^{-1} (corresponding to frequencies between 6.25 mHz and 5 Hz) with potential windows between 4 and 10 V (from $\pm 2 \text{ V}$ to $\pm 5 \text{ V}$). Redox process can be observed for every studied scan rate. When the scan rate increases, only one oxidation and reduction peak is observed. Moreover, oxidation and reduction peak potentials present an anodic and cathodic shift respectively. Then, a broadening of the applied potential window is necessary. Conducting polymer overoxidation can still be avoided since the frequency is high enough, typically above 1 Hz . This is due to the huge ohmic drop in the thickness of the sample (serial resistance) [37]. As a consequence, the effective potential is much lower than the applied potential and does not lead to PEDOT degradation.

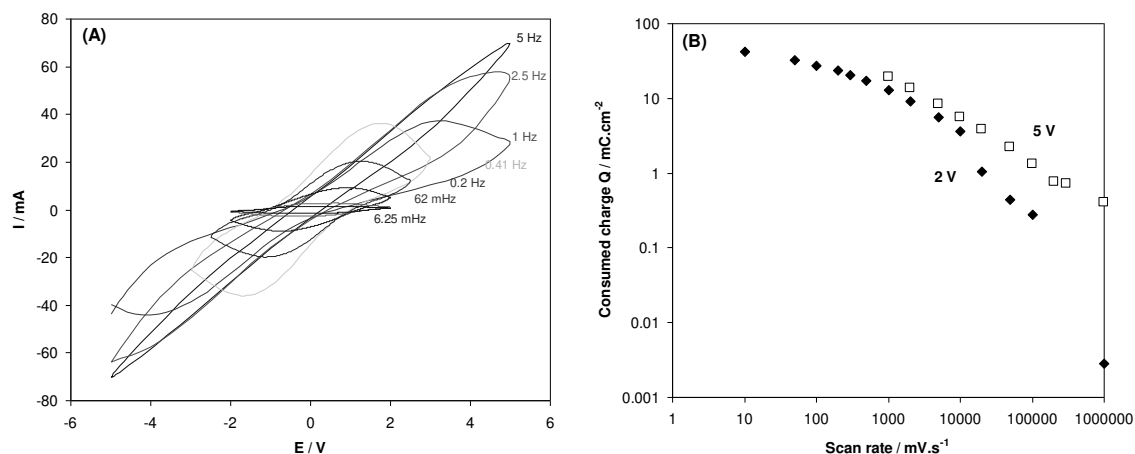


Figure 7: (A) cyclic voltametry of PEO/PTHF/PEDOT conducting IPN swollen with EMITFSI in a 2 electrode configuration at different scan rates. (B) Consumed charge Q during cyclic voltametry as a function of the applied frequency with (\blacklozenge) $+2V$ and (\square) $\pm 5 V$ electrochemical windows.

The consumed charge is reported on figure 7-B. As the frequency increases, the electrochemical charge decreases: from 19.1 to 0.4 $\text{mC}\cdot\text{cm}^{-2}$ for a $\pm 5 V$ potential windows and from 42.3 to 0.003 $\text{mC}\cdot\text{cm}^{-2}$ for $\pm 2V$ potential window. In other words, less and less PEDOT can undergo the redox process since ion diffusion remains the limiting step. The redox process of ECP based actuators is not well known at high frequencies but one can suppose that the capacitive component of the charge may become preponderant. In this case, the system starts to behave like a capacitor, mainly ruled by ion migration under the electric field. However, recent studies have described that a redox process can occur for polyaniline at frequencies as high as 1000 Hz [38]. These results allow making the assumption that a small fraction of the ECP can still undergo a redox process, even at high frequency.

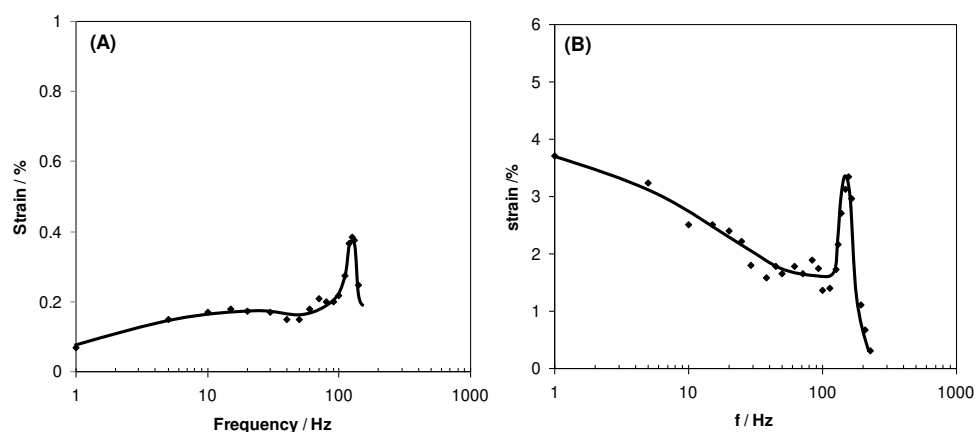


Figure 8: Frequency dependence of the generated strain for (A) PEO/PTHF/PEDOT ($2.56 \times 1.2 \times 0.02 \text{ mm}^3$) and (B) PEO/NBR/PEDOT ($3.27 \times 1.22 \times 0.09 \text{ mm}^3$) thin actuators in open-air under $\pm 5V$ square wave potential. Strain measured at 1 mm from the clamped beam.

Next, displacement measurements were recorded at an applied square-wave voltage of $\pm 5.0 V$ at different frequencies. Figures 8-A and B present the frequency dependence of the generated strain for PEO/PTHF/PEDOT (thickness: 20 μm) and PEO/NBR/PEDOT (thickness: 90 μm) actuators respectively. For PEO/PTHF/PEDOT actuator, the strain is almost constant up to 100 Hz, between 0.1 and 0.2 %. A slight increase can be observed when the applied frequency is increased and may be due to a Joule effect taking place through the thickness of the sample. This Joule effect leads to a temperature increase and then to an increase of the ionic conductivity of the SPE. In the case of the PEO/NBR/PEDOT thin actuators, the strain decreases as the frequency increases, from 3.7% at 1

Hz to 1.3% at 100 Hz. This behaviour has been already observed by several authors [35,36] and is consistent with the decrease of the electrochemical charges as a function of the frequency. However, for both conducting IPNs, when the frequency reaches 100 Hz, the tip displacement sharply increases. A maximum strain can be observed at 125 Hz and 156 Hz for PEO/PTHF/PEDOT and PEO/NBR/PEDOT respectively. This strain amplification is related with the fundamental resonance frequency of the beam and is dependent of the geometrical dimensions of the actuator. This behaviour has been previously observed in the frequency response [35] of trilayer actuators based on polypyrrole, with a resonance peak observed as high as 90 Hz. The resonance peak occurs when the actuator is operated near the system's natural frequency.

Conclusion

Actuators from conducting IPNs architecture have been demonstrated and lead to a bending deformation with different electrolytes i.e; salts and solvents. For a LiClO₄/organic solvent electrolyte, only the anion is mobile whereas for LiClO₄/water system mixed ion movement can occur according to the amount of salt loaded. The actuator thickness can also affect the actuation rate and fast response has been obtained by decreasing the thickness of the SPE. Thin actuators show a mechanical resonance higher than 100Hz for an applied potential of ±5V.

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