# ELECTROCHEMOMECHANICAL ACTUATORS BASED ON POLYPYRROLE: INFLUENCE OF THE DIMENSIONS ON THE MOVEMENT

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# ABSTRACT

Conducting polypyrrole actuators as a triple layer: (polypyrrole/adhesive tape/polypyrrole) can be operated in aqueous media. Is known that the actuator capacity in this device depends on variables that control the oxidation-reduction reaction in the conducting polymer <sup>[11]</sup>, but it has not been studied the influence of the quantity of polymer in the movement (dimensions). This study reports on investigations into responses in the triple layer, with specific reference to the influence of the mass, thickness and area of polypyrrole. Chronopotenciometry is used to study the movement of the triple layer. Parameters as consumed charge, consumed energy and movement rate are used to evaluate the behaviour of the triple layer under different dimensional and electrical conditions. According to the results is demonstrated that the movement of the triple layer can be electrochemically controlled by current flow since the volume change depend on the consumed charge per mg of polypyrrole. The relations between the amount of active polymer and the movement rate, the consumed energy and the consumed charge under galvanostatic conditions have been established.

Keywords: Electrochemomechanical, actuator, polypyrrole, conducting polymers.

### INTRODUCTION

Actuator devices specially based on polypyrrole has been developed during the past decade, their motion is based on the change of volume that the conductive polymers experience when are submitted to a oxidation or reduction reaction in a electrolytic media<sup>[2]</sup>. When a conducting polymer film is electrochemically oxidized in an electrolytic media, positive charges are generated along the polymer, counterions enter from the solution in order to keep the electroneutrality in the solid. This promotes the opening of the polymeric structure and a significant increase in free volume. Opposite processes occur during reduction: electrons are injected into the solid, positive charges are

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eliminated and counterions and solvent molecules are expelled to the solution. As result of this, two main effects occur: the polymer recovers its neutral state and the volume of the film decreases. So, to effect a change in volume in these devices, one changes the oxidation state of the polymer. This microscopical process we can see it represented in the Figure 1b.

There are many different kinds of conducting polymers, but especially polyanylyne and polypyrrole have been used to fabricate actuator devices<sup>[1,3-5]</sup>. In this paper we will refer on polypyrrole doped with  $ClO_4$ , in which the electrochemical reduction/oxidation process can be shown as in the reaction [1]:

# $(Polymer)_{s} + (nClO_{4})_{aq} \Leftrightarrow [Poiymer^{n+}(ClO_{4})_{n}]_{s} + ne_{metal}$ [1]

The volume change is linked to conformational changes generated by the presence of oxidation and reduction processes in the conducting film of Ppy. But is necessary the translation of these microscopic conformational changes to macroscopic movements. In our laboratory this problem was solved in the first stages of the research with the design of a bilayer (Ppy/adhesive tape)<sup>[6]</sup>. Although the bilayer device was very useful to demonstrate that it is possible to fabricate systems with electrochemomechanical properties, this device has important limitations, for example the necessity of using a counter electrode to allow the current flow. To solve this problem and improve the operation of the bilayer a new device was developed in order to profit the same current two times: a triple layer (Ppy film / adhesive and flexible tape / Ppy film). Its construction is easy and is explained with detail in the experimental section. In the triple layer one of the Ppy films acts as anode and another Ppy film as cathode. The Ppy film as counter electrode was short-circuited with the reference electrode contact of the potenciostat. At macroscopic level when a Ppy film of the triple layer is oxidized this progressively expands, this active movement is transmitted to the adjacent adhesive tape. Thereby creating a gradient of tension across it. Meanwhile the other polymer reduces and shrinks, the gradient of tension created in the interface Ppy-adhesive tape pushes the triple layer into the Ppy film that is reducing. These expansion-shrinking processes occurring in both Ppy films cause a gradient of tension in the corresponding interfaces Ppy/adhesive tape. These gradients of tension are contraries, while one is of expansion another one is of contraction. However, as the Ppy films are opposed the gradients of tension are supplemented and the effects are joined for taking place the movement. All these processes are produced reversibly (see Figure 1). Previously has been demonstrated that the motion of the bi and triple layer depend on the variables that control the kinetics of electrochemical reaction occurring in the polymer (voltage, current

density, electrolyte concentration, concentration of positive charges) <sup>[1,7]</sup>. The study of the influence of these variables has shown that is possible to control the position and movement rate of the triple layer through of potential or current. So, there is a defined potential to each position of the triple layer and a unique number of positive charges formed in the polymeric chains (consumed charge) related with a crossed angle. The absolute control of the motion of the triple layer by means of the electrical conditions makes Ppy an intelligent material. But to use successfully Ppy as an electroactuator the response must be stable when the triple layer has different dimensions. To determine whether this is achievable with triple layer (Ppy/ adhesive and flexible tape/Ppy) Ppy films of different areas, weight and thickness must be used to fabricate this device. Also it is important to know the movement rate that would reach the triple layer when this has different dimensions of electroactive material, as well as the power necessities for their movement.

This paper reports on work aimed at characterising these dimensional parameters in actuator response by means of chronopotentiometry. Integrating the chronopotentiograms is possible to get the consumed charge and electrical energy by the Ppy during a movement. These parameters and the movement rate will be used to evaluate the actuator response of the different triple layers.

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Figure 1. The figure 1b represents the change of reversible volume that happens microscopically in the Ppy when it is submitted to a reaction of oxidation or reduction in an electrolytic media. This volume change is due to the entry of counterions (ClO<sub>4</sub>) from solution to the polymeric chains, these ions neutralise the positive charges that are formed during an oxidation reaction. When the polymer is reduced, the ions exit towards the solution to maintain the electroneutrality diminishing the volume polymer. The transformation of this change of volume in the polymeric chains at a macrocospical level, has been obtained through a triple layer device (Ppy/adhesive tape/Ppy). In this triple layer, one of the films of Ppy acts as work electrode (W.E.) and the other as counter electrode (C.E.), the contact of the reference electrode (R.E.) of the potenciostato is short-circuited with the C.E. When the film that acts as W.E. is oxidized this one expands progressively (by the anion entrance from the solution), while the film that acts as C.E. is being contracted (by the exit of anions of polymer). This expansion and contraction are complementary and push the triple layer towards the film that is reducing (shrinking). This is a totally reversible process. In the Figure 1a is shown a movement of 180 degrees of the triple layer: when it is moving towards the left, the film acting as C.E. is oxidising and when it moves towards the right the film acting as W.E. is oxidising. This triple layer has an area of 3cm<sup>2</sup> and 12 mg of Ppy, it acts at 1M LiClO4 aqueous solution and with a current of 10 mA.

## RESULTS AND DISCUSSION

#### Surface Area Influence on the Movement

According to the microscopic model of the triple layer, the oxidation reaction of a Ppy film generates positives charges along their polymeric chains. To maintain the electroneutrality in the polymer some ClO<sub>4</sub><sup>-</sup> ions of the solution enter to neutralise the positive charges in the polymer, the entry of these anions produces a increase of volume in the polymer. Meanwhile the film acting as

counter electrode is being reduced producing the exit of ClO<sub>4</sub><sup>-</sup> to continue the electroneutrality in the film and therefore decreasing their volume. The volume changes (increase and decrease) are complementary in the two Ppy films pushing the triple layer towards the film that are being reduced (contracted) in a totally reversible process. Thus, the consumed charge (oxidation degree) during a movement of the triple layer will have to be related directly to the crossed angle, independently of the electrical, physical and chemical conditions (e.g. current or potential applied, temperature, electrolyte concentration). This has been demonstrated when a constant movement of a triple layer (with Ppy films of 3 cm<sup>2</sup> and thickness 13  $\mu$ m) has been evaluated under different conditions<sup>[7]</sup>. Now is necessary to demonstrate that the relationship between the consumed charge and the crossed angle is maintained when the dimensions of the triple layer are different: When the mass, the thickness or the area of the Ppy films has any value. This finally will demonstrate the electrical charge (a unique oxidation degree) so that the triple layer crosses a given distance at an electrolytic media.

So, triple layers of different areas  $(3, 2.5, 2, 1.5 \text{ and } 1 \text{ cm}^2)$  were constructed with Ppy films having a constant thickness of 13 µm (see experimental section). A 90 degrees movement was evaluated under different galvanostatic conditions (10, 15, 20, 25 and 30 mA) with choronopotentiometry. Consumed electrical charge, movement rate and consumed energy during the movement of each triple layer had been calculated from the obtained chronopotentiograms. The consumed charge (Q) is defined as the integral of the current that goes by the working electrode during the time that the movement lasts. So, the consumed electrical charge (Q) in mC was calculated as:

$$Q(mC) = JI \cdot dt \qquad (mA) (s) \tag{1}$$

The electrical energy consumed by the triple layer to cross an angle is calculated by means of the integration of the electrochemical registers, for what the electrical energy (Ee) is the integral of the electric power in the time that the movement lasts and is given by the following equation:

$$Ee (mJ) = \int I \cdot E dt \quad (mA) (V) (s)$$
(2)

The movement rate (r) is given by the relation between the crossed over angle ( $\theta$ ) and the required time to cross over it (t).

$$r (rad/s) = d\theta / dt (rad)/(s)$$
(3)

The obtained chronopotentiograms during the movements registered an increase of the electrical potential through of the triple layer when the area of the triple layer decreased. Consequently, the necessary times to cross over a same angle are shorter for the smallest areas. Since the consumed charge is given by the multiplication of the applied current and the time that the movement lasts (galvanostatically), at the same electrical conditions the charge will be smaller for the smallest areas

(Figure 2). On the other hand, when the movement of a triple layer is evaluated with different currents, its movement is faster with the highest currents. However, the consumed charge stays constant. This is because the triple layer reaches an only state of oxidation when crossing a determined angle, independently of the applied current density (Figure 2). Nevertheless, if we consider the consumed charge per Ppy cm<sup>2</sup> this is independent of the current and the area of the triple layer (Table 1). According to these results the consumed charge during a movement of 90 degrees by a triple layer made with Ppy films of 13 µm thickness has the following behaviour:

Q (mC) / A (cm<sup>2</sup>) = 93.5 mC/cm<sup>2</sup> Q = consumed electrical charge (mC) A = area of the triple layer (cm<sup>2</sup>)(4)

Therefore, the consumed charge by the triple layer during a movement of 90 degrees at 1M  $LiClO_4$  aqueous solution is 93.5 mC·cm<sup>-2</sup>, a value non dependent on the area of the Ppy films.

As we mentioned previously, the necessary times so that a triple layer crosses 90 degrees, become shorter when the area of the triple layer is smaller. Therefore to smaller area, higher movement rate. At the same time, when the applied current is greater the movement rate of the system is higher. However, it was found that the movement rate has a direct relation with the applied current per area unit, this relation is independent on the area of the triple layer (Figure 2a). So, the rate of a triple layer made with Ppy films of 13 µm of thickness is given by the equation 5.

 $r (rad/s) = 0.029 (rad cm<sup>2</sup>/s mA) \cdot I (mA) / A (cm<sup>2</sup>)$ (5) r = movement rate (rad/s) I = applied current (mA)A = area of the triple layer (cm<sup>2</sup>) \_\_\_\_\_

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Figure 2. Influence of Ppy area on the triple layer movement at different galvanostatic conditions: B area=  $3 \text{ cm}^2$ , mass $\cong 6 \text{ mg}$ ;  $\blacklozenge$  area =  $2.5 \text{ cm}^2$ , mass $\cong 5 \text{ mg}$ ;  $\Box$  area =  $2 \text{ cm}^2$ , mass $\cong 4 \text{ mg}$ ;  $\blacklozenge$  area=  $1.5 \text{ cm}^2$ , mass $\cong 3 \text{ mg}$ ;  $\neg$  area=  $1 \text{ cm}^2$ , mass $\cong 2 \text{ mg}$ . The figure shows the behaviour of the consumed charge, consumed energy and the movement rate of the triple layers during a movement of 90 degrees at 1M LiClO<sub>4</sub> aqueous solution and under 10, 15, 20, 25 and 30 mA.

Table 1: Consumed charge per cm<sup>2</sup> by triple layers of different areas during a movement of 90 degrees and under different anodic currents. The thickness of the Ppy films was 13  $\mu$ m and the movements were made in 1M LiClO<sub>4</sub> aqueous solution.

I/mA	Charge/mC·cm <sup>2</sup>				
	$1 \text{ cm}^2$	1.5 cm <sup>2</sup>	$2 \text{ cm}^2$	2.5 cm <sup>2</sup>	3 cm
10	88	87	89	90	90
15	91	92	88	90	91
20	90	90	90	92	89
25	88	91	91	92	90
30	89	92	88	92	90

The consumed electrical energy (Ee) by the triple layer increases when is greater the area of Ppy in the triple layer (Figure 2). This is related to the longest times necessary to cross the same angle when the Ppy area is bigger. However, the consumed electrical energy per Ppy area unit is directly proportional to the applied current and independent on the Ppy area. The relationship between the consumed energy per Ppy  $cm^2$  by a triple layer (thickness of the Ppy films 13 µm) during a movement of 90 degrees and the applied current is given in the following equation:

Ee (mJ) / A (cm<sup>2</sup>) = 7.5 (mJ/cm<sup>2</sup> mA)  $\cdot$  I (mA) (6) Ee = consumed electrical energy (mJ) A = area of the Polypyrrole film (cm<sup>2</sup>) I = applied current (mA)

That is to say, the consumed energy in mJ per cm<sup>2</sup> Ppy by a triple layer during a movement of 90 degrees at 1M LiClO<sub>4</sub> aqueous solution, is 7.5 times the applied current in mA.

### Thickness Influence on the Movement

This study comprehend the fabrication of triple layers with Ppy films of different thickness (16, 13, 11, 8, and 5  $\mu$ m) when the area of Ppy films was maintained constant (3 cm<sup>2</sup>). A movement of 90 degrees of the triple layers in 1M LiClO<sub>4</sub> aqueous solution had been evaluated with different anodic currents (10, 15, 20, 25 and 30 mA). Applying to a constant current the triple layers with smaller thickness move more quickly due to highest potentials through the Ppy films. In the same way, when the current through a triple layer is increased, this one is submitted to higher potentials and therefore it moves at higher rate (Figure 3). Relating these results we found that the movement rate of a triple layer is proportional to the applied current per thickness according to the following equation (7).

r (rad/s) = 0,12 (rad μm/mA s) · I (mA) / μ (μm) (7) r= movement rate (rad /s) I= electrical current (mA) μ= thickness (μm)

Similarly to the evaluation of the area, the characterisation of the movement of the triple layer with films of different thickness demonstrates that independently of the applied current, the consumed charge by a triple layer during a given movement stays constant. This we can see it in the Figure 3,

the triple layer with the films of smaller thickness consumes around 120 mC during the movement with any current density, whereas the triple layer with the films of greater thickness consumes 370 mC. Therefore, a triple layer of any area or thickness consumes an only charge when crossing a defined distance, independently of the applied electrical current. Also we found that the consumed charge per thickness is independent on the thickness of the Ppy films. So, the consumed charges by the triple layers were given by the following equation.

 $Q (mC) / \mu (\mu m) = 23 mC/\mu m$  Q = consumed charge (mC)  $\mu = \text{thickness of the Ppy films (\mu m)}$ (8)

Therefore, the triple layer consumes 23 mC/ Ppy µm during a 90 degrees movement at 1M LiClO<sub>4</sub> aqueous solution and under any applied current.

In relation to the consumed electrical energy, this increases when is higher the applied current to a triple layer (Figure 3). When the current is the same, the consumed energy increases with the thickness of the Ppy films due to the longest times for the same movement. These results allow to establish a relation between the consumed energy and the thickness. So, the consumed energy per thickness of Ppy increases linearly with the applied current with a slope of 1.9, non dependent of the thickness Ppy films.

Ee (mJ) /  $\mu$  ( $\mu$ m) = 1,9 (mJ/ $\mu$ m mA) · I (mA) Ee = consumed electrical energy (mJ)  $\mu$  = thickness of the polypyrrole films ( $\mu$ m) I = applied electrical current (mA) (9)



Figure 3. Influence of Ppy thickness on the triple layer movement at different galvanostatic conditions: B thickness= 16  $\mu$ m, mass $\cong$  6 mg;  $\blacklozenge$  thickness= 13  $\mu$ m, mass $\cong$  5 mg;  $\Box$  thickness= 11  $\mu$ m, mass $\cong$  4 mg;  $\blacksquare$  thickness= 8  $\mu$ m, mass $\cong$  3 mg;  $\tau$ thickness= 5 $\mu$ m, mass $\cong$  2 mg. The figure shows the behaviour of the consumed charge, consumed energy and the movement rate of the triple layers during a movement of 90 degrees at 1M LiClO<sub>4</sub> aqueous solution and under 10, 15, 20, 25 and 30 mA.

## Mass Influence on the Movement

In order to complete this work is necessary to know the equations that characterise the movement of the triple layer when its mass varies. The Ppy masses in the evaluated triple layers are the following ones: 6, 5, 4, 3, 2 and 1 mg.

Knowing the masses of Ppy is possible to relate them to the previous results to establish the influence of the mass in the movement of the triple layer. This way, we found the results that are showed in Figure 4. The analysis of them shows a lineal relationship between the movement rate and the applied current per Ppy mass (Figure 4a). This relationship between the movement rate of the triple layer and the applied current per mg follows the equation 10.

 $r (rad/s) = 0,06 (rad mg/s mA) \cdot I (mA) / m (mg)$  (10)

r= movement rate (rad /s) I= electrical current (mA) m= mass of the Ppy (mg)

The calculations of the consumed charge show that the consumed charge per Ppy mass is independent on the applied current and on the dimensions of the triple layer (Figure 4b). The charge in mC is given by the following equation:

Q (mC) / m (mg) = 46 mC/mg Q = consumed charge (mC) m = mass of the Ppy (mg)(11)

This means that a triple layer with any dimension consumes 46 mC / Ppy mg during a movement of 90 degrees at 1M LiClO<sub>4</sub> aqueous solution at under any applied current.

From the figure 4c we can establish that the consumed energy per Ppy mass and the applied current are related directly. The consumed energy per Ppy mass unit increases linearly when applied current increases. This relationship is given by the following equation:

Ee (mJ) / m (mg) =  $3.9 \text{ (mJ/mg mA)} \cdot \text{I} \text{ (mA)}$  (12) Ee = consumed electrical energy (mJ) m = mass of the polypyrrole (mg) I = Applied electrical current (mA)

In the conditions of this work the equations 10 - 12 and the Figure 4 demonstrate us that the energy per Ppy mass unit and movement rate of a triple layer are directly related with the applied current density. But the consumed electrical charge per mg is independent of the electrical conditions. With the information processed from chronopotentiograms obtained experimentally, it has been possible to deduce the equations 4-12. These equations allow us previously know the consumed charge, the consumed energy and the movement rate of a triple layer (polypyrrole / adhesive tape / polypyrrole ) during a movement of 90 degrees at 1M LiClO<sub>4</sub> aqueous solution at 25 °C regardless of the dimensions of the device.

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Figure 4. Influence of Ppy mass on the triple layer movement at different galvanostatic conditions: a) Movement rate (rad s<sup>-1</sup>) of triple layers related to the applied current per milligram of Ppy (mA mg<sup>-1</sup>). The movement was of 90 degrees and has been evaluated at 1M LiClO<sub>4</sub> aqueous solution and under 10, 15, 20, 25 and 30 mA. b) Evolution of the consumed electrical charge per mg of Ppy (mC mg<sup>-1</sup>) with the applied current (10, 15, 20, 25 and 30 mA), during a movement of 90 degrees in 1M LiClO<sub>4</sub> aqueous solution. c) Evolution of the consumed electrical energy per mg of Ppy (mJ mg<sup>-1</sup>) with the applied current (10, 15, 20, 25 and 30 mA), during a movement of 90 degrees in 1M LiClO<sub>4</sub> aqueous solution.

#### EXPERIMENTAL

The polypyrrole films were synthesised and checked in a one compartment electrochemical cell connected to a PAR M273 potentiostat-galvanostat and controlled through a PC Pentium computer by means of the software PAR 270/250. Three AISI 304 stainless steel sheets, having a surface area of 3.5 cm<sup>2</sup>, were used; one of them as working electrode the others as counter-electrode. An Ag/AgCl electrode from Crison Instruments was introduced directly into the cell. Pyrrole (Jansen) was distilled under vacuum before use. Water (obtained from Milli Q Reagent Water system equipment, fed by Milli RO 4 water purification system), acetonitrile (Lab. Scan, HPLC grade) an anhydrous Lithium Perchlorate (Jansen, min 99% content) were used as received.

Polypyrrole films were synthesised using potential square waves applied between -0.3 V (2s) and 0.872 V (8s) to control the morphology of the film and its adherence to the metal. Subsequently the polypyrrole films were partially oxidized. The solutions used for the synthesis of the polypyrrole film were: pyrrole 0.2 M and LiClO<sub>4</sub> 0.1 M in acetonitrile + 2% water needed to provide films having a high ability to store charge. This method has being optimised to obtain homogeneous, flexible, good quality Ppy films.

All the prepared solutions were de-areated by bubbling N2 gas for 10 minutes before the current flow. Both sides of the working electrode were uniformly covered. The uniformity on the electric field was maintained using two stainless steel counter-electrodes, 3.5 square centimetres of surface area each. Once extracted from the solution the film is rinsed with acetonitrile and dried. Once rinsed and dried the polypyrrole films, a double-sided tape, protected on one side, is adhered to the partially oxidized polypyrrole film coating on one side of the stainless steel. The triple layer formed by polypyrrole/double -sided tape/protecting plastic layer, was removed from the stainless steel plate. Then the protecting plastic is removed and the bilayer polypyrrole/double-sided plastic tape was stuck to the remaining polypyrrole coated face of the stainless steel electrode. The double-sided plastic tape acts both, as an electronic insulator between the polypyrrole films and as a support for stress gradients, origin of the macroscopic movements, during muscle work. One of the conducting film is connected to the anode, thereby swelling and pushing the free bottom of the triple layer. The second conducting film is connected to the cathode thereby shrinking, by reduction processes, and trailing the triple layer. The polypyrrole film as counter electrode was short-circuited with the reference electrode contact of the potenciostat. No electric contact exists between the two polypyrrole films due to the presence of the non-conducting layer in between. The electric flow between the conducting films takes place by jonic conduction through the solution. Triple layers of different areas (3, 2.5, 2, 1.5, 1 cm<sup>2</sup>) were obtained from the original triple layer (2 x 1.5 cm, Ppy thickness 13 µm each film, mass of 6 mg): it was carefully cut with a scissors to obtain the wanted area. The thickness of those films was controlled through the overall charge consumed, on each cathodic step to reduce (the extent depends on the polarization time) the polymer film. During each anodic step the polymer is oxidised again and the polymerization goes on. The thickness and the mass follow the correlation shown in the Figure 5. From the polypyrrole weight obtained by means of a microbalance (precision  $10^{-7}$  g) and the polymer density of 1.51 g cm<sup>-3</sup> obtained by flotation in a CHCl<sub>3</sub> + CCl<sub>4</sub> mixture, the thickness was calculated for the Ppy films. The equation that relation the mass, the area and the thickness with the density of the film is the following:

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(13)

 $\rho = m v^{-1} = m \cdot (\mu \cdot A)^{-1}$   $\rho = Ppy \text{ density } (g \text{ cm}^{-3})$  m = Ppy mass (g)  $v = Ppy \text{ volume } (cm^3)$   $\mu = Ppy \text{ thickness } (cm)$   $A = Ppy \text{ area } (cm^2)$ 

The evaluations were made in 1M LiClO<sub>4</sub> aqueous solution and the movement studied was a bending of 90 degrees related to the vertical position. This angle was chosen with the purpose of always beginning the movement from the equilibrium potential of the system and always having an ascent movement. The trajectory of this angle is more easily definable and visible.



Figure 5. Experimental calibration that relates the Ppy mass deposited over working electrode and the polimerization charge.

# FINAL COMMENTS

The results presented in this paper complete the characterisation of the movement of the triple layer device. Now it is possible to confirm the direct relation between the degree of oxidation of Ppy and the crossed angle by the device: The consumed charge per Ppy mg by a triple layer is only related

with its movement and is independent on electrical conditions. The consumed electrical energy and the movement rate per Ppy mass unit increase when is higher the applied current to the triple layer. Therefore, the consumed charge, consumed energy and the movement rate depend on the amount of electroactive Ppy in the triple layer. The equations presented have shown a suitable correlation to each other, this will allow us to use them to predict the movement parameters (consumed charge, consumed energy and rate) accurately when the dimensions of Ppy are known. This is important in the design of actuator tools with triple layers of different dimensions.

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