

Poly (3,4-ethylenedioxythiophene): Poly (styrene sulfonate)(PEDOT/PSS) / Potassium dihydrogen phosphate (KDP) composite thick film for use in an actuator device

Poli (3,4-etilenodioxiotiófeno): Película espessa composta de poli (sulfonato de estireno)(PEDOT/PSS) / Di-hidrogenofosfato de potássio (KDP) para utilização num dispositivo actuador

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ABSTRACT

An actuator based on a thick film of the composite Poly (3,4-ethylenedioxythiophene): Poly (styrene sulfonate) (PEDOT: PSS) / Potassium dihydrogen phosphate (KDP) is proposed in this work. Both materials are water-soluble and easy to purchase. The composite is easy to produce and is very reproducible. Three different types of substrates were used for the mechanical support of the thick film: polyethylene terephthalate (PET) covered with tin and indium oxide (ITO) (PET / ITO), polyester and aluminium foil. Film thicknesses ranged from 6.9 μm to 43.2 μm . The concentration of KDP in the films varied between 2 mg and 10 mg per mL of PEDOT: PSS. The thick films were electrically characterised and then subjected to performance tests. Displacements were observed in all the samples tested, for which DC voltages from 1 to 6 V were applied. Measurements of displacement as a function of the voltage application time, and as a function of the applied

voltage, were collected. The former showed linear behaviour while the latter showed non-linear behaviour. It was observed that the actuator effect is monopolar in these samples. The observed displacement was of the order of 5 mm. The samples sustain displacement while the voltage is applied. All samples operate in an ambient condition. The strain (ϵ) value calculated, using the data obtained experimentally, was 2.6%.

Keywords: PEDOT:PSS/KDP, thick films, actuator device, flexible

RESUMO

Um actuador baseado num filme espesso de Poli (3,4-etilenodioxiotiófeno) composto: Poli (sulfonato de estireno) (PEDOT: PSS) / Di-hidrogenofosfato de potássio (KDP) é proposto neste trabalho. Ambos os materiais são solúveis em água e fáceis de adquirir. O composto é fácil de produzir e é muito reprodutível. Foram utilizados três tipos diferentes de substratos para o suporte mecânico da película espessa: tereftalato de polietileno (PET) coberto com estanho e óxido de índio (ITO) (PET / ITO), poliéster e folha de alumínio. A espessura da película variava entre 6,9 μm a 43,2 μm . A concentração de KDP nos filmes variou entre 2 mg e 10 mg por mL de PEDOT: PSS. As películas espessas foram caracterizadas electricamente e depois submetidas a testes de desempenho. Foram observados deslocamentos em todas as amostras testadas, às quais foram aplicadas tensões DC de 1 a 6 V. Foram recolhidas medidas de deslocamento em função do tempo de aplicação da tensão, e em função da tensão aplicada. As primeiras mostraram um comportamento linear, enquanto que as segundas mostraram um comportamento não linear. Observou-se que o efeito do actuador é monopolar nestas amostras. O deslocamento observado foi da ordem de 5 mm. As amostras sustentam o deslocamento enquanto a tensão é aplicada. Todas as amostras funcionam em condições ambientais. O valor da tensão (ϵ) calculado, utilizando os dados obtidos experimentalmente, foi de 2,6%.

Palavras-chave: PEDOT:PSS/KDP, filmes espessos, dispositivo actuador, flexível

1 INTRODUÇÃO

Research on new materials and methods for the manufacture of actuators for use in robotics, artificial muscles, applications in bioelectronic devices, etc. has developed a lot in the last decade [1-3]. The main objective is to find materials that are compatible with human tissue, and that are light, soft, have mechanical flexibility, easy processing, low cost, large-scale production and use manufacturing methods that do not harm the environment. Polymers are materials that have the characteristics and properties listed above. At the time of writing, the type of actuator that draws the most attention is the electroactive polymer actuator (EAP). This type of actuator is either an ionic EAP or a field-activated EAP [4-5]. The ionic EAP works by ion diffusion and operates at low voltages (1-2 V), while the field-activated EAP operates from Coulomb force and operates at higher voltages. There are advantages and disadvantages to these two groups of actuators and these have been well-summarised in the work of Yoseph Bar-Cohen and collaborators

[6]. The requirements for a good actuator are: production of displacements with great inclination, need for low voltage for its use, bidirectional actuation that depends on the polarity of the applied voltage, operation in ambient conditions, fast response and that is able to maintain the displacement with inclination under A.D. activation. The field-activated EAP can still be of two types: ferroelectric polymer actuator and dielectric elastomer actuator [7-8]. In the case of the ferroelectric polymer actuator, the most used material is Poly (vinylidene fluoride) (PVDF) [8]. PVDF is a polymer that has piezoelectricity, however, it needs to be stretched to orient the chains, in addition to being polarised with a relatively high voltage. It is also an expensive material. An actuator using composite material with an organic filler that has a high electrical permittivity value (greater than 10) mixed in a polymeric matrix, was developed by Zhang et al. [9]. Currently, conductive polymers (CP) have been considered as an alternative to the actuator based on piezoelectric materials [10]. A sandwich of two conductive polymer electrodes (polypyrrole, or PEDOT:PSS) with an electrolyte between them forms an EAP actuator [11], also called a three-layer actuator. As for the manufacturing techniques for these EAP actuators, a wide variety (such as spin coater, casting, micro contact printing and syringe-based printing [12-14]) have been used. It has already been observed that, in addition to the mechanical response to electricity, some EAP materials also exhibit the ability to transform a mechanical strain into electrical energy [15].

In this work, we present an actuator based on the thick film of a composite of a conductive polymer: Poly (3,4-ethylenedioxythiophene): Poly (Styrene Sulfonate) (PEDOT: PSS) and the piezoelectric salt Potassium Dihydrogen Phosphate (KDP). Both materials are soluble in water. We must make it clear here, that the use of liquid electrolytes is not necessary for the operation of this actuator. (KDP) is a salt of minimal formula KH_2PO_4 with a solubility coefficient equal to 22 g / 100 g of H_2O) is used as a fungicide, a food additive in isotonic drinks, a food source of phosphorus and potassium, and a buffering agent in fertiliser mixtures [16], which shows its non-toxicity. In addition, it has applications in the areas of electronics and physics, due to its interesting optical and piezoelectric properties. This composite has already been used to make a pressure sensor [17-18]. The advantage of using this composite is in the synergy that appears between the conductive polymer and the piezoelectric salt. When the composite was used to build the pressure sensor, it was observed that the charges generated by the salt when the sample was pressed were better conducted by the polymer to the external circuit. Now, the application of a voltage to the film produces an expansion / reduction in the salt crystals and its

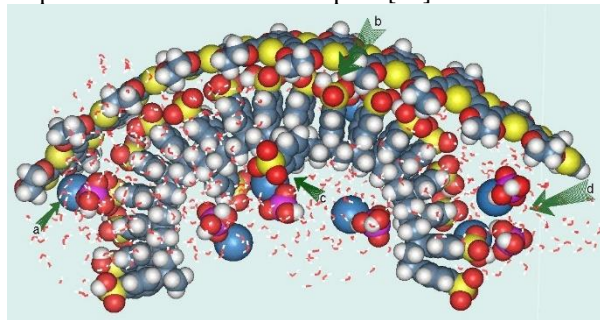
rearrangement within the polymer matrix causes a displacement in the film. Through the molecular modelling performed in reference [19] it can be seen that KDP does not dissociate into ions, as might be expected. Thus, this actuator cannot be classified as an ionic EAP or as a polymer - ionic metal (IPMC) composite [20]. Nor can it be classified as an EAP, since the polymer itself is not electroactive. In this way we have a new type of actuator that is based on polymer but is not an EAP. In this study, the field-activated composite actuator is not manufactured with a ferroelectric polymer but with a salt that is piezoelectric and, therefore, does not require prior polarisation. The developed actuator consists of a substrate for mechanical support that can also be an electrode and the thick layer (micrometres in size) of PEDOT: PSS / KDP being another electrode. The casting technique was used to form the thick film. Performance tests were performed on the samples. The displacements under DC voltages ranging from 1 to 6 V were measured. The manufacture of this type of actuator is very easy and fast and does not require any expensive equipment or a long preparation time. The actuator proposed in this work operates in an ambient condition and does not require an aqueous environment (it is not based on an electrolyte).

2 EXPERIMENTAL

2.1 MATERIALS

KDP (molar mass of 136.09g / mol) in the form of crystals was purchased from Sigma Aldrich Chemical. PEDOT: PSS with 1.3% by weight of conductive water dispersion was also purchased from Sigma Aldrich Chemical. It was decided that the KDP solubilisation would be carried out in the PEDOT: PSS solution itself, without adding more water. The solubility of KDP in PEDOT: PSS has been tested and amounts of KDP below 40 mg / mL are fully solubilised. For the complete homogenisation of the composite, the PEDOT: PSS / KDP solution was stirred for 30 min at 50°C. In a previous work [19], to observe the interactions of the composite components with each other and with water, dynamic molecular modelling was performed, as shown in Figure 1. The arrows a, b, c and d show the KDP crystals. It can be noted that the crystal does not dissociate into ions, which indicates that our film is not of the ionic type. On the other hand, it can be observed that, when submitted to an electric voltage, the expansion or reduction of the crystals tends to cause movement of the PEDOT: PSS chains.

Figure 1. Representation of the PEDOT: PSS / KDP mixture in aqueous medium after molecular dynamics calculations [19]. Sulphur atoms (S) are yellow, oxygen (O) red, hydrogen (H) white, carbon (C) grey, potassium (K) blue and phosphorus atoms are shown as pink [19].



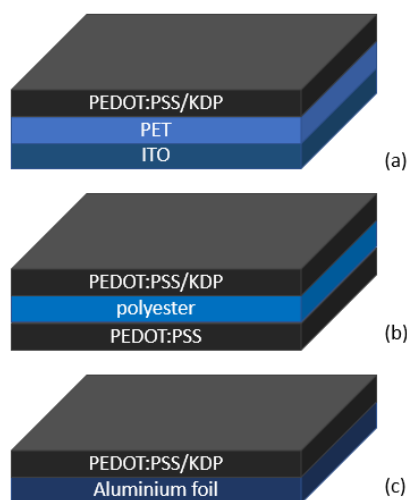
The substrates used for the mechanical support of the composite film are all flexible and light: polyethylene terephthalate (PET) covered with tin and indium oxide (ITO) with a resistance of $60 \Omega / \square$ purchased from Sigma Aldrich. The polyester foil and aluminium foil was purchased from local stores. The choice to use aluminium foil was made for two reasons: 1) it can be used as an electrode to improve the electrical conductivity of the actuator and, 2) it reduces manufacturing cost, since it can replace electrodes made of precious materials like gold, silver and platinum. The PET / ITO sheet also fulfils the role of an electrode. All substrates were cleaned with isopropyl alcohol. 7 mm wide, 45 mm long strips were cut for PET / ITO and polyester substrates before depositing the composite. The aluminium substrate was cut to the dimensions: 5 mm wide and 30 mm long, only after the deposition of the composite.

2.2 SAMPLE PREPARATION

For samples with PET / ITO substrate, three concentrations of KDP in PEDOT: PSS were used: sample 1 with 2 mg of KDP for each mL of PEDOT: PSS, sample 2 with 6 mg of KDP for each mL of PEDOT : PSS and sample 3 with 10 mg KDP for each mL of PEDOT: PSS. To obtain films with different thicknesses, three different amounts of composite were deposited by drop casting on the substrates. For samples 1.1, 2.1, and 3.1, 0.4 mL of the composite solution was deposited by casting. 0.8 ml of composite solution was deposited by drop casting for samples 1.2, 2.2 and 3.2 and 1.3 ml for the third set of samples (1.3, 2.3 and 3.3). The structure made for these samples is shown in Figure 2a, a substrate and a thick composite film PEDOT: PSS / KDP deposited by casting on its surface. Samples were made by replicating the quantities used in samples 1 to 3 only with PEDOT: PSS, to verify that the actuator effect is only due to the addition of KDP.

In the polyester samples, a layer of PEDOT: PSS was deposited by casting on the underside of the substrate to function as an electrode. After drying, the PEDOT: PSS / KDP composite solution was deposited on top of the substrate. For samples with polyester substrate, the concentration of the composite used was 7.5 mg KDP for each mL of PEDOT: PSS. Three samples were taken: sample 4.1 with a solution of 0.1 mL, sample 4.2 with a solution of 0.2 mL and sample 4.3 with a solution of 0.4 mL. Figure 2b shows the structure for these samples.

Figure 2: Sample structure with a) PET / ITO substrate; b) polyester substrate and c) aluminium substrate.



As the samples on a polyester substrate proved to be light and flexible, another type of light substrate that is a good conductor was tested: aluminium foil. This structure can be seen in Figure 2c. Aluminium foil, in addition to serving as a substrate, is also an accessible and inexpensive electrode that can replace more expensive electrodes such as gold, silver and copper. Two samples were produced: sample 5.1 was produced by casting the composite at a concentration of 7.5 mg KDP for each 1 mL of PEDOT: PSS on the surface of the aluminium foil in the amount of 0.2 mL; sample 5.2 is only aluminium foil, used as a reference, to ensure that the movement/displacement observed in sample 5.1 was not caused by the thermal effect (expansion of the aluminium foil by a large current).

All samples were placed in the oven at 40° C for 24 h.

3 RESULTS AND DISCUSSION

3.1 THICKNESS MEASUREMENTS

The thickness measurements of each substrate and each of the samples were performed using an Instrutherm model ME-240 layer thickness gauge. This equipment has calibration tapes for some thickness ranges whose errors are about 0.05 μm . The errors in the measurements taken are expected to be of this order. The measurements were made at five different positions of the substrate / sample and the values were averaged. The thickness of each of the three substrates was measured. The PET / ITO substrate thickness was 125.4 μm ; for the polyester substrate it was 85.5 μm and for the aluminium substrate it was 16.6 μm . The thickness of the samples was then measured (substrate plus film). The thickness of the substrate was removed from the total thickness so that the thickness of the composite film was obtained. The thickness of the thick film is important for determining the conductivity of the films. As expected, it was observed that the greater the amount of composite deposited on the substrate, the greater the thickness of the film.

The values of the thickness of the films for all samples made are shown in Table I. The thickness obtained varies from 6.9 to 43.2 μm . These values characterise the films as thick. Note that for samples 4 (polyester substrate) and 5 (aluminium substrate), the thicknesses were smaller because the deposited PEDOT: PSS / KDP solutions were smaller.

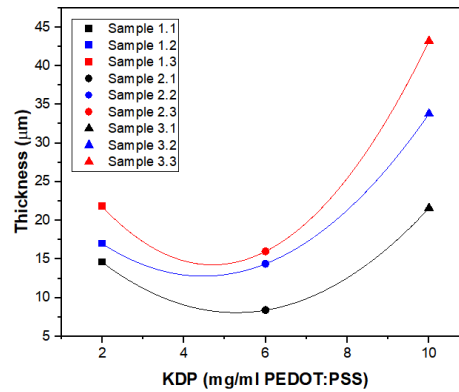
Table I. Thickness and resistance measurements for PEDOT:PSS/KDP thick film samples on different substrates.

Sample	Film resistance (k Ω /cm ²)	Total device average thickness (μm)	Film average thickness (μm)
1.1	12.3	142.4	14.6
1.2	7.6	147.2	17.0
1.3	9.7	140.0	21.8
2.1	9.9	133.8	8.4
2.2	6.5	139.8	14.4
2.3	8.8	141.4	16.0
3.1	37.8	147.0	21.6
3.2	26.5	159.2	33.8
3.3	38.3	168.6	43.2
4.1	1063.0	97.7	6.1
4.2	1411.0	109.6	12.0
4.3	1730.0	115.8	15.1
5	1411.0	23.5	6.9

Figure 3 shows the thickness of films with different KDP concentrations for samples made on PET / ITO substrate. We can see that, for a concentration of 6 mg of

KDP, the thicknesses of samples 2 are lower than those of samples 1 and 3. This is because the amount of KDP solubilised in PEDOT: PSS was excellent, i.e. the KDP crystals are distributed so as not to form agglomerates and spread evenly over the substrate surface.

Figure 3: Thick film thickness of PEDOT: PSS / KDP composite on PET / ITO substrate as a function of KDP concentration. The lines are only guides for the eyes.

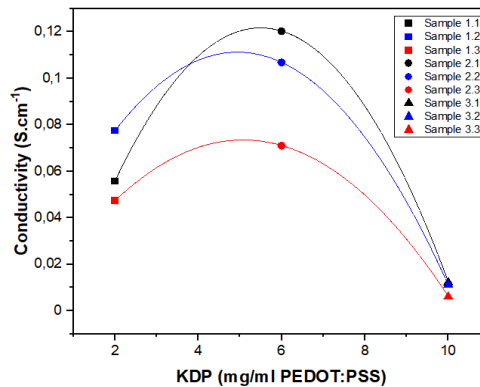


3.2 RESISTANCE MEASUREMENTS

The sheet resistance of the samples was determined using a conventional multimeter. The measurement tips were positioned to guarantee sections of the same area in the order of 1 cm². Ten measurements were taken for each sample and the average values obtained, see Table 1. It should be noted that, in general, the higher the concentration of KDP in the film, the greater its resistance. For example, sample 1.1 has a resistance of 12.3 kΩ / cm² and sample 3.1 has a resistance of 37.8 kΩ / cm². Samples made on PET / ITO substrate showed the lowest resistance. This is due to the fact that the films deposited on the PET / ITO substrate are more uniform / homogeneous and, possibly, have a less rough interface. In addition, the composite film shows greater adhesion to the PET / ITO substrate

Figure 4 shows the values of the conductivity of the samples on PET / ITO substrate as a function of the KDP concentration. The conductivity of films with 6 mg of KDP is higher than for samples 1 and 3. This result is in line with that observed in Section 3.1, which showed sample 2 with less thickness.

Figure 4: Conductivity of thick films of the PEDOT: PSS / KDP composite as a function of the KDP concentration for all samples. The lines are only guides for the eyes.



3.3 PERFORMANCE TEST SYSTEM

Figure 5 shows the measurement system used for the performance test. It is a home-made system consisting of a styrofoam box, to thermally and physically isolate the test environment. The sample is held by a clamp with two metal contacts. At the bottom of the box is graph paper that serves to measure the displacement of the sample. During measurement, the box is closed but there are holes for measuring temperature and also for capturing images. Externally, there is a protoboard where the DC source is connected. The displacement/movement was recorded with a cell phone camera. The precision of the measurements and the values obtained are of the order of 0.025 mm and it was obtained through the measurements in the own captured images (each real 1 mm measured in the image corresponds to 40 mm measured through the image with a conventional ruler).

Figure 5: System for sample displacement measurements due to the actuator effect.

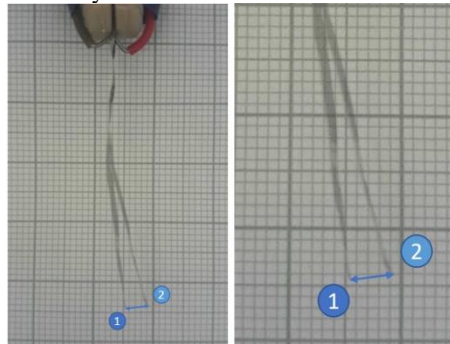


For all types of substrates, it was verified that the displacement of the samples was caused by the presence of KDP. Three displacement measures were taken for each sample and it was found that the values are the same, confirming the reproducibility of the effect. Samples that were manufactured only with PEDOT: PSS did not show any displacement

when subjected to voltages from 1 to 6 V. The temperature during the measurements was monitored by a laser thermometer and the maximum variations reached 0.1°C , insufficient variation to cause the displacement in the sample.

Figure 6 shows the observed displacement for sample 5.1. The images of the sample were superimposed without applying voltage (position 1) and with an applied voltage of 6 V (position 2). When the voltage is removed, the sample returns to position 1.

Figure 6: Superposition of two photographs showing state 1 with no voltage applied and state 2 with an applied voltage of 6V. See supplementary video.



Although PET / ITO samples showed the lowest resistance, the measured displacements were smaller than those obtained for samples with polyester and aluminium substrates. This is because the PET / ITO substrate is thicker and heavier than the other two. The samples with polyester and aluminium substrate showed displacements up to ten times greater, as can be seen in Table 2. The effect of the amount of KDP in the composite can also be seen in Table 2. For sample 1.1, which contains 2 mg of KDP, the displacement for an applied voltage of 6 V (applied for 60 s) is 0.250 mm. For sample 2.1, containing 6 mg, the displacement was 0.575 mm. However, when the amount of KDP was increased to 10 mg (as in sample 3.1), the displacement decreased again and was only 0.075 mm. This suggests that there must be an optimal concentration of KDP that should be between these values. Not so low that it is not possible to observe the effects of displacement and not too high so that salt does not hinder the conduction of loads.

Voltages of 1 to 6 V were applied to the samples for 60 s. During this time the sample position was photographed every 10 s. With an image editing program, the sample displacement positions were collected from the beginning to the end of the voltage application. Table II shows the relationship between the applied voltage (V) and the displacement (mm) for the different samples at times of 10 s and 60 s. It can be seen that, with the exception of sample 3.3, all of the others presented displacement when subjected

to voltages of 1 and 6 V. Despite having the highest resistance, sample 5.1 presented a displacement of 5.000 mm. Certainly, the low weight and the smaller thickness of the aluminium substrate were responsible for this better performance.

Table II. Displacement values obtained for PEDOT:PSS/KDP thick film samples on different substrates. Applied voltage of 1 V and 6 V for 10 s and 60 s.

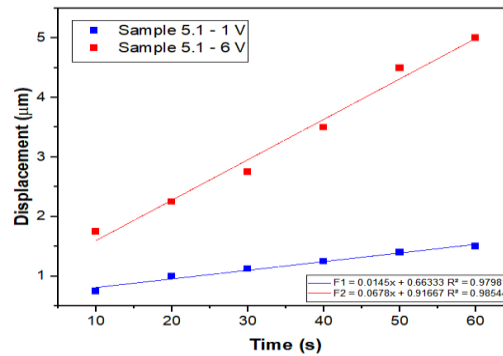
Sample	Voltage (V)	Displacement t = 10 s (mm)	Displacement t = 60 s (mm)
1.1	1.0	0.025	0.175
1.1	6.0	0.050	0.250
1.2	1.0	0.175	0.400
1.2	6.0	0.200	0.575
1.3	1.0	0.025	0.150
1.3	6.0	0.025	0.200
2.1	1.0	0.050	0.225
2.1	6.0	0.075	0.275
2.2	1.0	0.125	0.425
2.2	6.0	0.200	0.600
2.3	1.0	0.025	0.200
2.3	6.0	0.025	0.250
3.1	1.0	0.000	0.075
3.1	6.0	0.025	0.125
3.2	1.0	0.025	0.125
3.2	6.0	0.050	0.275
3.3	1.0	0.000	0.050
3.3	6.0	0.000	0.100
4.1	1.0	0.050	0.200
4.1	6.0	0.175	0.525
4.2	1.0	0.100	0.425
4.2	6.0	0.125	0.850
4.3	1.0	0.100	0.600
4.3	6.0	0.500	2.000
5.1	1.0	0.750	1.500
5.1	6.0	1.750	5.000

Figure 7 shows displacement versus time for the voltage application to sample 5.1 (for the voltages of 1 V and 6 V). For higher voltages, the displacement was greater. It can also be observed that, for a voltage of 1 V, the displacement tends to saturation while, for a voltage of 6 V, the displacement presents almost linear growth. Linear regression of the data reported in Figure 7 show a good linearity with $R^2 = 0.98544$, for the line voltage of 6 V, and $R^2 = 0.97981$, for the line voltage of 1 V.

Figure 7: displacement of sample 5.1 (composite film with 7.5 mg of KDP on aluminium substrate) when subjected to voltages of 1 V and 6 V in the time interval of 10 to 60 s.

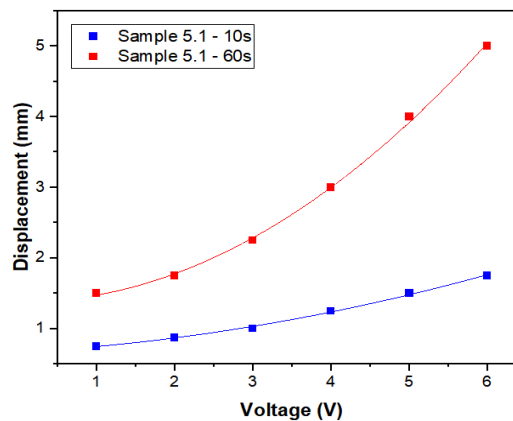
Figure 7: displacement of sample 5.1 (composite film with 7.5 mg of KDP on aluminium substrate) when subjected to voltages of 1 V and 6 V in the time interval of 10 to 60 s.

Figure 7: displacement of sample 5.1 (composite film with 7.5 mg of KDP on aluminium substrate) when subjected to voltages of 1 V and 6 V in the time interval of 10 to 60 s.



When we observe the variation of the displacement as a function of the voltage applied to sample 5.1, both during 10,s and for 60,s, we observe non-linear behaviour, as shown in Figure 8. The polynomial adjustment resulted in the coefficients R2 = 0.99744 for the 10 s curve and R2=0.99880 for the 60 s curve.

Figure 8: Displacement of sample 5.1 (composite film with 7.5 mg of KDP on aluminium substrate) as a function of the voltage applied during the 10 to 60 s time interval.



For sample 5.1, with a length $L = 30\text{mm}$, a strain (ϵ) of approximately 2.6% was calculated, using the equation (1) [21]:

$$\epsilon(x) = 2 \frac{[h(L-x)^2]}{L^4} y_{tip} \quad (1)$$

where the value of x is equal to zero at the point where the actuator is fixed, h is the thickness of the actuator and y_{tip} is the displacement of the actuator. This value is in good agreement with that obtained for polymeric films with thicknesses between 70 and 100 μm , reported in the literature [3].

Table III summarises the displacement values measured for the three substrates with applied voltages of 1 and 6 V. It was observed that the samples with the thick films deposited on the polyester and aluminium substrates present greater displacement: 2 mm for polyester and 5 mm for aluminium.

Table III. Comparative summary of the displacement values of the samples with thick PEDOT:PSS films

Substrate	Voltage (V)	Maximum displacement (mm)
ITO-PET	1.0	0.425
ITO-PET	6.0	0.600
Polyester	1.0	0.600
Polyester	6.0	2.000
Aluminium	1.0	1.500
Aluminium	6.0	5.000

It was observed that the actuator effect is monopolar for all studied samples, i.e. it is independent of the polarity of the applied voltage. The response is considered slow because the time to move is approximately 10 s. The required voltage is low. The samples sustain displacement under the applied DC voltage and all samples operate under ambient conditions. The PEDOT: PSS / KDP composite is easy to produce and highly reproducible. The results obtained in this work are compared to the work of references [3] and [20]. The findings in [3] show a displacement / inclination of the order of 10 mm for the voltage of 1 V, for a sample with a thickness of approximately 100 μm after being immersed in an ionic liquid solution. The results presented in this work are relevant, since the thick film actuators of the PEDOT: PSS / KDP composite operate in a non-aqueous environment.

4 CONCLUSION

For all types of substrate, it was observed that the movement was caused by the presence of KDP. Samples made only with PEDOT: PSS showed no displacement. In addition, the temperature of the samples was monitored using a laser thermometer and the maximum temperature variations reached 0.1°C. This reinforces the conclusion that the KDP is responsible for the displacement. In this work, actuators were developed using three types of substrate, whose performance responses were of the order of millimetres. For the

voltage level applied, the results are satisfactory and for a fast action of up to 10 seconds, they are capable of producing noticeable displacements.

The three actuator models show that the displacement of the actuator is proportional to the salt concentration up to an optimal concentration of 7.5 mg KDP for each 1 mL of PEDOT: PSS. It was observed that there is a linear relationship between the voltage application time and the displacement. On the other hand, the relationship between displacement and applied voltage is not linear. It was verified that the displacement is always in the same direction and does not depend on the applied voltage signal. The weight of the substrate strongly influences the response.

Organic actuators can be manufactured using simple manufacturing methods and without the need to use materials that are toxic or difficult to access. Finally, it can be concluded that the PEDOT: PSS / KDP mixture can comprise the active layer of an organic actuator and that, using an aluminium foil substrate, there can be displacements of up to 5 mm using voltage levels of the order of 6 V. Research with the aim of making thick films of the PEDOT: PSS / KDP composite without the need for a substrate for mechanical support is ongoing.

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The authors declare that they have no conflict of interest.

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