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CENTRO DE CIÊNCIAS EXATAS E DE TECNOLOGIA

DEPARTAMENTO DE ENGENHARIA DE MATERIAIS

IPMC ELECTROMECHANICAL BEHAVIOR USING REDUCED PLATINUM LAYER AIMING GREEN ECONOMY

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IPMC ELECTROMECHANICAL BEHAVIOR USING REDUCED PLATINUM LAYER AIMING GREEN ECONOMY

Trabalho de conclusão de curso apresentado ao Departamento de Engenharia de Materiais da Universidade Federal de São Carlos, como requisito para obtenção do título de bacharel em Engenharia de Materiais.

Orientador: Prof. Dr Carlos Henrique Scuracchio

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DEDICATÓRIA

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Boston, ensinou-me que tudo é possível.

“Keep Hungry. Keep foolish”

Steve Jobs

RESUMO

Compositos de Polímero-Metal Ionoméricos (IPMCs) são materiais inteligentes formados por uma membrana polimérica eletroativa revestida em ambos os lados por eletrodos de metais nobres. Quando um estímulo elétrico é aplicado, um campo elétrico é gerado entre os eletrodos, e íons solvatados migram dentro da membrana polimérica, causando um gradiente de pressão interna e dobrando o dispositivo. Apesar das inúmeras vantagens, o uso de metais nobres, como ouro e platina, é extremamente caro. Além disso, o processo de deposição gera uma grande quantidade de resíduos tóxicos. Portanto, neste trabalho, foi preparado um IPMC à base de Nafion® com uma camada reduzida de platina (PR-IPMC) e testado eletromecanicamente, comparando-o com um IPMC de referência (R-IPMC). As análises foram realizadas sob controle preciso de umidade relativa (RH). A força de bloqueio e a resposta de corrente foram usadas para avaliar seu desempenho, e a morfologia da camada de Pt foi avaliada por Microscopia Eletrônica de Varredura (MEV). O PR-IPMC apresentou uma camada de eletrodo 60% mais fina. Os dispositivos tiveram melhor desempenho quando RH = 90%. No entanto, apresentaram apenas 40% menos força de bloqueio em comparação com o R-IPMC. Além disso, a quantidade reduzida de complexo de platina usada para obter o dispositivo representa uma economia de reagentes e menor geração de resíduos, com desempenho mecânico satisfatório.

Keywords: IPMC. Blocking Force. Platinum Layer. Waste Generation. Performance.

ABSTRACT

Ionomeric Polymer-Metal Composites (IPMCs) are smart materials formed by an electroactive polymer membrane coated on both sides by noble metal electrodes. When an electrical stimulus is applied, an electric field is generated between electrodes, and solvated ions migrate inside the polymeric membrane, causing an internal pressure gradient and bending the device. Despite numerous advantages, it is exceedingly expensive to use noble metals, such as gold and platinum. In addition, the deposition process generates a large amount of toxic waste. Thus, in this work, a Nafion®-based IPMC with a reduced platinum layer (PR-IPMC) was prepared and electromechanically tested, comparing it with a reference IPMC (R-IPMC). The analyses were performed under precise relative humidity (RH) control. Blocking force and current response were used to evaluate its performance, and the morphology of the Pt layer was evaluated through Scanning Electron Microscopy (SEM). The PR-IPMC presented a 60% thinner electrode layer. The devices performed better when RH = 90%. However, just 40% less blocking force compared to R-IPMC. Besides, the reduced amount of platinum complex used to obtain the device represents a saving of reactants and less waste generation, with satisfactory mechanical performance.

Keywords: IPMC. Blocking Force. Platinum Layer. Waste Generation. Performance.

FIGURE LIST

TABLE LIST

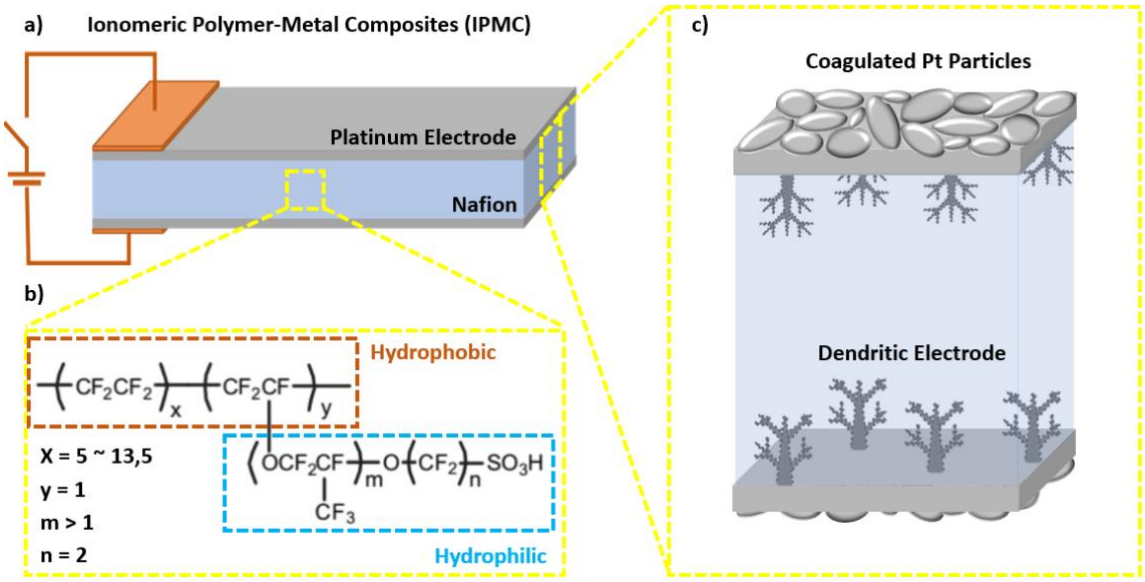
SUMÁRIO

1. INTRODUCTION

For several decades, it has been reported as a special class of materials, the *smart materials* (TANI; TAKAGI; GIU, 1998), due to its ability to modify one or more characteristics when submitted to an external stimulus, such as stress, moisture, electric or magnetic field. Among the most relevant smart materials are the electroactive polymers (EAPs). Some devices based on EAPSs can change their shapes or dimensions in response to external electrical stimuli. They are divided into two main categories related to the polymeric phase activation mechanism, which can be ionic (iEAP) or electronic (eEAP) (BAR-COHEN *et al,* 2015; BARBOSA *et al.,* 2022). The most common iEAP are Ionomeric Polymer-Metal Composites (IPMCs) (BAR-COHEN *et al,* 2000).

IPMCs are smart materials with a metal/polymer/metal sandwich-like structure (Figure 1a), so they can also be considered both a material and a device. These materials can perform bending movements in response to an external electrical stimulus and vice versa (SHAHINPOOR, 2015). Furthermore, IPMCs also have low density, flexibility, biocompatibility, and low driven voltage (< 5V) (KAASIK *et al.,* 2017). For this reason, they are promising materials for a wide range of applications, such as actuators (BHATTACHARYA; BEPARI; BHAUMIK, 2019; LI *et al.,* 2022), sensors (DAS *et al.*, 2022; MOHDISA; HUNT; HOSSEINNIA, 2019), artificial muscles (HE *et al.*, 2019; MA *et al.,* 2020) and robotics (ZHANG *et al.*, 2019; JAIN *et al.*, 2019). Due to its excellent mechanical resistance, high proton conductivity and thermochemical stability, Nafion® is the most used iEAP for IPMCs development (GONÇALVES *et al.*, 2020). Its chemical structure is formed by the main chain, poly (tetrafluoroethylene), with sulfonated side chain groups (Figure 1b).

Figure 1 – Ionomeric Polymer-Metal Composites schematic representation. a) IPMC clamped in a cantilever configuration by electrical contacts. b) Nafion® chemical structure. c) Platinum electrode



Source: Author.

The ionic polymer amphiphilic characteristic, where the sulfonated groups form the hydrophilic part, and the backbone is hydrophobic, generates the nano-scale phase separation. The first is structured in inverted micelles composed of charged groups, in which the main chain is outside of the micelle, and the side chains are shifted inward, while the latter has the structure of a semicrystalline polymer (MAURITZ; MOORE, 2004). The system's neutrality is guaranteed by mobile cations (counterions) that are exchanged with saline solutions or through air moisture. This structure is responsible for the ionic conductivity of ionomers with charged side chains, where large hydrophilic channels formed by the coalescence of the inverted micelles can absorb a large amount of water, which solvates the ions inside the polymeric matrix (ELLIOT *et al.*, 2011).

This device's operation mechanism consists of hydrated ions migration inside the polymer's ionomeric channels in response to an electric field generated after applying a potential difference in the metallic electrodes (PARK *et al.*, 2022). This ionic movement leads to internal pressure gradients, resulting in an anisotropic accumulation of mass, which causes the device to bend (LERONNI; BARDELLA, 2021). For this reason, its electromechanical performance depends on the operating environment (temperature and humidity) (LEI; LIM; TAN, 2015; MUST, 2014), type of

cation (KIKUCHI; TSUCHITANU, 2009; SACCARDO *et al.*, 2020; SACCARDO *et al.*, 2021), dimensions of the polymeric membrane (especially thickness), and physical properties of the electrode (PORFIRI, 2008).

Noble metal electrodes, such as Platinum (Pt) and Gold (Au), are commonly deposited on the Nafion® surface through an electroless process to form an IPMC. A Nafion®-based IPMC with platinum electrodes has excellent performance, such as large tip displacement, high blocking force and high capacitance (CHANG *et al.,* 2018). Kim and Shahinpoor (2002; 2003) described the metallic electrodes as the junction of coagulated metallic particles, which are found on the membrane's surface and in the layer just below the surface, penetrating the membrane by diffusion mechanisms (Figure 1c). These layers were called outer electrodes or metallic electrodes, where there is only the metallic film, and inner electrodes, or composite electrodes, where there is a mixture between the polymer and the reduced metal, also known as the blocking interface (Figure 1c).

The electrode characteristics such as porosity, thickness and surface roughness directly influence the IPMC performance. According to Kim *et al.* (2007), these properties can be controlled by monitoring process conditions such as reactant concentration, reaction time and the number of depositions. Although widely used, the metal electrode has some disadvantages, mainly concerning the high cost and toxic waste generation during the preparation process, but it's still a challenge to develop electrodes as good as the metallic ones (PUNNING; KRUUSMAA; AABLOO, 2007; NAKABO *et al.*, 2005; PALMRE *et al.*, 2009). Therefore, this paper aims to study the electromechanical performance of IPMC actuators over time with a reduced platinum layer, seeking a better understanding of its influence on the electromechanical properties of the IPMC.

2. THEORICAL FOUNDATION

The rapid advancement of material science has introduced many "smart" materials with new sensing and actuation capabilities. An example of these materials is EAPs, which are offering attractive alternatives to conventional sensors and actuators (HUNT *et al.,* 2016).

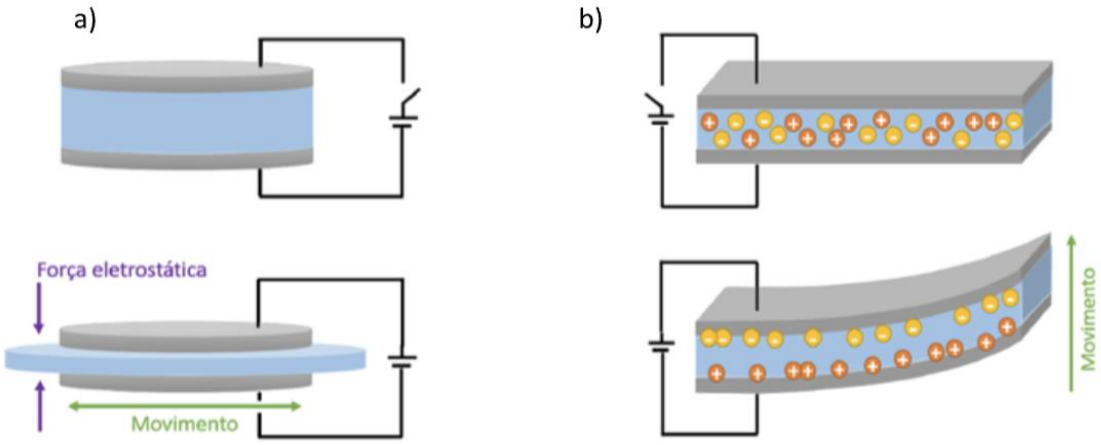
2.1. ELECTROACTIVE POLYMERS

EAPs are materials that can modify their shape and/or size in response to an electrical stimulus. They are typically found in the form of sandwich-type composites (electrode/polymer/electrode) (HIRANO, 2013). The polymer matrix has low density and flexibility, enhancing its use as soft actuators and sensors (NEMAT-NASSER; ZAMANI, 2003).

EAPs are classified according to the activation mechanism of the polymer matrix, which can be electronic or ionic. Electronic EAPs (eEAPs) are materials that, when a voltage difference is applied across the electrodes, they compress the polymer phase, resulting in flattening and lateral expansion, as illustrated in Figure 2a.

In contrast, there are ionic EAPs (iEAPs), materials that involve the mobility or diffusion of ions. Their deformation mechanism is based on the movement of ions and solvent molecules contained within the polymer matrix. When a voltage difference is applied across the electrodes (a few volts), the ions, along with solvent molecules, migrate in response to the electric field (BAR-COHEN, 2004). A common example of iEAPs is Ionomeric Polymer-Metal Composites (IPMCs), illustrated in Figure 2b.

Figure 2 – Schematic representation of electroactive polymers: a) Electronic; b) Ionic



2.2. IONOMERIC POLYMER-METAL COMPOSITES

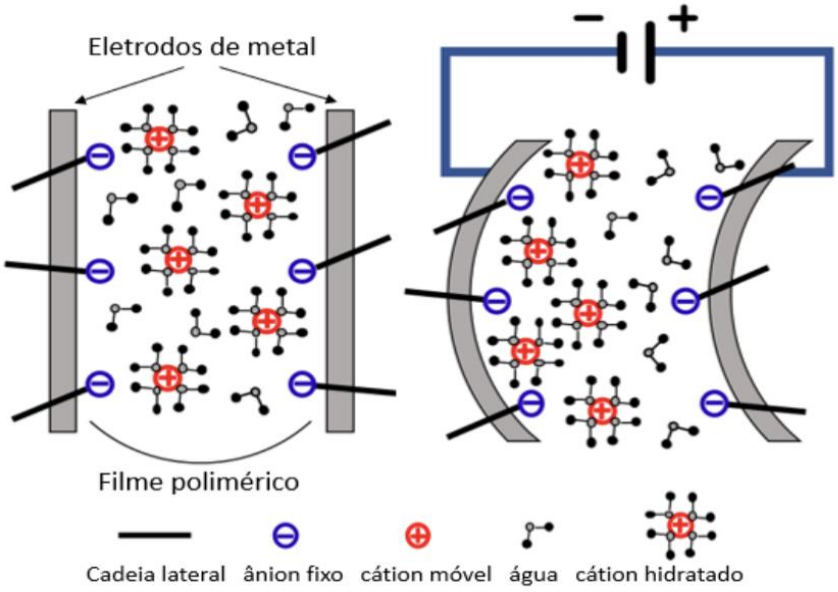
IPMCs are multifunctional smart materials with actuation, energy harvesting, and sensing capabilities. These materials were first introduced in 1997-1998 by Shahinpoor, Bar-Cohen, and collaborators, and were classified as members of the electroactive polymer family. This classification resulted from a research project supported by NASA's Jet Propulsion Laboratory (JPL). However, the original idea of ionic polymers and gel polymer actuators dates to the early 1990s (SHAHINPOOR, 2015).

In 1993, the first two patents on IPMCs were granted to Japanese researchers, Adolf *et al.* (1993), and Oguro, Takenaka, and Kawami (1993). These patents were followed by others related to both actuation and sensing, as well as numerous studies on development and applications (SHAHINPOOR, 2015).

The method traditionally used to fabricate these composites involves the primary reduction of a metal salt, typically using sodium borohydride or hydrazine. After the reduction process, the metal, usually silver or gold, chemically bonds to the surface of the polymer film, forming an IPMC (NAJI; SAFARI; MOAVEN, 2016). Noble metals are used due to their high resistance to corrosion.

The most accepted actuation mechanism is ion migration, where ions, which may or may not be solvated in water, migrate under the application of an electric field on the faces of a polymer film (HIRANO *et al.*, 2011). This migration generates a pressure gradient that causes the film to bend, as schematized in Figure 3.

Figure 3 – Schematic representation showing the folding mechanism of an IPMC based on Nafion®



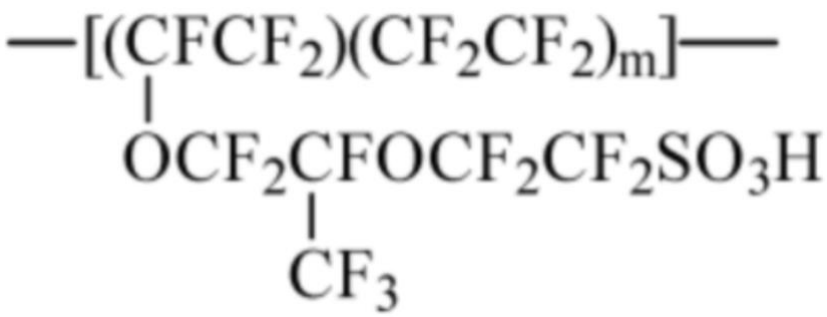
Source: Adapted from Chen (2008).

The most used polymer for obtaining these actuators is Nafion®. Basically, the chemical structure of Nafion® is divided into two parts: one hydrophobic and the other hydrophilic. The hydrophobic part is completely nonpolar. The hydrophilic part, on the other hand, is polar and thus absorbs a large amount of water (HIRANO, 2013). This amount will depend on atmospheric conditions, especially the relative humidity. Since a greater amount of water causes greater swelling of the ionomeric channels, many H_2O molecules will be carried along with the cation during its movement (MAURITZ; MOORE, 2004).

2.3. NAFION®

Nafion® ionomers were developed and are produced by the American company DuPont (MAURITZ; MOORE, 2004). This material is created by the copolymerization of a perfluorinated vinyl ether comonomer with tetrafluoroethylene (TFE), resulting in the chemical structure shown in Figure 4. As can be seen, Nafion® is composed of a main chain like poly(tetrafluoroethylene), PTFE, with sulfonated groups grafted laterally to the chain. It is a polyanion where the counter-ion can be H^+, Na^+, or another cationic species (HIRANO, 2013).

Figure 4 – Schematic representation of the chemical structure of Nafion®, showing H+ with the counter-ion



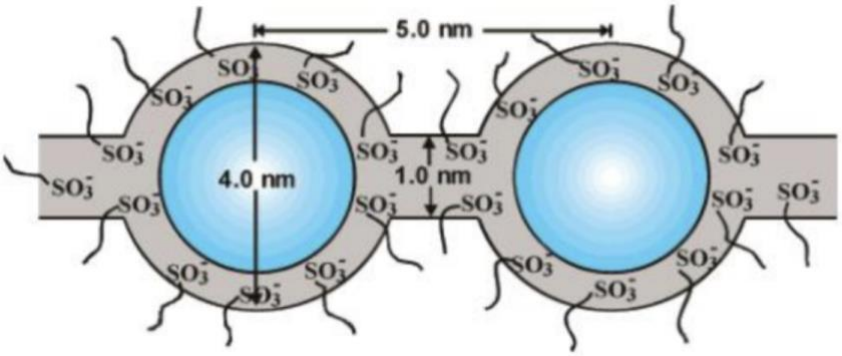
Source: Mauritz and Moore (2004).

The main chain forms the hydrophobic phase of Nafion®, which is amorphous with small crystalline domains. The mechanical, thermal, and chemical stability of Nafion® is strongly related to the degree of crystallinity and the morphology of these crystallites. Due to the difference in polarity in the main chain, the sulfonic groups assume an inverted micelle structure, where the ionic part of the polymer remains confined. Within these micelles, mobile cations (counter-ions) ensure the neutrality of the system. A sufficient amount of sulfonated groups guarantees a coalescence of these micelles into channels that extend three-dimensionally (MAURITZ; MOORE, 2004).

Some studies in the literature have already highlighted the importance of and attempted to explain the mechanism of water absorption, relating it to the material's structure. Many models have been developed over the years, with one notable example being the ionic cluster network model by Gierke *et al.* (1983). The model assumes that there are cylindrical clusters approximately 40 Å in diameter formed by perfluoroalkyl ether groups with sulfonated terminations, organized as inverted micelles and arranged in a network.

The size of the clusters can vary according to the degree of hydration of the film. These micelles are connected by pores or cylindrical channels approximately 10 Å in size (HSU; GIERKE,1983). These SO_3^--lined channels were used to explain the passage of positively charged species between the clusters, while negatively charged species were rejected. Figure 5 represents the ionic cluster network model.

Figure 5 – Ionic cluster model for hydrated Nafion®



Source: Hsu and Gierke (1983).

Other models that studied the morphology of Nafion® should also be mentioned due to their importance. Among them are the modified Core-Shell Model proposed by Fujimura *et al.* (1982), the Local-Order Model proposed by Dreyfus *et al.* (1990), the Lamellar Model proposed by Litt [20], the Sandwich Model proposed by Haubold *et al.* (2001), and the Rod-Like Model proposed by Rubatat *et al.* (2002).

All these models share the common understanding that ionic groups aggregate within the polymer matrix to form a network of clusters that allow significant swelling by polar solvents and efficient ion transport through these nanoscale domains. However, they differ significantly in terms of the geometry and spatial distribution of the ionic clusters. The aforementioned models describe the relationship between the morphology of Nafion® and water absorption. However, they do not describe the relationship between the presence of different counter-ions and water absorption.

This relationship has been studied by only a few authors. Shahinpoor and Kim (2000) evaluated the influence of different counter-ions (H^+, Li^+, Na^+, K^+, Ca^{2+}) in mechanical force tests by actuating the material with 1.2V. However, the study did not consider the influence of the degree of hydration on the mechanical behavior of the actuator, as the tests were conducted under constant humidity conditions.

On the other hand, Hirano *et al.* (2011) measured the effect of the degree of hydration on the electromechanical response of IPMC samples conditioned with H^+, recording the force exerted by the IPMC after applying 1V, with humidity varying from 20% to 90%. The author found that the electromechanical response differs for each humidity level, and the measured force increases with the degree of hydration. A similar study was conducted by Lee *et al.* (2006), who investigated the electrolysis of the film in operation, combining different cations and solvents to find the best

combination of both in force tests.

As can be observed, there are studies that seek to understand the relationship between water absorption and electromechanical behavior, as well as the use of different counter-ions in the electromechanical behavior of IPMCs. However, a detailed study encompassing the relationship between water absorption, the use of different counter-ions, and electromechanical behavior has not yet been conducted. Furthermore, many of the water absorption analyses consist of measuring the mass of dry samples before and after immersion in H_2O, without considering the kinetics of absorption or the time required to reach osmotic equilibrium with the external environment.

3. MATERIALS AND METHODS

3.1. REACTANTS

Nafion® 117 membranes were obtained from IonPower. Hydrochloric acid (HCl) (Sigma-Aldrich Corporation® analytical grade, used as received), tetraammineplatinum chloride (Pt(NH3)4Cl2) – 98%, hydrogen peroxide (H_2O_2), and sodium borohydride (NaBH4) – 90% from Sigma-Aldrich Corporation® were used as precursors for the cleaning, ion diffusion, and reduction processes, respectively.

3.2. IPMC SAMPLES PREPARATION

Before the electroless plating, the membrane was cut, sized (35 x 5 mm), and cleaned by the following process: the membranes were boiled twice in H_2O_2 5% (v/v) solution for 30 min, then rinsed and boiled in 2 mol L^{-1} HCl for 30 min. Finally, they were rinsed and boiled for 30 minutes in ultrapure water, and the electroless plating was performed. Milli-Q deionized water (DW) was used all over these steps.

Two samples (the reference IPMC (R-IPMC) and the platinum reduced IPMC (PR-IPMC)) were prepared following the Oguro's procedure (OGURO, s.d.). For the R-IPMC, the membrane was immersed in a $[Pt(NH_3)_4]Cl_2$ water solution with a concentration of 5,24 mg of $[Pt(NH_3)_4]Cl_2$ per cm^2 of the membrane. Thus, 1 mL of NH_4OH 5% (v/v) was added for each 175 mg of the complex in the solution. The polymer was maintained in this solution for 12 hours, ensuring the Pt^{2+} ions absorption. After this period, the membrane was removed, rinsed, and immersed in DW at 60 °C. A $NaBH_4$ 5% (w/w) solution was added every 30 minutes four times in small aliquots to reduce the Pt^{2+} ions into Pt^0 at the membrane surface. The total amount of solution used was 1 mL cm^{-2} of polymer. The PR-IPMC samples were prepared using the same method described before. However, using only 1,31 mg of $[Pt(NH_3)_4]Cl_2$ per cm^2 of the membrane (1/4 of the amount described before). The stoichiometry of the following steps was maintained by adjusting the amount of the reactants. The reactions involved in the deposition process are shown in Table 1.

Table 1 – Reactions involved in the electrode assembling process

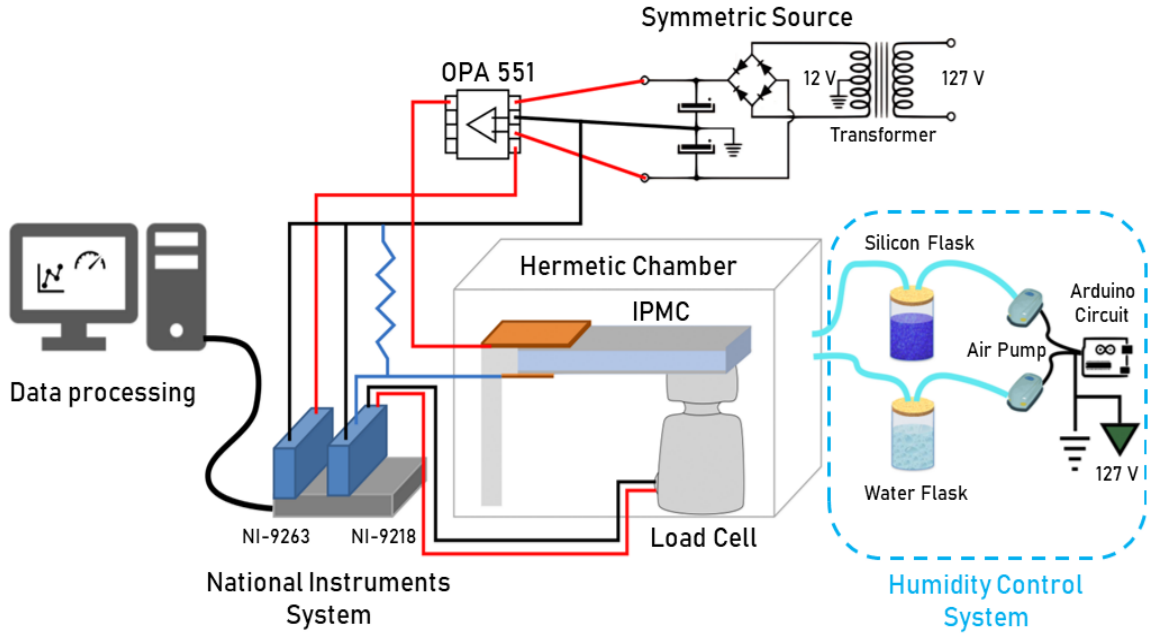
Reaction	Description
$[Pt(NH_3)_4(Cl)_2] + 2NH_4^+ + 2OH^- + 2H^+_{(in\ Nafion)} \rightarrow [Pt(NH_3)_6]^{+2}_{(in\ Nafion)} + 2H_2O + 2Cl^- + 2H^+$	Hydrolysis
$[Pt(NH_3)_6]^{+2}_{(in\ Nafion)} \rightarrow Pt^{2+}_{(in\ Nafion)} + 6NH_3$	Complex activation
$Pt^{+2}_{(in\ Nafion)} + 2NaBH_4 \rightarrow Pt^0 + H_2 + B_2H_6 + 2Na^+_{(in\ Nafion)}$	Redox reaction
$B_2H_6 + 6H_2O \rightarrow 2B(OH)_3 + 6\ H_2$	Disproportionation
$[Pt(NH_3)_4(Cl)_2] + 2NH_4^+ + 2OH^- + 2H^+_{(in\ Nafion)} + 2NaBH_4^+ 4H_2O \rightarrow Pt^0 + 7H_2 + 2Na^+_{(in\ Nafion)} + 2B(OH)_3 + 2Cl^- + 2H^+ + 6NH_3$	Global reaction

Source: Author.

3.3. INSTRUMENTATION

As reported in our previous papers (GONÇALVES *et al.*, 2020; SACCARDO *et al.*, 2020; HIRANO *et al.*, 2015), an experimental apparatus was developed to control the relative humidity and measure the IPMC electromechanical response, as seen in Figure 6.

Figure 6 – Scheme of the electromechanical test system under a controlled environment used in this work



Source: Author.

3.4. CHARACTERIZATION

3.4.1. Scanning electron microscopy

Scanning electron microscopy (SEM) was carried out on an SEM microscope equipped with an energy dispersive spectroscopy (EDS) system to investigate the morphology of the R-IPMC and PR-IPMC. An FEI Inspect 50 instrument operating at 10 kV was used. The EDS analysis was carried out with an EDAX EDS system.

3.4.2. Blocking force

Inside the hermetic acrylic chamber described in the instrumentation section, the IPMC sample was clamped by copper electrodes in a cantilever beam configuration. After chamber closing, its humidity was kept for 6 hours (for RH = 30 % and RH = 90 %) to ensure sample osmotic equilibrium with the environment's RH. After that period, a 2.75 voltage was applied, and the blocking force applied to the load cell was acquired using the NI-9218 data acquisition module.

3.4.3. Electromechanical response

After the period to ensure the osmotic equilibrium, a 5.5 V square wave amplitude was applied for 32 s, and the current response was acquired using a NI-9263 electrical signal generator module and a NI-9218 data acquisition module.

3.4.4. Thermogravimetric analysis

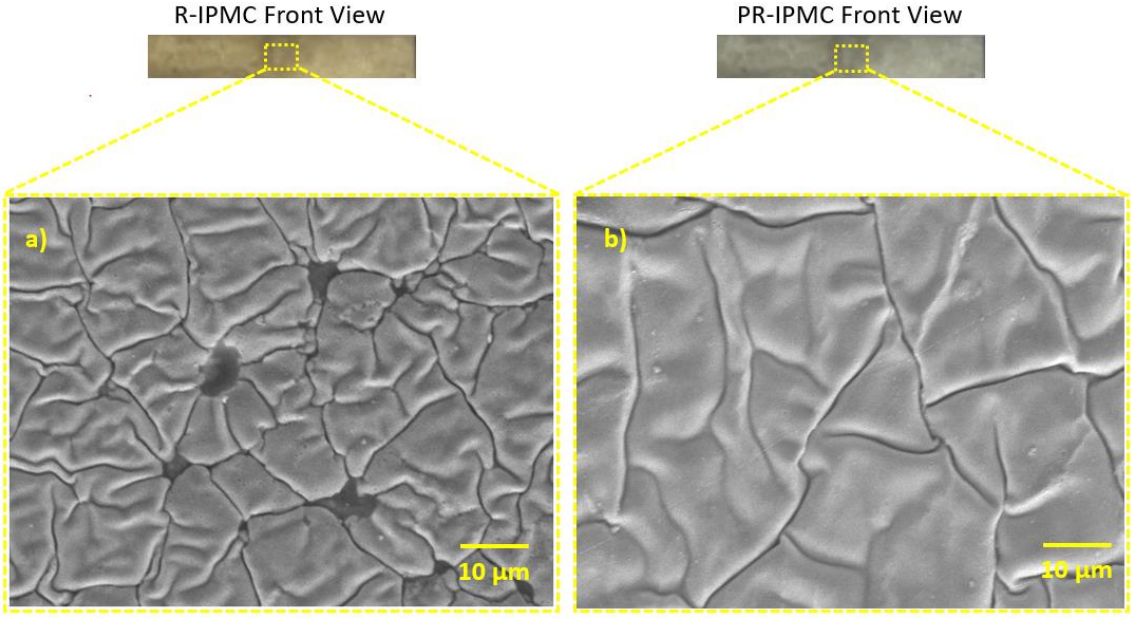
Thermogravimetry analysis (TGA) was used to investigate the heat degradation behavior of the R-IPMC and PR-IPMC and the amount of Pt that remained in each sample. Measurements were performed using a TA Instruments Q100 analyzer. Samples (ca. 15 mg) were heated from 25°C to 650°C with a rate of 20°C min^{-1} under an N_2 atmosphere (50 mL min^{-1}). Both samples were put in the acrylic chamber of the electromechanical test at RH=90% to ensure the channel's hydration before the test.

4. RESULTS AND DISCUSSIONS

4.1. ELECTRODE MORPHOLOGY

The platinum electrodes' surface characteristics were investigated through Scanning Electron Microscopy (SEM) technique. The analyses were carried out on the surface and in the interface area with Nafion®, as shown in Figures 7 and 8.

Figure 7 – IPMC surface analyses by SEM. a) R-IPMC and b) PR-IPMC



Source: Author.

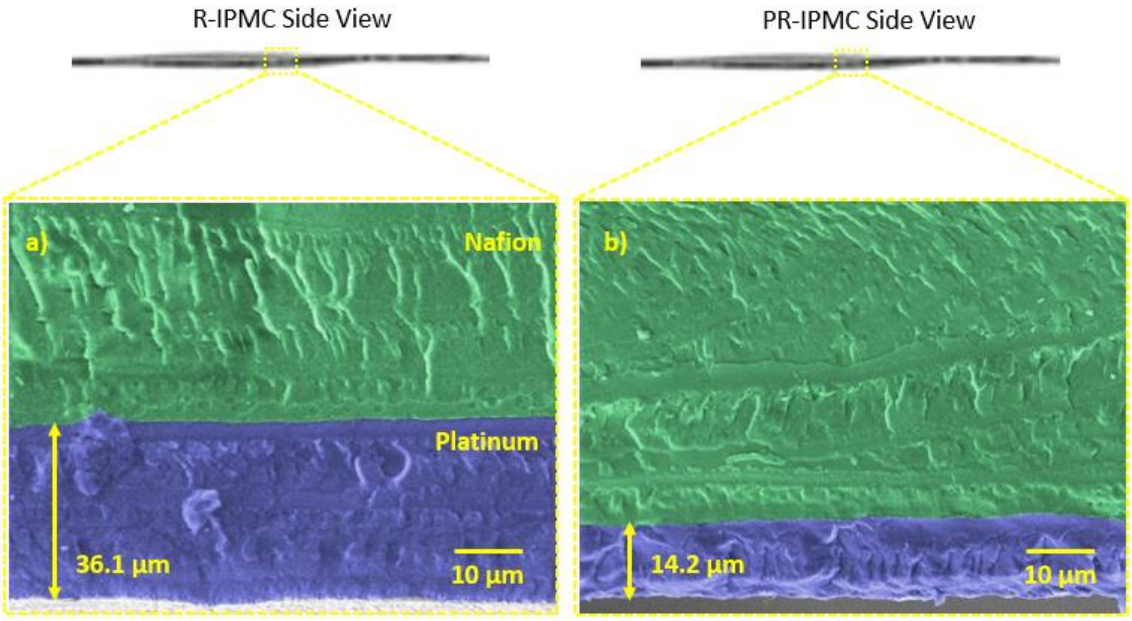
Figure 7 shows that agglomerates are spread evenly along the entire surface, forming platinum "islands". This phenomenon was already expected since the deposition method involves the metal salt reduction over the irregular polymer's surface, composed of several nanometric channels. However, the "islands" size differs; in the R-IPMC, it has five µm, and in the PR-IPMC, it has 20 µm. This difference occurs due to the amount of platinum adsorbed by the polymeric membrane, which, in greater quantity, results in a higher nucleation rate in the electrode region, resulting in a higher grains density, which prevents a sizeable individual growth front resulting in smaller grains in the R-IPMC electrode when compared to the PR-IPMC.

Platinum ions penetrate ionomeric channels that have distinct positions and sizes throughout Nafion®. During the reduction stage, the metallic platinum growth occurs in different regions simultaneously, resulting in a segmented structure, like platinum "islands". This problem intensifies with the deflection amplitude increase and the

number of cycles (LEE *et al.*, 2006). Still, cracks are generated along the grain boundaries under continuous bending cycles, further increasing resistivity (PARK *at al.*, 2010). As the resistance of electrodes increases, the electric field between them decreases. Therefore, the migration of cations and water molecules is slowed, diminishing the IPMC performance.

A key factor for the formation and performance of the electrode is the surface roughness presented by the ionomeric polymer membrane used. Nafion®, for example, is a membrane usually acquired in the form of thin films produced by extrusion or prepared by casting Nafion® in solution. These manufacturing processes generate smooth surfaces with an exceptionally low degree of roughness, which makes the adhesion of electrodes difficult depending on the method (HASANI *et al.*, 2019). The dendritic interfacial structure formed promotes mechanical and chemical adhesion. Therefore, for this assembly method, the surface state of the membrane is not predominant for the adhesion force. With this, the device's performance is directly affected, as the greater area between electrodes and membrane increases the capacitance of the IPMC due to the intensification of the electrical double layer (KIM; KIM, 2005; RASOULI; NAJI, 2017).

Figure 8 - IPMC lateral morphology analyzed by SEM. a) R-IPMC and b) PR-IPMC



Source: Author.

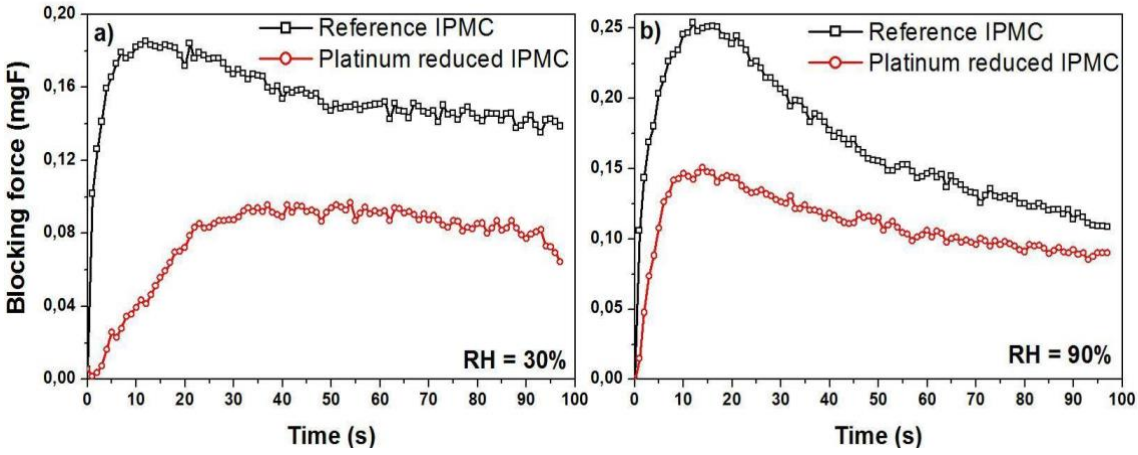
Figure 8 shows the IPMC side view SEM images. Sample cryogenic fracture was

performed with the cut perpendicular to the IPMC surface. The PR-IPMC electrode thickness is 14.2 µm, while the R-IPMC electrode is 36.1 µm, resulting in a 40% thickness by using a 25% of amminoplatinum complex. These data call attention to the fact that the amount of Pt^{2+} used during the impregnation does not linearly affect the thickness of the Pt^0 layer. In this way, it can be inferred that there is a limit to how much Pt^{2+} can be absorbed by the membrane, suggesting that this process can be further optimized.

4.2. BLOCKING FORCE

Figure 9 shows the reference IPMC (R-IPMC) and platinum reduced IPMC (PR-IPMC) blocking force *versus* time for different relative humidity conditions, dry (30%) and humid (90%) conditions, respectively, when a constant 2.75 V voltage was applied, respectively. This value was chosen because it is the lowest value that both samples have an appreciable electromechanical response when RH = 30%.

Figure 9 – Blocking force (mgF) versus Time (s) when a) RH = 30% and b) RH = 90%



Source: Author.

As observed, after applying the voltage, the blocking force increases until it reaches a maximum value and decreases as a time function. This expected behavior is known as back relaxation and will be discussed later. Besides, for both samples, the blocking force has increased with the increase in RH. This is due to the number of water molecules inside the device. When an electrical stimulus is applied to an IPMC, the charged species inside the ionomeric channels move towards the cathode,

generating a pressure gradient into the membrane, and resulting in the bending movement (ARUMUGAM; REDDY, 2018; LEE; HONG; KOO, 2014; JUNG; NAM; CHOI, 2003). The ionic migration occurs easier when more water molecules are present (LEE *et al.*, 2005), promoting a device with more intense movement. For this reason, the blocking force increases with RH.

Also, the R-IPMC sample demonstrated a more significant blocking force response than the PR-IPMC for both RH conditions. This effect shows that ionic migration also strongly depends on the electric field generated between the electrodes (GONÇALVES *et al.*, 2020). Since the R-IPMC has a thicker platinum layer, the electric field generated was more intense once the conductivity of this electrode was outstanding. For this reason, the ionic migration was more pronounced, and the R-IPMC sample showed a higher mechanical response than the PR-IPMC sample for all RH conditions tested. Despite reducing the amount of platinum used by 75%, the PR-IPMC blocking force was 40% less than the standard sample. Although it generates a smaller electric field, a thinner layer may exert less restriction on the device's movement.

About the back-relaxation, it occurs when H_2O molecules migrate in the opposite direction, causing the device "relaxation" (SHAHINPOOR, 2015; HIRANO *et al.*, 2015). For this reason, this mechanical response (blocking force) was observed for all RH. However, it was reduced for RH = 30% since the number of water molecules inside the ionomeric channels was small. Consequently, back relaxation can be mitigated by reducing the number of water molecules inside the ionomeric channels (SACCARDO *et al.*, 2020). However, it is essential to state that this phenomenon is not entirely understood, and there is no consensus about its origin.

Table 2 presents different blocking force values, such as peak blocking force (P-BF), BF after back relaxation (BF-BR) and relations for PR-IPMC and R-IPMC at RH = 30% and RH = 90%.

Table 2 – Different blocking force values, such as peak blocking force (P BF), BF after back relaxation (BF-BR), and relations for PR-IPMC and R-IPMC at RH = 30% and RH = 90%

RH (%)	IPMC	Peak Blocking Force (mgF)	Blocking Force after BR (mgF)	P BF PR-IPMC/R-IPMC Ratio (%)	BF-BR of PR-IPMC/R-IPMC Ratio (%)	Loss of Performance (%)
30	**PR-IPMC**	0,07142	0,05579	68,83	90,53	21,88
	R-IPMC	0,10375	0,06162			40,61
90	**PR-IPMC**	0,1506	0,09014	59,35	82,97	40,15
	R-IPMC	0,25374	0,10864			57,18

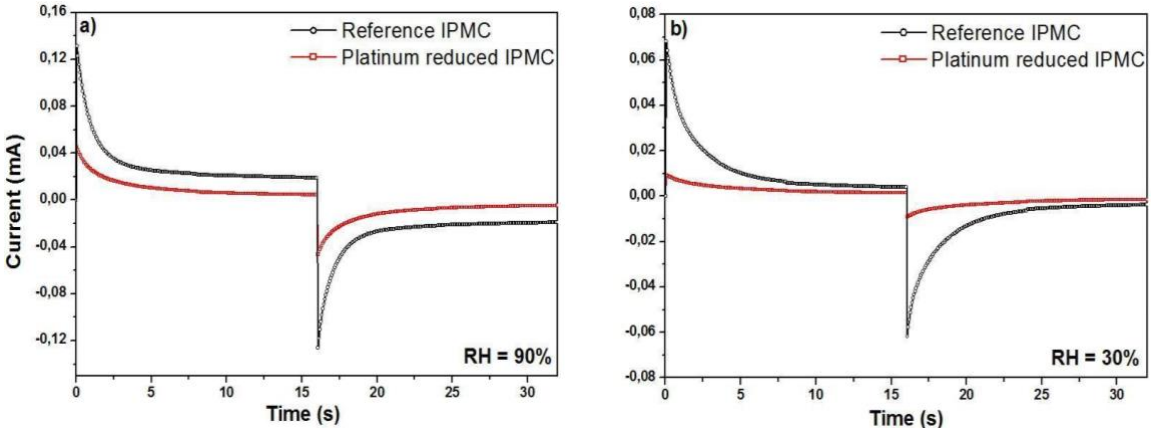
Source: Author.

Based on Table 2, it can be inferred that the higher the P BF, the more intense back relaxation, resulting in a performance loss over time. In addition, a thicker electrode could make the IPMC more rigid, restricting its movement and causing a significant performance reduction (YILMAZ *et al.*, 2019). Even though the R-IPMC has greater blocking forces at both RH, its performance loss is much more pronounced than the PR-IPMC. Despite its electrode being 2/5 of the thickness of the reference electrode, it demonstrates a blocking force of 90.53% of R-IPMC in RH=30% and 82.97% in RH=90%. Moreover, this suggests a saturation level for the platinum electrode; the thickest electrode had a more significant efficiency loss.

4.3. ELECTRIC PROPERTIES

Figure 10 presented the R-IPMC and the PR-IPMC current response versus RH when a square wave of 5.5 V amplitude was applied for 32 s.

Figure 10 - Current (mA) versus time when a) RH = 30% and b) RH=90%



Source: Author.

As observed, as soon as a bias was applied for both samples, the electric current that passes through the device reached its maximum value and decreased exponentially over time, regardless of the RH. When the polarity was reversed, the same was observed, however, in the opposite direction. This is the typical RC circuit behavior. As expected, the electrical sample response varies depending on the RH and the electrode thickness. It is observed that for RH = 90%, the maximum current intensity was obtained for both IPMCs.

Besides, its electrical performance is related to the water amount inside the ionomeric channels and the RH condition (GONG *et al.*, 2011; STOIMENOV; ROSSITER, 2006). Moreover, the R-IPMC electrical response was more intense than PR-IPMC. Ratifying that, a lower electric field is generated in the sample with the thicker electrode, which is related to the electrode conductivity. From Figure 6, it is also possible to obtain the total electrical charge stored, integrating the current as a function of time. When performing this procedure, integrating from t=0s to t=16s for both RH, the data shown in Table 3 were obtained.

Table 3 – Stored charge (mC) and the ratio for PR-IPMC and R-IPMC at RH = 30% and RH = 90%

RH (%)	IPMC	Stored charge (mC)	Stored charge R-IPMC/PR-IPMC Ration (%)
30	**PR-IPMC**	0,048	385,4
	R-IPMC	0,185	
90	**PR-IPMC**	0,16	287,5
	R-IPMC	0,46	

Source: Author.

It is also possible to observe that the accumulated electric charge (Q) during the test time is different for each sample, influenced by the RH variation and electrode thickness. When RH = 90%, more water molecules are present inside the channels, acting as lubricants to the ion's movement. So, the amount of electrical charge stored was more significant. In addition, the thicker the electrode, the greater the electric field generated, resulting in a greater electrostatic force acting on the ions, which makes them migrate more easily. Thus, a greater stored charge for R-IPMC is explained at RH=90%.

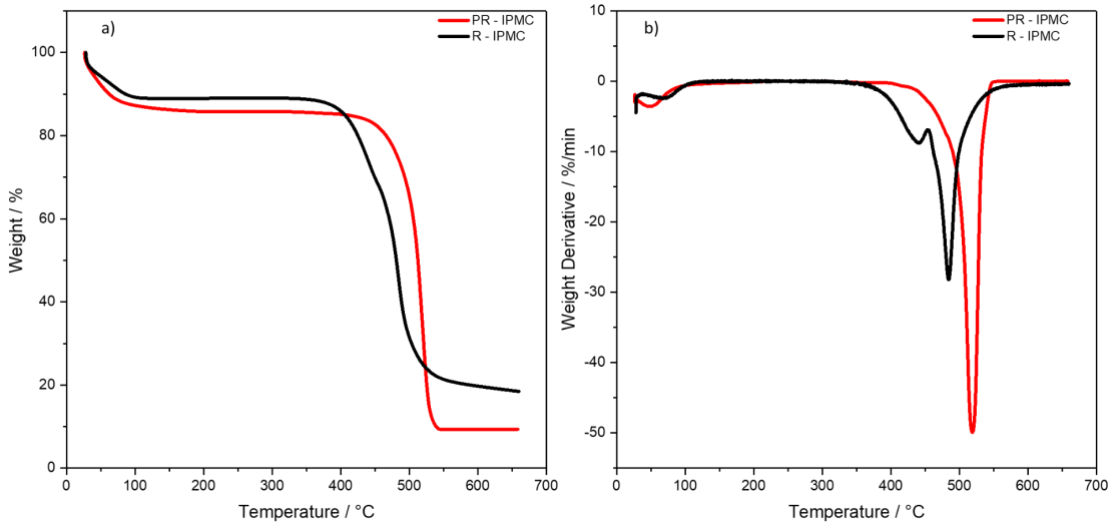
4.4. THERMOGRAVIMETRIC ANALYSIS

Figure 11 below shows the analyzed samples' weight loss (TG) and derivative weight loss (DTG). The data extracted from the graphs are summarized in Table 4. Interestingly, even though the materials for both samples are the same, the PR-IPMC had two significant weight loss events, while the R-IPMC had three. This may be a first indication that the thickness of the Pt layer has an influence not just on the electromechanical activity as a blocking interface but also play a role in the degradation of the samples.

The R-IPMC had the three common events of loss weight of Nafion® (DE ALMEIDA; KAWANO, 1999): the water loss in the range of 25°C - 105°C, the side chain degradation in the range of 340°C - 455°C, and finally the last one is due to the polymer backbone degradation in the range of 393°C - 493°C. The other sample, instead, had only two stages of mass loss, where the first stage was due to the loss of water, and the second one comprised both side chain and backbone degradation. If the second stage were an overlapped signal, the DTG would have been capable of

separating the events, but as it is possible to see, there is just one event after water loss for PR-IPMC. This could be directly related to the platinum layer as for the PR-IPMC, the side chain degradation was shifted to greater temperatures. To better comprehend this change, a deep study must be done. We will investigate it in our following paper because it's not the scope of this work. Finally, the PR-IPMC, as expected, had less mass of Pt since it was mounted with ¼ of the standard deposition amount. The amount of Pt can be attributed to the remaining mass in the thermogram, where the difference between the two samples is around 10% of the weight.

Figure 11 - a) TG of PR-IPMC and R-IPMC; b) DTG of PR-IPMC and R-IPMC.



Source: Author.

Table 4 – Thermal properties from TGA curves for PR-IPMC and R-IPMC

IPMC	Weight loss (%)	ΔT (°C)	T_{peak} (°C)
R-IPMC	11	30 - 105	70
	22	340 - 455	440
	46	455 - 563	485
PR-IPMC	13	30 - 105	48
	78	393 - 553	520

Source: Author.

5. CONCLUSIONS

A series of experiments was carried out to investigate the feasibility of developing new electromechanical actuators with electrodes reduced in the quantity of platinum to reduce both cost and environmental impacts. For this, two IPMCs were prepared, one standard with the amount of platinum salt suggested by the Oguro Method and the other with an amount four times lower. It has been proven that PR-IPMC, despite presenting a lower capacitance, demonstrates over time a performance similar to that of R-IPMC, of 90.53% in RH = 30%, in addition to a lower loss of performance after reaching the peak blocking force.

A thicker platinum electrode results in a greater intensity of the double electric layer phenomenon and, consequently, a more significant amount of stored charge. In addition, the higher the relative humidity level in the environment, the easier the ions will migrate inside the ionomeric channels, generating a greater electric field between the electrodes for higher humidity levels. However, the thickness of the electrode is related to the greater flexural rigidity of the IPMC, making it difficult to deform the device and facilitate the propagation of cracks.

This could explain why, due to the factors mentioned, there is a more significant loss of efficiency and a greater stored load for the R-IPMC at RH=90% while obtaining better performance, given the lower loss of efficiency, for the PR-IPMC in the same conditions. Therefore, it was possible to obtain new electromechanical actuators with a reduction in the cost of traditional IPMCs with platinum electrodes and an efficiency of 90.53% in low humidity levels (RH=30%) and 82.97% in wetter conditions (RH=90%). In addition, there was also a 75% reduction in environmental impacts, knowing that platinum is a heavy metal and only 25% of the amount used in the standard preparation was used.

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