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# Low resistance, highly corrugated structures based on poly(3,4-ethylenedioxythiophene) doped with a D-glucopyranoside-derived ionic liquid



Katarzyna Krukiewicz<sup>a,b,\*,1</sup>, Dominika Kobus<sup>c</sup>, Roman Turczyn<sup>b</sup>, Karol Erfurt<sup>d</sup>, Anna Chrobok<sup>d</sup>, Manus J.P. Biggs<sup>a</sup>

<sup>a</sup> Centre for Research in Medical Devices, National University of Ireland, Galway, Ireland

<sup>b</sup> Department of Physical Chemistry and Technology of Polymers, Silesian University of Technology, Gliwice, Poland

<sup>c</sup> Institute of Organic Chemistry, Polish Academy of Sciences, Warsaw, Poland

<sup>d</sup> Department of Chemical Organic Technology and Petrochemistry, Silesian University of Technology, Gliwice, Poland

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## ABSTRACT

Conjugated polymers have gained significant interest as highly conducting organic materials with versatile surface morphology. In this study, we demonstrate that the electrodeposition of poly(3,4-ethylenedioxythiophene), PEDOT, in the presence of a D-glucopyranoside-derived ionic liquid (IL) results in the formation of highly corrugated three-dimensional structures. The as-formed PEDOT/IL is found to outperform PEDOT electrodeposited in the presence of a conventional electrolyte (KCl) in terms of the low impedance at the biologically relevant frequency (1 kHz) and low charge transfer resistance. Consequently, it can be inferred that the unique surface morphology and beneficial electrochemical performance will facilitate the application of PEDOT/IL in biomedical engineering, especially in the field of neural interfaces and tissue scaffolds.

## 1. Introduction

Conducting polymers are versatile materials with a wide spectrum of applications, ranging from organic electronics to neural interfaces [1,2]. The robustness of this group of polymers is based on their high electrical conductivity, easiness of deposition on different types of substrates, flexibility, and versatile morphology [3]. Furthermore, these properties can be easily tailored through modulation of the polymerization conditions. Therefore, by changing the synthesis method, reaction medium, electrolyte solution or oxidation potential, it is possible to form conducting polymer derived materials tailored for specific needs. Since the properties of conducting polymers are strongly affected by their topography, current research is focused on the development of methods allowing to obtain morphology-controlled micro- or nano-structured conducting films [4].

Due to its substantial stability and high conductivity, poly(3,4-ethylenedioxythiophene), PEDOT, is currently one of the most widely used conducting polymers, with applications in energy conversion, sensing devices and bioelectronics [5]. Recently, Xu et al. [4] described an electrodeposition approach for the production of three dimensional

macroporous materials (3D-P-PEDOT), composed of micrometer sized craters and bumps and nanometer size fibrous structures, and their use as nitrite and ascorbic acid sensors. A 3D formulation of PEDOT functionalised with Cu<sub>x</sub>O was also used to serve as a sensitive hydrazine sensor [6]. Micro-structured conducting polymers coatings have gained significant interest in biomedical engineering, since macroporous PEDOT/poly(styrene sulfonate) (PEDOT/PSS) were successfully employed to increase the electrochemical surface area and the efficiency of recording and stimulation of neural electrodes [7]. Besides, PEDOT/PSS interfaces stabilised using a PEGylated crosslinker were found to yield improved conductivity and biocompatibility, resulting in a formation of more advanced biomaterials and promising scaffolds for tissue engineering [8]. In this communication, we present a facile electrodeposition technique for the fabrication of highly corrugated, structured polymer films based on PEDOT doped or co-doped with a (2-D-glucopyranosyloxyethyl)trimethylammonium bistriflimide ionic liquid (IL). The use of D-glucose as the ionic liquid precursor presents IL as a green electrolyte, since it is formed from a renewable compound, possesses lower toxicity and undergoes biodegradation [9]. Moreover, it can be inferred that the presence of D-glucose will enhance the

\* Corresponding author at: Centre for Research in Medical Devices, National University of Ireland, Galway, Ireland.

E-mail address: [katarzyna.krukiewicz@nuigalway.ie](mailto:katarzyna.krukiewicz@nuigalway.ie) (K. Krukiewicz).

<sup>1</sup> ISE member.

biocompatibility of the ionic liquid, and, therefore, also the biocompatibility of the conducting polymer formed in its presence. Previously, ionic liquids were observed to promote the electrodeposition of conducting polymers with a rough surface morphology [10] and high conductivity [11]. Here, we examine the effect of IL-mediated PEDOT electrodeposition on the coatings electrochemical behaviour as well as their surface morphology.

## 2. Materials and methods

(2-D-glucopyranosyloxyethyl)trimethylammonium bistriflimide (IL) was synthesized as described previously [9]. PEDOT films were fabricated through the electrochemical polymerization by means of a PARSTAT 2273 potentiostat in a three electrode system, comprising a commercial ITO-coated PET (ITO/PET) working electrode (Sigma-Aldrich, 60  $\Omega$ /sq) or a sputter-coated Pt/glass electrode, Ag/AgCl (3 M KCl) reference electrode (EDAQ) and a platinum wire as an auxiliary electrode. Cyclic voltammetry (CV) was used to electropolymerize 10 mM EDOT (Sigma-Aldrich) in the presence of 0.1 M KCl (Sigma-Aldrich) or 0.1 M IL, or the 1:1 M ratio mixture of these two electrolytes. CV curves were collected at 100 mV/s for 20 CV cycles within the potential range from 0 V to 1.4 V (vs. Ag/AgCl) (first CV cycle) and -0.5 V to 1.4 V (vs. Ag/AgCl) (next CV cycles). The “charging/discharging” CV curves of the as-formed PEDOT materials were collected in the monomer-free 0.1 M KCl solution at 5 mV/s for 3 CV cycles within the potential range from -0.5 V to 1.0 V (vs. Ag/AgCl).

The electrical properties of PEDOT films were characterized through an electrochemical impedance spectroscopy (EIS, Gamry 600ref) in 0.1 M KCl solution within a frequency range from 500 mHz to 10 kHz, an AC amplitude of 40 mV (vs. Ag/AgCl) and a DC potential of 0 V (vs. Ag/AgCl). The data fitting analysis was performed using EIS Spectrum Analyzer 1.0 software with the application of a Powell algorithm. Capacitances were calculated basing on the parameters of a constant phase element (CPE) according to the formula:

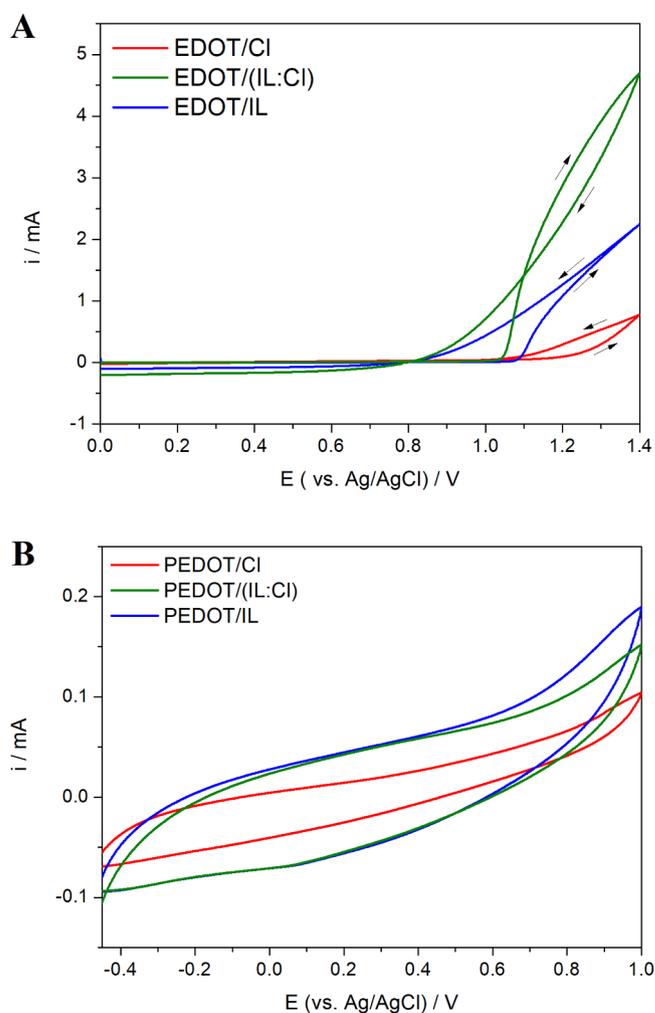
$$C = \frac{(Q_0 \cdot R)^{1/n}}{R}$$

where  $C$  is the film capacitance (F),  $R$  is its resistance ( $\Omega$ ),  $Q_0$  and  $n$  are CPE parameters.

SEM images were collected by means of a Hitachi S-4700 Scanning Electron Microscope operating at 5 kV. IR spectra were recorded using a Varian 660-IR FT-IR spectrometer in the range between 1700 and 700  $\text{cm}^{-1}$  for 16 scans. Thickness and area roughness parameter ( $S_a$ ) were determined using a Filmetrics Profilm 3D optical profilometer.

## 3. Results and discussion

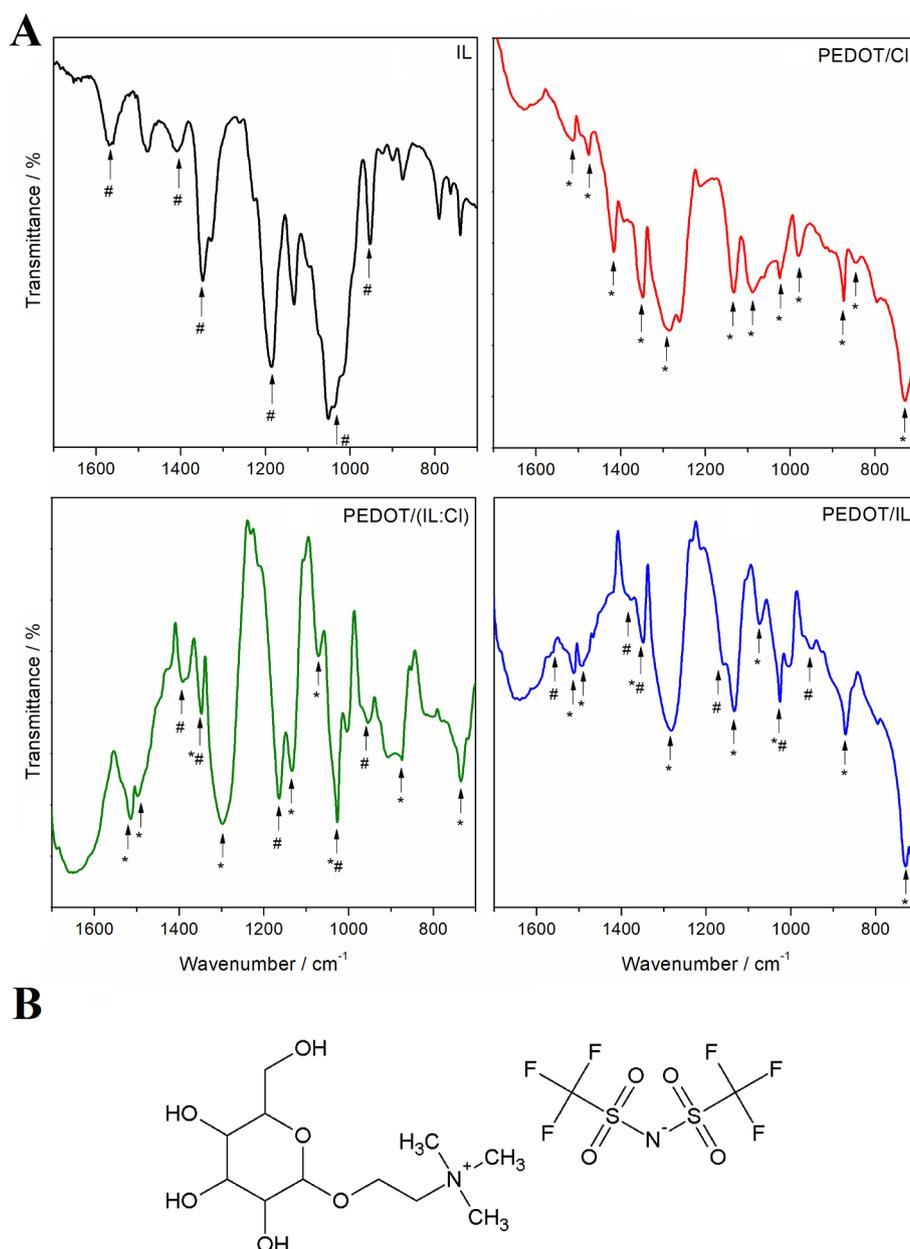
Electrochemical polymerization was used to deposit PEDOT films on the surface of ITO/PET electrodes. This process led to the formation of conducting polymer coatings with the comparable thickness of 736 nm, 800 nm and 718 nm for PEDOT/Cl, PEDOT/IL:Cl and PEDOT/IL, respectively. [Supplementary Fig. 1](#) shows a set of CV curves recorded during the electrodeposition of EDOT in three electrolytes, namely 0.1 M KCl, 0.1 M IL and 0.05 M KCl/0.05 M IL mixture, respectively. Although they appear typical for the polymerization of EDOT in aqueous environment [12], the voltammograms are strongly distorted by the iR effects. Therefore, the evaluation of CV curves needs to be limited to the analysis of the first CV cycle, representing the oxidation of the monomer ([Fig. 1A](#)). Accordingly, the CV curve recorded in the presence of IL show the abrupt onset of the monomer oxidation peak that occurs at lower potentials than for KCl. Consequently, it is necessary to achieve the potential of 1.25 V (vs. Ag/AgCl) to initiate the polymerization in the presence of KCl, and only 1.1 V or 1.05 V (vs. Ag/AgCl) in the case of IL or IL:Cl (1:1), respectively. This could be the effect of the stabilization of radical cations formed as a result of monomer oxidation by the ionic liquid – similarly as observed by



**Fig. 1.** CV curves of the electrochemical oxidation of EDOT (10 mM) in the presence of 0.1 M KCl, 0.05 M KCl/0.05 M IL and 0.1 M IL on the ITO/PET electrode (A), and CV curves of PEDOT/Cl, PEDOT/(IL:Cl) and PEDOT/IL collected in the monomer-free 0.1 M KCl at the scan rate of 5 mV/s (B).

Naudin et al. [13] for some common solvents. Considering that the CV curves of the process of electropolymerization were collected in different electrolytes, to adequately compare the redox behaviour of as-formed films CV curves of PEDOT/Cl, PEDOT/(IL:Cl) and PEDOT/IL were recorded in the monomer-free 0.1 M KCl solution at a low scan rate of 5 mV/s ([Fig. 1B](#)). The shape of the resulting CV curves is typical for PEDOT fabricated in the aqueous medium [12], namely it does not exhibit a distinctive system of redox peaks, as it occurs for PEDOT grown in organic medium [14]. Instead, CV curves show a capacitive behaviour and the faradaic charge capacity of PEDOT/IL and PEDOT/IL:Cl is significantly higher than that of PEDOT/Cl, having its effect on the resistance of the materials (as shown by the results of EIS analysis).

Providing that the IL can act as a doping counterion for the growing polymer, the presence of the IL in the structure of the electrodeposited polymer was subsequently investigated. ATR FTIR spectra ([Fig. 2](#)) were collected for PEDOT/Cl, PEDOT/IL and PEDOT/(IL:Cl) films, as well as for the IL itself, and the bands were assigned to the features of PEDOT and IL structures [15–18]. Spectrum of PEDOT/Cl exhibits all peaks characteristic for this polymer, namely bands associated with C–C and C=C stretching modes in the thiophene ring (1476  $\text{cm}^{-1}$ , 1416  $\text{cm}^{-1}$  and 1348  $\text{cm}^{-1}$ ), ethylenedioxy unit (–O–C<sub>2</sub>H<sub>4</sub>–O–) stretching modes (1285  $\text{cm}^{-1}$ , 1134  $\text{cm}^{-1}$ , 1089  $\text{cm}^{-1}$ , 1025  $\text{cm}^{-1}$ ), C–S vibration modes (980  $\text{cm}^{-1}$ , 874  $\text{cm}^{-1}$  and 842  $\text{cm}^{-1}$ ) and C–S–C deformation (729  $\text{cm}^{-1}$ ) [15–17]. These bands are also present in the spectra of



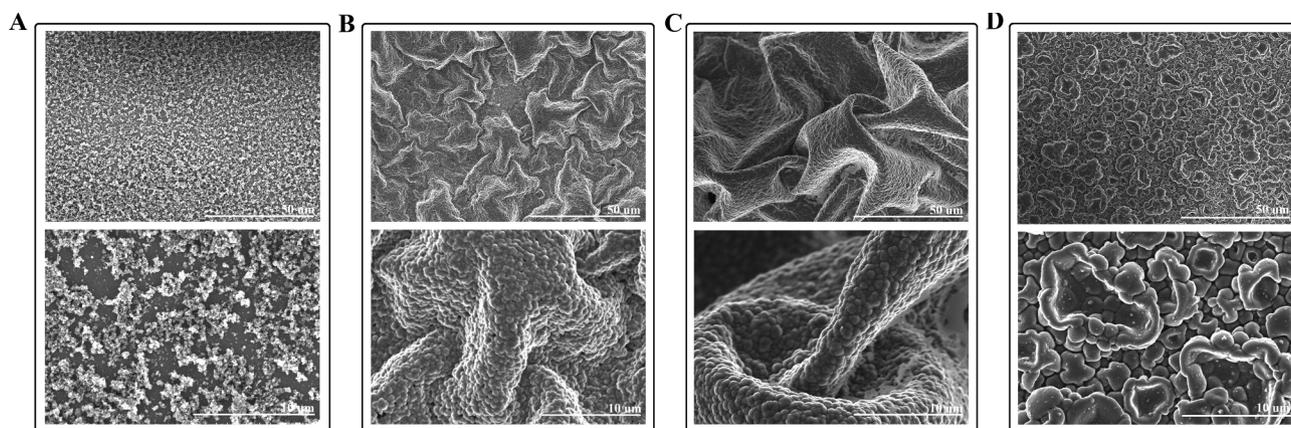
**Fig. 2.** FTIR spectra of IL, PEDOT/Cl, PEDOT/(IL:Cl) and PEDOT/IL (A), and chemical structure of IL (B): \* and # are used to mark the bands characteristic for PEDOT and IL, respectively.

PEDOT/IL and PEDOT/(IL:Cl), clearly demonstrating that polymer has been formed also in the presence of IL. According to Sandoval et al. [17], describing the in situ FTIR studies of PEDOT doped with an ionic liquid [EMMIM]Tf<sub>2</sub>N, FTIR spectra of this polymer contain two set of signals that can be related to the process of p-doping, namely 1415 cm<sup>-1</sup> and 1529 cm<sup>-1</sup>. In the case of PEDOT doped and co-doped with IL, the signal corresponding to the process of p-doping is assumed to appear at 1510 cm<sup>-1</sup> (the second signal is overlaid with the peak at 1406 cm<sup>-1</sup> attributed to C–H bending of IL). Therefore, the presence of a band at 1510 cm<sup>-1</sup> in all three cases indicates that the polymer is present in a p-doped state.

The presence of the IL moiety in PEDOT /IL and PEDOT/(IL:Cl) could be mainly observed by the appearance of band assigned to the –CF<sub>3</sub> stretching (1186 cm<sup>-1</sup>) and peaks attributed to C–H bending (954 cm<sup>-1</sup> and 1406 cm<sup>-1</sup>), and strengthening the band at 1025 cm<sup>-1</sup> due to the overlapping of signal coming from ethylenedioxy unit (1025 cm<sup>-1</sup>) and the signal associated with the stretching modes of –SO<sub>2</sub> and –C–N<sup>+</sup>(CH<sub>3</sub>)<sub>3</sub> (1027 cm<sup>-1</sup>). The signal attributed to the

–NH<sub>3</sub><sup>+</sup> cation, namely the symmetric deformation at 1560 cm<sup>-1</sup>, is also present in the spectrum of PEDOT/IL. Even though the necessity to maintain the electroneutrality of the growing chain suggests that the anion is primarily immobilized within the structure of PEDOT, the presence of peaks attributed to the C–H bending indicates that the (2-D-glucopyranosyloxyethyl) trimethylammonium cation has been also captured by the polymer matrix, likely through encapsulation due to its relatively large size. It can be hypothesised that the encapsulation of IL molecules may affect the structure of the growing polymer, resulting in the generation of a complex 3D morphology as well as favourable electrical performance [19].

Numerous previous studies reported on the effects of the choice of a doping ion on the morphology of electrodeposited conducting polymer films [20,21]. Here, it was observed that the electrodeposition of PEDOT in the presence of IL results in the formation of a unique, highly corrugated surface morphology (Fig. 3). As observed in the SEM images, PEDOT electrodeposited in the presence of KCl only (Fig. 3A) presents a rough morphology with grain structures, usually observed

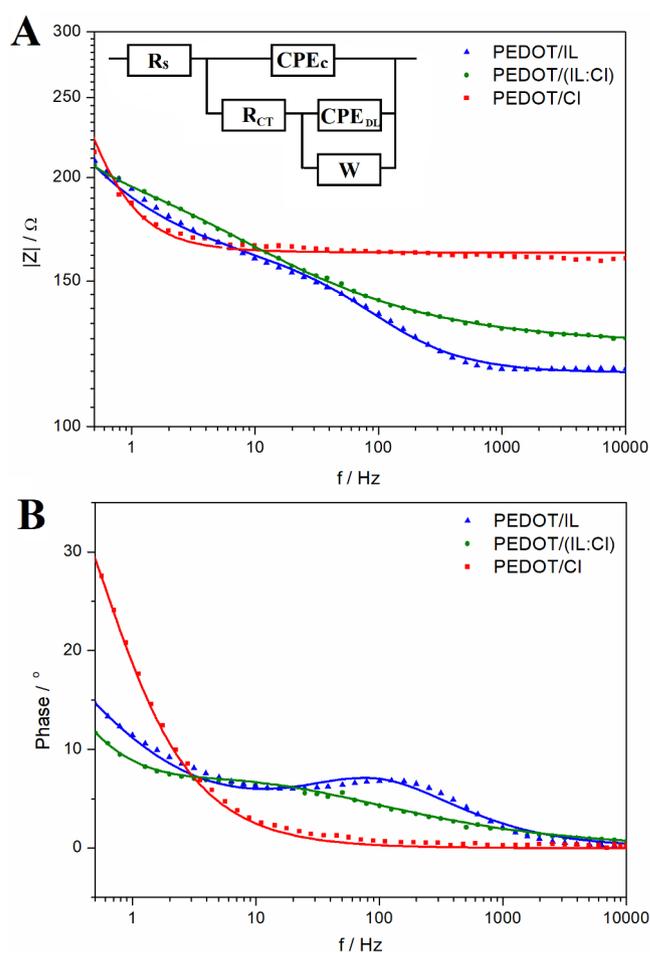


**Fig. 3.** SEM images of PEDOT/Cl (A), PEDOT/(IL:Cl) (B), PEDOT/IL (C) formed on ITO/PET electrodes and PEDOT/IL formed on a sputter-coated Pt/glass electrode (D) at low (top, scale bar 50  $\mu\text{m}$ ) and high (bottom, scale bar 10  $\mu\text{m}$ ) magnification.

for PEDOT doped with small anions [22]. The addition of IL (Fig. 3B&C) is observed to increase the surface area of the polymer coating resulting in the formation of highly corrugated 3D structures. Furthermore, the level of corrugation is found to be dependent on the concentration of IL in the electrolyte, since PEDOT synthesised in 0.1 M IL solution presents more developed structures with respect to the PEDOT electrodeposited in a IL:Cl electrolyte. Interestingly, the formation of highly developed 3D structures is found to occur on the surface of ITO/PET electrodes but not on the surface of a sputter-coated Pt/glass electrode (Fig. 3D). This may be a consequence of a limited adhesion of PEDOT to ITO/PET, as described in [23], derived from its relatively low roughness ( $S_a = 1 \text{ nm}$ ) compared to Pt electrodes ( $S_a = 7 \text{ nm}$ ).

The unique surface morphology of electrodeposited PEDOT/IL and PEDOT/(IL:Cl) is unprecedented in the literature, relative to PEDOT polymerised in the salt solution electrolytes and in the presence of other ionic liquids, e.g. 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide [24], 1-ethyl-3-methylimidazolium tetrafluoroborate, 1-hexyl-3-methylimidazolium tetrafluoroborate, 1-decyl-3-methylimidazolium tetrafluoroborate [10]. Interestingly, a study by Gao et al. [25] describes a composite material possessing a similar wrinkled morphology and formed through a polymer swelling approach coupled with the electroless deposition of silver. These surface structures were shown to enhance the sensitivity of a pressure sensing device, indicating the potential of highly corrugated materials in numerous applications.

The results of EIS analysis (Fig. 4) indicates a significant decrease in electrochemical impedance at frequencies above 10 Hz for both PEDOT/IL and PEDOT/(IL:Cl) coatings relative to the PEDOT/Cl one. This is of a particular importance for the engineering of neural electrodes, which require a low impedance at biologically relevant frequencies and which can be often achieved via conducting polymer coatings [26]. Although PEDOT is known to decrease the impedance of bare metal electrodes, mainly due to its rough surface with developed surface area [27], the introduction of IL results in a further decrease in electrochemical impedance by 19% and 27% for PEDOT/(IL:Cl) and PEDOT/IL films, respectively. Since both the profile of the impedance plot (Fig. 4a) and the phase plot (Fig. 4b) are different for these three types of materials, it suggests that the use of IL as a dopant has a significant effect on the mechanism of charge transfer, derived from variations in conductance and capacitance of the conjugated polymers. In order to describe more precisely the electrical properties of the electrodeposited materials, EIS spectra fitting was performed based on the equivalent circuit proposed by Ates et al. [28], involving the presence of a solution resistance ( $R_s$ ), constant phase element associated with the capacitance of a conducting polymer film ( $CPE_C$ ), charge transfer resistance ( $R_{CT}$ ), constant phase element associated with the processes at the double layer interface ( $CPE_{DL}$ ) and Warburg impedance associated



**Fig. 4.** EIS data in the form of Bode plots showing the impedance (A) and phase (B) dependence on the frequency for PEDOT/Cl, PEDOT/(IL:Cl) and PEDOT/IL coatings; dots represent the experimental results and lines represent the fitted spectra.

with the diffusion of charges through the conducting polymer layer (W). When the EIS spectra of PEDOT/CL, PEDOT/(IL:Cl) and PEDOT/IL formulations were fitted according to the proposed equivalent circuits, the electrical properties were determined (Table 1) with a high certainty ( $\chi^2 < 0.0004$ ), denoting that in every case the relative deviation of the calculated spectrum from the measured data did not exceed 2%. The results show that the presence of IL as a dopant or a co-dopant has a strong effect on the conductance of conjugated polymer, as manifested

**Table 1**

Electrical properties of PEDOT/Cl, PEDOT/(IL:Cl) and PEDOT/IL films determined from the equivalent circuit analysis of EIS data.

	PEDOT/Cl	PEDOT/(IL:Cl)	PEDOT/IL
$R_s$ ( $\Omega$ )	162 $\pm$ 3	126 $\pm$ 2	116 $\pm$ 3
$R_{CT}$ ( $\Omega$ )	690 $\pm$ 55	109 $\pm$ 5	44 $\pm$ 4
$C_C$ (mF)	2.807 $\pm$ 0.056	0.567 $\pm$ 0.011	0.057 $\pm$ 0.006
$C_{DL}$ (mF)	8.709 $\pm$ 1.526	4.568 $\pm$ 0.183	1.382 $\pm$ 0.083
$\chi^2 \times 10^4$	3.05	0.36	2.38

by a decrease in a charge transfer resistance ( $R_{CT}$ ) from 690  $\pm$  55  $\Omega$  for PEDOT/Cl, through 109  $\pm$  5  $\Omega$  for PEDOT/(IL:Cl), achieving the lowest value of 44  $\pm$  4  $\Omega$  for PEDOT/IL. This significant decrease in  $R_{CT}$  occurs as a result of the increased electroactive surface area of PEDOT electrodeposited at the presence of IL, what is observed in the SEM images as highly developed, wrinkled 3D surface, facilitating the charge transfer at the polymer/solution interface. Simultaneously, this phenomenon is responsible for the decrease in the capacitance of IL doped polymer films relative to Cl<sup>-</sup> doped formulations. Interestingly, the electrochemical properties of PEDOT fabricated in the presence of IL were also partially enhanced compared to PEDOT/Cl on rough Pt/glass electrode (Supplementary Fig. 2), even though the unique, highly developed 3D structures were not observed.

#### 4. Conclusions

In this study, it is shown that doping and co-doping of PEDOT with a ionic liquid, (2-D-glucopyranosyloxyethyl)trimethylammonium bistriflimide, results in the formation of unprecedented in the literature, highly corrugated and ridged 3D structures, exhibiting significantly low charge transfer resistance when compared with PEDOT/Cl. Interestingly, this unique structures can be achieved exclusively in PEDOT films electrodeposited onto the surface of ITO/PET electrodes, and are not produced in PEDOT films electrodeposited onto the surface of sputter-coated Pt/glass electrodes. Finally, it can be inferred that its developed surface morphology coupled with an enhanced electrochemical performance make PEDOT/IL an excellent candidate biomaterial with applications in biomedical engineering, especially in the field of neural interfaces and tissue scaffolds.

#### Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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#### Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.elecom.2019.106615>.

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