

## Electroless deposition of silver on poly(3,4-ethylenedioxythiophene) obtained in the presence of polystyrene sulfonate or dodecyl sulfate ions – effect of polymer layer thickness

V.I. Karabozhikova, V.Ts.Tsakova\*

*Institute of Physical Chemistry, Bulgarian Academy of Sciences, 1113 Sofia, Bulgaria*

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Poly(3,4-ethylenedioxythiophene) (PEDOT) layers are electrochemically synthesized in the presence of excess of perchlorate ions and either polystyrene sulfonate (PSS) or dodecyl sulfate (DDS) as co-doping ions. Electroless deposition of silver is studied in two silver plating solutions containing  $\text{Ag}^+$  cations or  $[\text{AgEDTA}]^{3-}$  anion complexes. The electroless reduction of metal ions occurs at the expense of oxidation of mildly pre-reduced PEDOT/PSS and PEDOT/DDS layers with different thickness. It is established that the amount of deposited silver,  $Q_{\text{Ag}}$  depends linearly on thickness with steeper dependence obtained in solution of silver anion complexes in comparison to silver cations. At constant thickness,  $Q_{\text{Ag}}$  depends on the type of PEDOT layers with PEDOT/DDS providing higher  $Q_{\text{Ag}}$ , especially in  $[\text{AgEDTA}]^{3-}$  solution. The results are discussed in terms of limited diffusion of  $[\text{AgEDTA}]^{3-}$  ions inside the polymer structure, possible switching of the rate determining step from diffusion (in the  $\text{Ag}^+$  case) to charge transfer across the polymer layer (in the  $[\text{AgEDTA}]^{3-}$  case), and expected organic ions-induced difference in the internal PEDOT structure. SEM images reveal the possibility to affect markedly the number, size and size distribution of the metallic particles. In terms of homogeneous surface coverage with small monodisperse silver particles best results are obtained with thin PEDOT/PSS/ $\text{Ag}^+$  and thick PEDOT/DDS/ $[\text{AgEDTA}]^{3-}$  layers.

**Keywords:** electroless deposition, silver, PEDOT, polystyrene sulfonate, dodecyl sulfate

### INTRODUCTION

Conducting polymers (CP) are often used as supporting materials for metal particles deposition due to their high electrical conductivity, well developed surface and opportunities for using different chemical and electrochemical deposition techniques to obtain well dispersed metal phase (see [1-6] and literature cited there in). Along with the conventional chemical deposition approach based on metal ions reduction occurring at the expense of oxidation of dissolved reductant species, CPs present another opportunity to drive electroless metal deposition in a way that is close to the immersion techniques for chemical deposition [2, 6]. This method is based on the intrinsic ability of CPs to occupy different interconvertible oxidation states and couples, following reactions:



where  $n$  is the number of electrons exchanged for reducing a single metal ion and  $m$  denotes the extent of initial oxidation of the CP material. For instance, if pre-reducing polyaniline (PANI) in the leucoemeraldine state and immersing it in solution of metal ions with positive enough equilibrium potential, such as silver or palladium, PANI will

undergo oxidative transition to the emeraldine state. This oxidative transition will go in parallel with reduction of the corresponding metal ions. Apart from complete transition between two chemically different oxidation states, electroless metal deposition may occur also in the potential region, where CPs show only pseudo-capacitive behavior characterized with different amount of oxidized monomeric units within one and the same formal oxidation state. In any case the amount of deposited metal phase is limited by the available intrinsic redox charge of the CP layers and depends on the extent of CP initial reduction and the amount of the polymer material. In general, smaller amounts of intrinsic CP redox charge are involved in electroless metal deposition when working in the pseudo-capacitive potential region and thus, smaller amounts of metal become dispersed in the CP material.

Silver-modified CP-based electrodes have been recently intensively investigated for various applications, such as solar cells [7, 8], electrochromic devices [9], sensing applications [10, 11], Surface Enhanced Raman Spectroscopy (SERS) substrates [12], etc. Silver is considered also as an antimicrobial agent that becomes easily combined with polymeric materials [13, 14]. For all these applications the way of dispersing the silver phase is of utmost importance. Silver electroless

\* To whom all correspondence should be sent:

E-mail: [tsakova@ipc.bas.bg](mailto:tsakova@ipc.bas.bg)

deposition at the expense of CP oxidation was studied mainly for PANI [15-25] and polypyrrole [17, 25-30]. Few studies address silver electroless deposition on polythiophenes, such as polyalkylthiophenes [31, 32] and poly(3,4-ethylenedioxythiophene) (PEDOT) [30, 33], although the latter is one of the most studied CP material. The investigations so far performed concern PEDOT layers obtained in aqueous solutions containing either perchlorate or nitrate ions. It is known that PEDOT is very often synthesized in the presence of organic anions such as polystyrene sulfonate (PSS) or dodecyl sulfate (DDS) which act as both surfactant and doping species and affect markedly the CP properties.

The aim of the present investigation is to study the dependence of the amount and type of electroless deposited silver on the thickness of PEDOT layers, synthesized in the presence of PSS or DDS ions. A further goal concerns the comparison between electroless deposition from plating solutions containing two different silver ionic species - silver cations and silver complex anions. In all cases, before immersion in the silver plating solution, PEDOT is subjected to mild reduction in the pseudocapacitive potential region for this polymer, in order to limit the amount of deposited silver. This investigation is a further extension of a series of studies [34-37] trying to elucidate the effect of the co-doping organic anions, used in the course of synthesis of PEDOT, for various characteristics of the polymer layers, such as surface morphology, electroanalytical sensitivity and selectivity with respect to oxidation of organic species and electrochemical and electroless metal deposition.

## EXPERIMENTAL

The electrochemical experiments were carried out by means of Autolab PGSTAT 12 potentiostat/galvanostat equipped with GPES software (Eco Chemie, Utrecht, the Netherlands). A three electrode set-up was used with glassy carbon disk as working electrode (with surface area  $S = 0.08 \text{ cm}^2$ ) and a platinum plate as counter electrode. The reference electrode was saturated mercury/mercury sulfate ( $\text{Hg}/\text{Hg}_2\text{SO}_4/0.5 \text{ M K}_2\text{SO}_4$ ) electrode (MSE).  $E_{MSE} = 0.66 \text{ V}$  vs standard hydrogen electrode. All potentials in the text and figures refer to MSE. Argon gas was used to remove dissolved oxygen from the electrolyte solutions before electrochemical measurements.

Electrochemical polymerization of EDOT was performed at constant potential  $E_a = 0.38 \text{ V}$  in aqueous solutions of 10 mM EDOT, 0.5 M  $\text{LiClO}_4$

and 34 mM organic anionic dopants - sodium PSS or sodium DDS. The PEDOT coatings obtained in the presence of the organic dopants will be further denoted by PEDOT/PSS and PEDOT/DDS, respectively. The thickness of the PEDOT layers was varied by using different polymerization times and fixing the polymerization charges at  $Q_{poly} = 1, 2$  or 4 mC.

Electroless deposition of silver was carried out at pre-reduced PEDOT layers. The electrochemical reduction was accomplished in supporting electrolyte (0.4 M  $\text{HClO}_4$ ) at constant potential (-0.62 V for 15 min), corresponding to the high conducting state of PEDOT. The reduced PEDOT-coated electrodes were transferred in the silver plating solution consisting of either 10 mM  $\text{AgNO}_3$  and 0.4 M  $\text{HClO}_4$ , or 10 mM  $\text{AgNO}_3$ , 0.02 M  $\text{Na}_2\text{C}_{10}\text{H}_{14}\text{O}_8\text{N}_2$  (NaEDTA) and 0.5 M  $\text{KNO}_3$ . In the latter solution the silver species form  $[\text{AgEDTA}]^{3-}$  anionic complexes [16]. The equilibrium potentials of silver in these solutions are  $E^0 = -0.002 \text{ V}$  vs MSE for  $\text{Ag}^+$  and  $E^0 = -0.040 \text{ V}$  vs MSE for  $[\text{AgEDTA}]^{3-}$ . The time for silver electroless precipitation was set at 50 s. After each silver deposition experiment silver was dissolved in 0.4 M  $\text{HClO}_4$  by applying voltammetric scans (at 5 mV  $\text{s}^{-1}$ ) at potentials more positive than the equilibrium potential of Ag. The amount of silver was calculated by integrating the dissolutions peaks registered within the first two scans. The presented data for the amount of silver deposited under each set of experimental conditions (i.e. polymerization charge, type of layer and type of silver ionic species) is the average of three deposition/dissolution experiments. Within individual experiments at otherwise fixed experimental conditions the amount of the silver dissolution-related charge varied within 15 % of the average value.

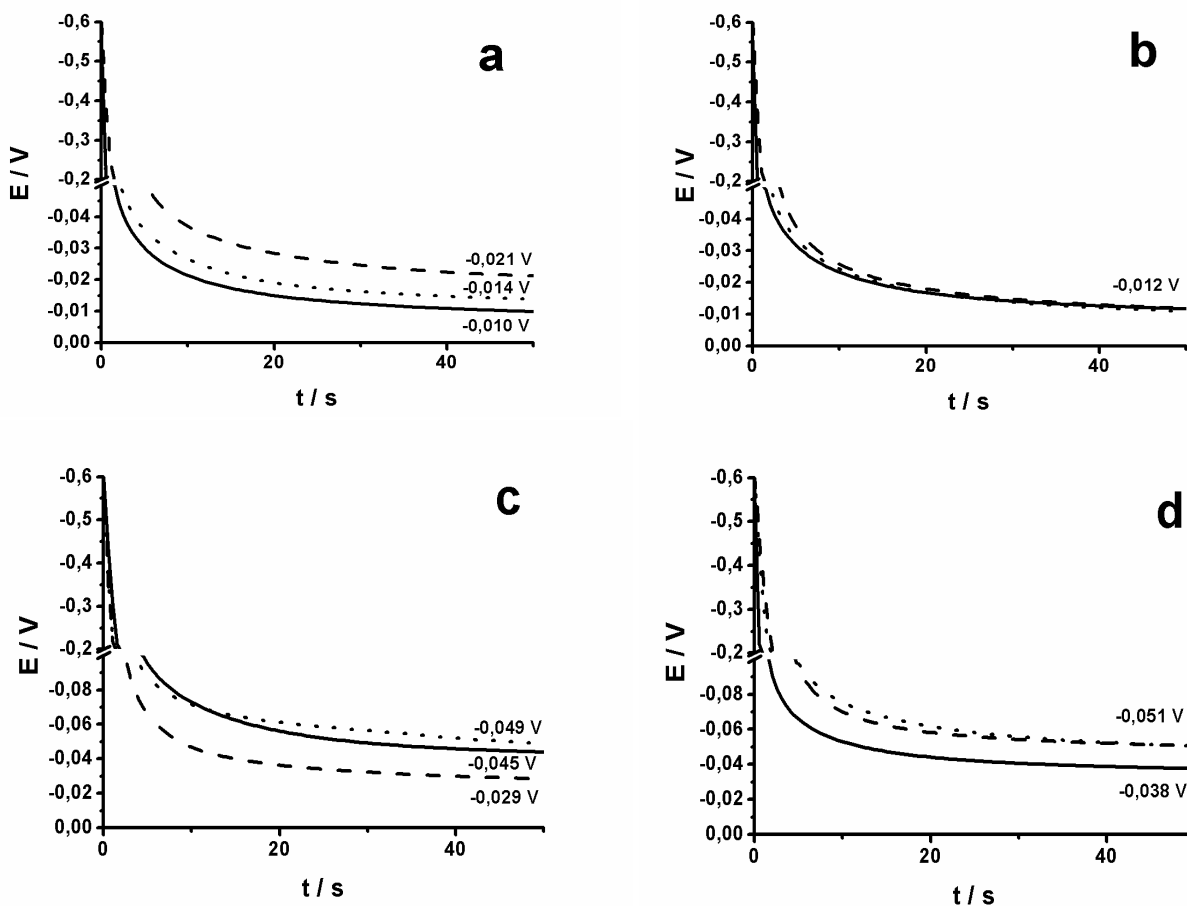
The surface morphology of the various Ag-precipitated PEDOT layers was examined by scanning electron microscope JEOL 6390 equipped with Inca Oxford EDX analyzer.

## RESULTS AND DISCUSSION

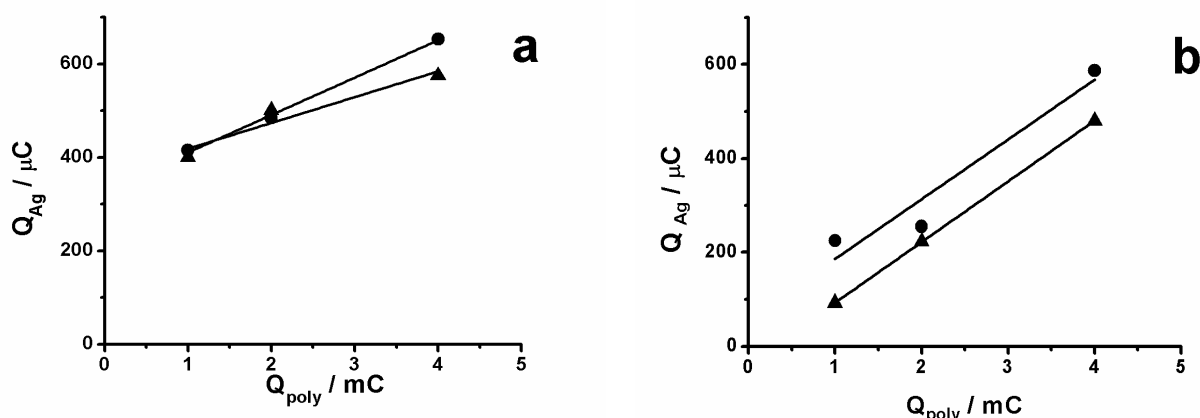
Fig. 1 shows open circuit potential (OCP) transients measured in the course of electroless silver deposition from  $\text{Ag}^+$  and  $[\text{AgEDTA}]^{3-}$  solutions at PEDOT/PSS and PEDOT/DDS layers obtained at different polymerization charges. For all transients a steep OCP drop is observed within the first few seconds followed by a gradual establishment of steady state OCP values. The final OCP values are in all cases more negative than the equilibrium potential of Ag in the respective silver

plating solutions, indicating the establishment of mixed potentials affected by the redox potential of the PEDOT layers themselves. With increasing  $Q_{poly}$  the transients measured at PEDOT/DDS do

not show a strong thickness effect (Fig. 1 b and d). A stronger effect with thickness on OCP transients is observed for PEDOT/PSS layers (Fig. 1 a and c).



**Figure 1.** OCP transients obtained in  $Ag^+$  (a, b) and  $[AgEDTA]^{3-}$  (c, d) solutions at PEDOT/PSS (a, c) and PEDOT/DDS (b, d) layers with different amounts of polymerization charge: 1 mC (dashed line), 2 mC (dotted line), 4 mC (full line).



**Figure 2.** Amount of deposited silver from  $Ag^+$  (a) and  $[AgEDTA]^{3-}$  (b) solutions at PEDOT/PSS (▲) and PEDOT/DDS (●) layers with different polymerization charge.

In this case, the final OCP value shifts to more negative potentials for  $Ag^+$  solution, and to more positive values for  $[AgEDTA]^{3-}$  solution, pointing

to establishment of different mixed potential equilibria depending on PEDOT/PSS thickness. Comparison of the OCP transients obtained in  $Ag^+$

and  $[\text{AgEDTA}]^{3-}$  solution for the thickest PEDOT/PSS layers show that the final OCP values coincide irrespective of the type of depositing silver ions. This result, together with the shift in OCP with increasing PEDOT layer thickness, may be considered as indications for possible PSS-induced Ag complex formation. The possibility for formation of Ag/PSS complex species was considered in [38]. Formation of a complex of Ag with another polysulfonic acid was also discussed in the context of polysulfonic-acid-doped polypyrrole [39]. It can be suggested that with increasing  $Q_{poly}$ , the amount of PSS involved in doping of the PEDOT increases, and gives rise to complexation of silver. The latter may affect the establishment of a mixed potential in the PEDOT/PSS|Ag systems.

The amount of deposited silver obtained in the course of electroless deposition in the two silver plating solutions is presented in Fig. 2. The comparison shows a much steeper thickness dependence of  $Q_{Ag}$  in the presence of silver complex ions, than in the presence silver cations. At doubled polymerization charge  $Q_{poly}$ , the silver amount increases by a factor of 1.25 for  $\text{Ag}^+$  solution and by a factor of about 2 in the  $[\text{AgEDTA}]^{3-}$  solution. There is also a difference in the amount of silver depending on the type of PEDOT with PEDOT/DDS layers, providing larger amounts of silver in the  $[\text{AgEDTA}]^{3-}$  plating solution.

To discuss these results let us consider the individual steps that may affect the overall rate for electroless metal deposition under OCP conditions [30]: i/ ions transport from bulk solution to the polymer surface; ii/ ions mass transfer within the polymer phase; iii/ inter and intra electronic charge transfer across the polymer chains and iv/ charge transfer between the metal ions and the polymer phase. Phase formation phenomena (i.e. metal nucleation and growth) may additionally complicate the situation. Obviously, one or several of these steps are affected by the type of silver ions involved in the electroless deposition process. Bulk diffusion of  $\text{Ag}^+$  is faster than bulk diffusion of  $[\text{AgEDTA}]^{3-}$ . Ionic transport within the PEDOT structure should be easier for  $\text{Ag}^+$  than for the bulky  $[\text{AgEDTA}]^{3-}$  ions, which means that  $\text{Ag}^+$  should have faster and easier access to both external and internal polymer phase surface. This may be in the origin of the large amounts of silver deposited even at low polymerization charges. The somewhat lower amount of  $Q_{Ag}$ , observed for the thickest PEDOT/PSS layer, should be attributed to the fact that equilibrium is established at a more negative

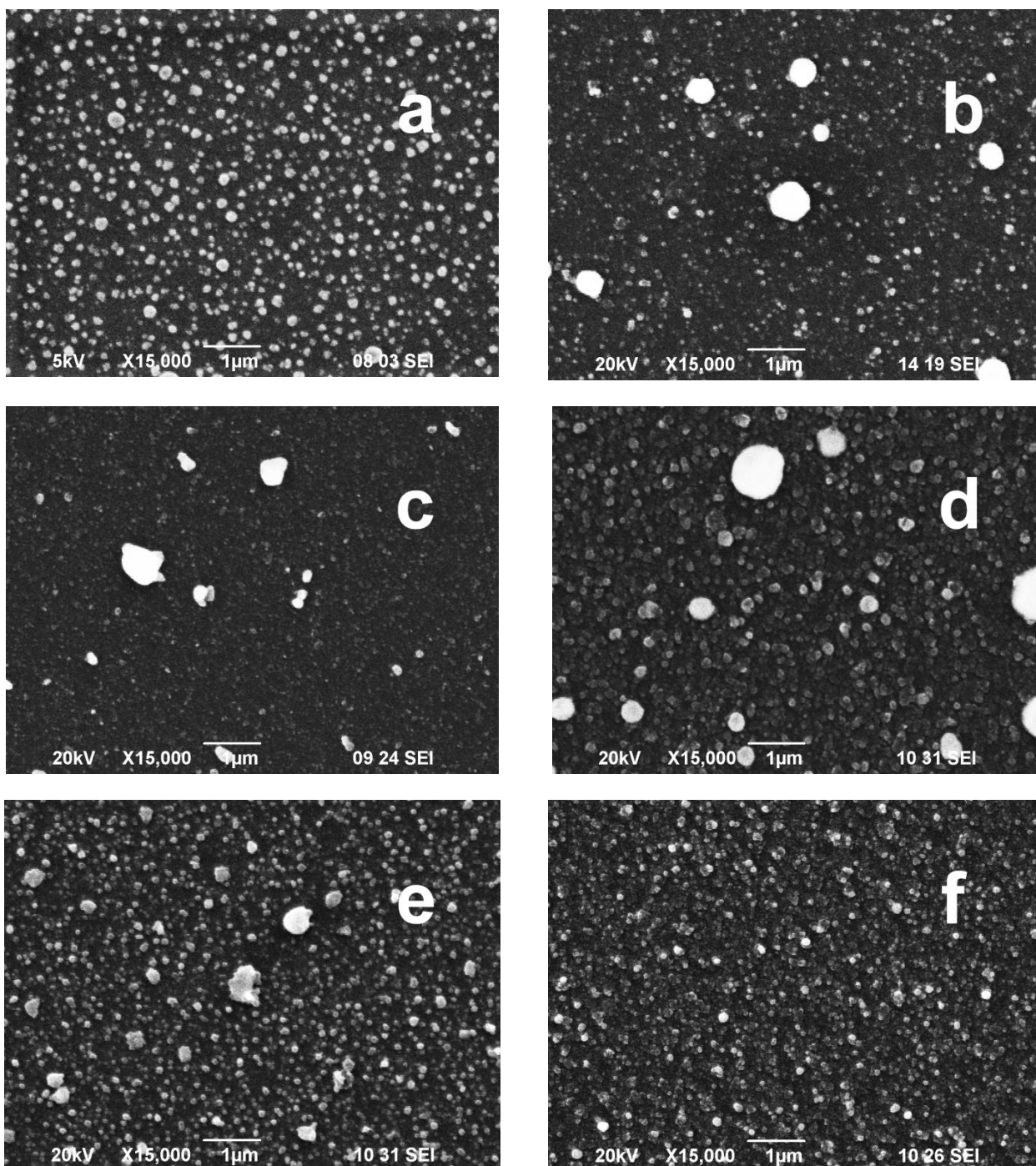
potential. The latter corresponds to less oxidized PEDOT which predetermines the deposition of smaller amount of metal.

In general, the charge transfer between a conducting electrode and a metal anion complex is expected to be slower, and to require more negative potentials than the charge transfer of the corresponding cationic species. Electrodeposition in metal ion complex solutions requires usually higher overpotentials. According to the OCP transients in both solutions, the electroless deposition process occurs in the same range of potentials, which corresponds formally to lower overpotentials in the silver anion complex solution case. The role of the  $[\text{AgEDTA}]^{3-}$  species should be considered also from another point of view. Diffusion of these relatively large anionic species inside the polymer structure will be impeded in comparison to cationic silver. On the other hand, anionic species are involved in the doping of PEDOT occurring in the course of oxidation. However, bearing in mind the large excess of nitrate ions present in the silver plating solution, this should occur at the expense of these inorganic anions. Thus, electroless deposition in the  $[\text{AgEDTA}]^{3-}$  solution is expected to take place at the polymer/solution interface, rather than inside the polymer structure, which means that the transfer of charge across the polymer layer may become the rate determining step of the overall process. This transfer will definitely depend on the polymer layer thickness and specific internal polymer structure. The latter is expected to be influenced by the doping ions used in the course of synthesis. Thus, the difference in the behavior of PEDOT/DDS and PEDOT/PSS layers, established in  $[\text{AgEDTA}]^{3-}$  solution, could be attributed to a different internal structure affecting the charge transfer across the polymer phase. Although, it is difficult to study and reveal the polymer structure, especially inside the polymer phase, SEM studies of PEDOT/PSS and PEDOT/DDS layers give evidence for a difference in the surface morphology of these two materials [35].

SEM observations of the silver deposit, carried out for the various PEDOT layers (Fig. 3), give additional evidence in support of the above considerations. The micrographs demonstrate that both the type of PEDOT layer and the type of the silver species used for deposition affect the number, size and size distribution of the metallic particles. It is readily seen that use of silver complex anions results in a narrow size distribution of smaller crystals and homogeneous coverage of the whole surface (Fig. 3 e and f). This is an expected effect of metal anionic complexes in metal deposition that

was demonstrated in studies on silver plating in the presence of EDTA [40]. Thin and thick PEDOT layers (compare Fig. 3a and 3b with Fig. 3c and 3d) behave in different ways depending on the type of organic dopant used for their synthesis. With increasing thickness the silver deposit on the electrode surface becomes more homogeneous

(smaller size distribution and larger number of crystals) in the PEDOT/DDS case. On the opposite, a more homogeneous silver deposit is found on the PEDOT/PSS layer with smaller thickness (Fig. 3 a). These different trends should relate again to organic dopants-induced difference in the PEDOT structure.



**Figure 3.** SEM of Ag-modified polymer layers with  $Q_{poly}=2$  mC (a, c, e) and  $Q_{poly}=4$  mC (b, d, f) obtained at PEDOT/PSS (a, b) and PEDOT/DDS (c, d, e, f) in  $Ag^+$  (a, b, c, d) and  $[AgEDTA]^{3-}$  (e, f) solutions.

## CONCLUSIONS

The present study shows that electroless deposition of silver on PEDOT is a process that depends markedly on several parameters, such as polymerization charge, type of organic ions used in the course of synthesis, and type of the depositing metal ions (cations or complex anions). Varying these parameters provides the opportunity to obtain not only different amounts of silver, but also completely different number, size and size distribution of the metallic phase. The type of the metal ion provides the opportunity to affect the diffusion inside the polymer phase, and to complete the process by involving both internal and external or, alternatively, mainly external polymer interface. Thus, the rate determining step becomes very probably affected, and limiting diffusion becomes replaced by limiting charge transfer across the polymer phase. As far as internal polymer structure is determining for this process, organic doping ions-induced properties of PEDOT start playing a decisive role.

The results presented so far reveal also the possibility to affect the electroless deposition by metal-complex formation at the expense of the doping ions used in the course of CP synthesis. Indications in this respect are obtained in the PEDOT/PSS|Ag<sup>+</sup> case. This opportunity should be studied in further details by exploring PEDOT layers obtained in the presence of PSS alone, without addition of inorganic anions, in order to reveal better this effect.

Finally, from a practical point of view, the opportunity to obtain a highly homogeneous monodisperse surface distribution of metal particles as provided by thin PEDOT/PSS|Ag<sup>+</sup> and thick PEDOT/DDS|[AgEDTA]<sup>3-</sup> layers is important and supports various applications of the Ag-modified PEDOT material.

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## БЕЗТОКОВО ОТЛАГАНЕ НА СРЕБРО ВЪРХУ ПОЛИ(3,4-ЕТИЛЕНДИОКСИТИОФЕН), ПОЛУЧЕН В ПРИСЪСТВИЕ НА ПОЛИСТИРЕНСУЛФОНАТНИ ИЛИ ДОДЕЦИЛСУЛФАТНИ ЙОНИ – ЕФЕКТ НА ДЕБЕЛИНАТА НА ПОЛИМЕРНОТО ПОКРИТИЕ

Василена И. Карабожикова, Весела Ц. Цакова\*

*Институт по физикохимия, БАН, 1113 София, България*

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(Резюме)

Поли(3,4,етилендиокситиофен) (ПЕДОТ) е синтезиран електрохимично в присъствие на излишък от перхлоратни йони и на полистиренсулфонатни (ПСС) или додецилсулфатни (ДДС) ко-дотиращи йони. Безтоково отлагане на сребро е изследвано в два разтвора на сребърни йони, катиони и  $[\text{AgEDTA}]^{3-}$  анионни комплекси. Безтоковата редукция на металните йони се осъществява за сметка на окисление на предварително редуцирани слоеве от ПЕДОТ/ПСС и ПЕДОТ/ДДС с различна дебелина на полимерното покритие. Установено е, че количеството на отложеното сребро,  $Q_{\text{Ag}}$  зависи линейно от дебелината на полимерния слой като зависимостта е много по-стръмна при използване на разтвор на сребърни анионни комплекси в сравнение със сребърни катиони. При постоянна дебелина на полимерното покритие,  $Q_{\text{Ag}}$  зависи от вида на ПЕДОТ като слоевете от ПЕДОТ/ДДС осигуряват по-високи стойности на  $Q_{\text{Ag}}$ , особено в разтвор на  $[\text{AgEDTA}]^{3-}$ . Резултатите са обсъдени от гледна точка на ограничена дифузия на анионните комплекси на сребро в вътрешността на полимерната структура, възможна промяна на скоростоопределящата стъпка на процеса от дифузия (в случая на  $\text{Ag}^+$ ) към пренос на заряд през полимерното покритие (в случая на  $[\text{AgEDTA}]^{3-}$ ), както и очаквана, свързана с ко-дотиращите органични йони, структурна разлика на двата вида покрития от ПЕДОТ. Микроскопски снимки разкриват възможността да се влияе съществено върху броя, размера и разпределението по размер на металните частици в зависимост от вида и дебелината на полимерното покритие, както и на типа на отлагания се метален йон. Най-добри резултати от гледна точка на хомогенно покритие на полимерната повърхност с малки монодисперсни сребърни частици са получени в случая на тънки ПЕДОТ/ПСС/ $[\text{Ag}^+$  и дебели ПЕДОТ/ДДС/ $[\text{AgEDTA}]^{3-}$  слоеве.