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The Changing with Dielectric Constant of the Capacitance of Single Carbon Fiber Coated with PolyThiophene and their Derivatives

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Abstract

In this study, the effect of dielectric constant on the electrical, capacitive and morphological properties of single carbon fiber(SCF) electrodes coated with Poly(thiophene), Poly(3-methylthiophene), Poly(3-dodecylthiophene) or Poly(3-imidazoletthiophene) was investigated. Monomers were electropolymerized separately on SCF in 5 ml solutions with different dielectric constant(ϵ) by Cyclic Voltammetry technique. Capacitive and morphological characteristics were determined by using suitable techniques. It was found that the oxidation potential and maximum current values of the monomers and the specific and double layer capacitance(C_{sp} and C_{dl}) values of the electrode coated with their polymers decreased with the weight increase in the -R group in the 37,5 media. C_{sp} values were in the same range for derivative polymer coatings, which the range of 0-3,333 Fcm⁻². When the potential was applied to the electrode, C_{sp} value was decreased as -R weight was increased. It was seen that the value was increased for ring group in the same conditions. In $\epsilon:37,5$ C_{sp} value was increased to 36,666 Fcm⁻² from 21,666 Fcm⁻² when potential was increased to 0,6 V from -0,2V for Poly(3-imidazoletthiophene). It was thought that solution mixtures could be more effective to change and control the electrical and capacitive properties of the electrode covered with conductive polymer.

Keywords: Conducting polymer, electropolymerization, specific capacitance, dielectric constant

INTRODUCTION

In order to improve the strength of resin composites, either reinforcing materials are added to the composite or surface treatment methods are applied. In general, carbon fibers (CF) are one of the preferred reinforcing materials due to their high strength and good rigidity (Montes-Moran et al., 2004). To increase the interfacial performance in the composite, the carbon fiber surface is processed by applying methods such as plasma (Montes-Moran et al., 2001; Dilsiz et al., 1995; Bubert et al., 2002 and radiation (Li et al., 2005; Xu et al., 2007), etc. Recently, a method of electrochemically coating the carbon fiber surface with a thin layer of conducting polymer film has been proposed by researchers in addition to such methods. This method has been adopted as a suitable approach for covalently adding functionality to the carbon fiber surface (Kumru et al., 2001). The thickness, morphology and functionality of the resulting coating can be controlled using this method. Because in this method, parameters such as current density, potential, monomer concentration and scanning rate can be adjusted (Sarac et al., 2008; Dolas et al., 2011). The film layer is attached to the electrode surface by electrostatic interactions. These interactions reduce the attacks of the metal/polymer interface through the lamellar separation of the electrolyte. For this reason, the content of the electrolyte used affects the attacks at the interface. In addition, it also affects the electrical and capacitive features of the CF coated with conducting polymer. Polythiophene (PolyTh) plays an important role in research and industrial fields due to its high conductivity, thermal, chemical and environmental stability. Poly(Th) and its derivatives have been attractive for use in applications such as light-emitting diodes (LED), hydrogen storage, and removal of dye, paint and heavy metals from water (Gurunathan et al., 1999; Kanicki, 1983; Bergveld, 1991). It is known that the electropolymerization of thiophene is hard

and requires high potentials due to its ring has high electron density. For this reason, most studies have been done with 3-alkyl thiophene derivatives due to decreasing of electron density. Mousavi et. al. combined the Th monomer with nanoparticle materials to improve its stability and performance (Mousavi et al., 2016; Mousavi et al., 2015). Moreover, The was combined with nano-clay to enhance nano-microbial effects (Farazi et al., 2018), nanofiber to facilitate drug delivery (Zakeri et al., 2018), nanofiber to manage drug release (Savardashtaki et al., 2017), silica (Mousavi et al., 2014.), epoxy (Mousavi et al., 2013) and many other similar materials. The electropolymerization of thiophene is carried out in organic solvent media such as acetonitrile due to its limited solubility in aqueous and common inorganic solvents. However, recently electropolymerization of some thiophene derivatives has been done in different solvent mixtures such as water/methanol (Seki et al., 2019). This approach can be attributed to the fact that solvent mixtures affect the polarity of the medium. The dielectric constant of a solution is a measure of its polarity and is known to affect electrostatic interactions. Capacitance is known as the ability of a material to keep the load on or given to its structure. Since the capacitance is the opposite of conductivity, while the conductivity of a material increases, it can be thought that its conductivity increases because material cannot hold the load in its structure or given. Rather two types of capacitance were mentioned. One of them was specific capacitance (C_{sp}), which expresses the capacitance of the material per unit area or unit mass, and the other was the double layer (C_{dl}), which expresses the capacitance of the double layer formed in the material in case of loading. In the light of this informations, it was hoped that if the solvent used in electropolymerization is mixed with solvents having different dielectric constants, the electrical and capacitive features of the electrode coated with conducting polymer may change. In

our study, it was investigated how the electropolymerization of Th or its derivatives and the capacitance of the electrodes coated with the polymer or its poly derivatives would be affected in medium had different dielectric constants and/or when potential was applied. Electropolymerizations were carried out in solvent mixtures with different dielectric constants. Thus single carbon fibers (SCFs) were electrochemically coated separately with various Th or its derivatives bearing different $-R$ alkyl or aryl groups. On the other hand, the dielectric constant of solution was adjusted to the desired value by mixing various solvents.

EXPERIMENTAL SECTION

Material

In this study thiophene (Th), 3-methylthiophene (3-mTh) (98%, 374024), 3-dodecylthiophene (3-dTh) (97%, 456365) were used as monomer. A comonomer as named 3-imTh was synthesized according to ref. Dolas et. al., 2016c, bearing both Th and imidazole (im) units in its structure and im was bounded to Th at 3-position. All monomers were purchased from SIGMA-ALDRICH. Bortrifluoride-ether complex ($BF_3O(C_2H_5)_2$) was purchase from FLUKA (15720). Density of this compound was 1,20-1,35 g/L and its BF_3 content was 46,5-49,5%. Sodium perchlorate ($NaClO_4$ -98%-51513) and acetonitrile (ACN-34888) (E Chromasolv for HPLC for far UV) were purchased from SIGMA-ALDRICH. Dielectric constant was 4,33 for Diethyleter and 37,5 for ACN. All compounds were used in experimental tests as received.

Characterization

In this study, related instruments were used for each purpose. So, to polymerize electrochemically the monomers, a Princeton marked potentiostat was used. Polymerization was carried out in cell included three-electrode. The electrodes were Pt wire, Ag wire and SCF. Checked by a 0,1M of ferrocene solution, Ag wire was performed as reference electrode. Having 0,05 mm diameter Pt wire

was used as counter electrode. SCF was used as working electrode. This electrode had a diameter of 10 μm and was coated with the polymer to a height of 1 cm. the area covered by the polymer was approximately $3 \cdot 10^{-3} \text{ cm}^2$. All electrodes were smoothed and polished with alumina before being placed in the cell. Then they were cleaned by using water and acetone. Also the electrodes were positioned in solution at a distance of 1 cm from each other. After Pt electrode was coated electrically with polymer, the EIS data of the coated electrode were taken as using an electrochemical impedance software (PowerSine) from 10 mHz to 100kHz in three electrode cell system at room temperature ($25 \pm 1^\circ C$). AC signal was applied as amplitude of 10 mV. To investigate for morphological, the images of electrodes coated with conducting polymer was taken by using A Nano Eye desktop mini-SEM instrument (SEN-3000 M). Atomic force microscopy (AFM) with a scan head of 60 μm used tip (APPNANO Si, N-type) was performed to investigate the coatings as 3D, topographically. In all AFM analysis, the non-contact mode was employed by using Al-coated high-resonance frequency silicon tips.

RESULT and DISCUSSION

In this section it was explained how different dielectric constant having solutions were prepared. In order to see the effect of dielectric constant on the oxidation potential of the monomer, firstly solutions with different dielectric constants were prepared by using a mixture of ACN and Diethyleter in different ratios. The used electrolyte was BF_3 . The calculations were made according to followed equation:

$$\epsilon_{sol} = (\epsilon_1 * r_1) + (\epsilon_2 * r_2) \quad (1)$$

Where ϵ_{sol} . means the total dielectric constant of solution, ϵ_1 , first solution dielectric constant, r_1 ratio of first solution. ϵ_2 second solution dielectric constant and r_2 ratio of second solution. This equation can be applied to whole range of the composition of solution to calculate the

dielectric constant of the solution as it includes all the variables. The dielectric constants of the solution and used ratio were 37,5 (100% ACN), 30,866 (80% ACN), 24,232 (60% ACN), 17,598 (40% ACN),

10,964 (20% ACN), and 4,33 (0% ACN). The oxidation potential and maximum current (I_{max}) values were read from the CV plots for each monomer. Obtained plots were showed in Figure 1.

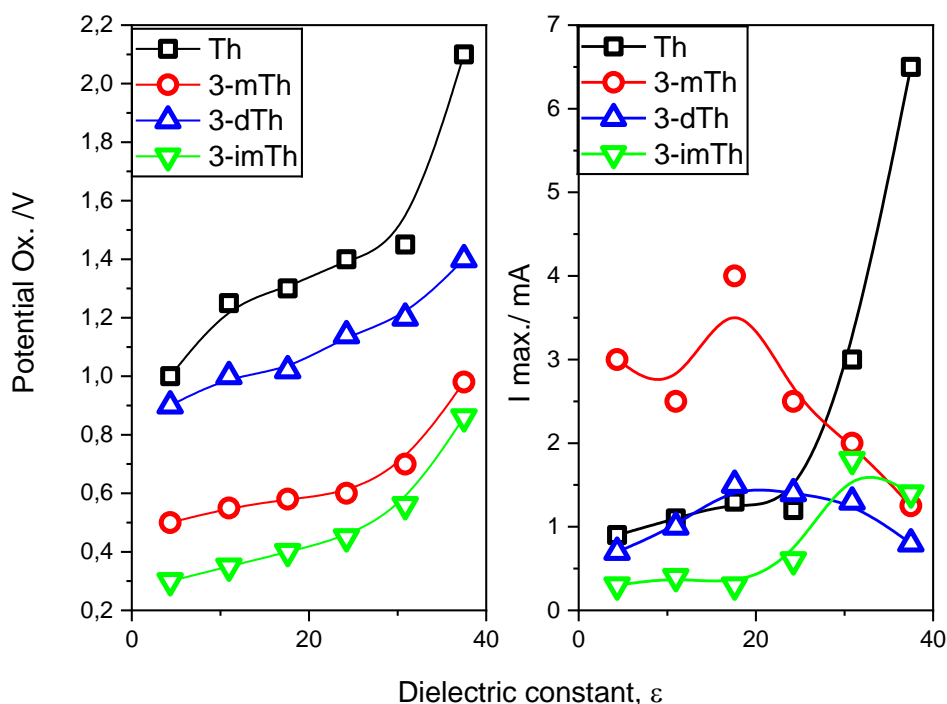


Figure 1. The plots of (a) the oxidation potential (P_{ox}) and (b) the maximum current (I_{max}) versus the dielectric constant of solution (ϵ).

As seen from these plots the oxidation potentials of the monomers were decreased as solution dielectric constant was decreased. This means that the polymerization of the monomer can be started at lower potentials by decreasing the dielectric constant of used solution. So, the monomer is prevented from over oxidation. This result was expected because of that dielectric constant of solution was affected the movement of electron or other ionic species. The over oxidation causes the decomposition of monomer structure. Therefore, lowering the oxidation potential of the monomer is vital. This decreasing was more for 3-imTh, as expected. In addition, this result was attributed to the effect of $-R$ alkyl group. As the group

length or ring structure was increased in low dielectric constant condition, I_{max} values were decreased as dielectric constant was decreased. So, the potential and current values were read as 0,86 V; 1,4 mA in high dielectric constant condition, 0,3 V; 0,3 mA in low dielectric constant condition for Poly(3-imTh). This result may be due to the Th and im transport of comonomer. Because Th and im monomers together give electron donor-acceptor properties to the structure. The current data can be give informations about the thickness and quality of coating on the electrode. It was seen that the obtained coating in high current was high quality and thick. So, it can be said that the quality and thickness of coating on the electrode was higher for 3-

mTh than that of the others at low dielectric constant. Figure 2 illustrated the polymer growth graphs of monomers in two

solutions having different dielectric constants, comparatively.

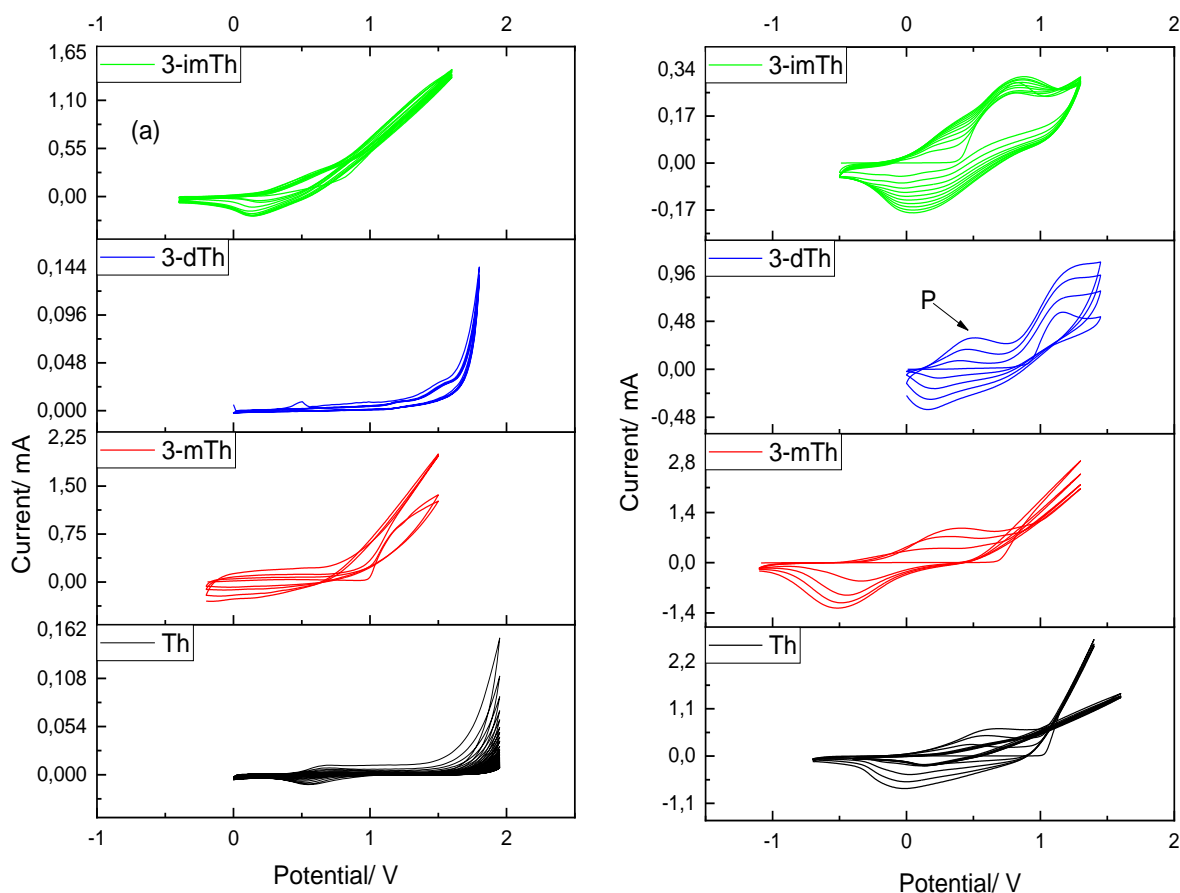


Figure 2. The CV graphs for each monomer in (a) the ϵ : 37.5 of solution; (b) the ϵ : 4.33 of solution. (monomer concentration: 0,01M, solution volume: 5 mL; temperature of media: 25 °C, scan rate: 50 mV/s; voltage range: 0-1,5/2 V; electrolyte: BF_3 (ϵ : 4.33) or NaClO_4 (ϵ : 37.5)).

The shapes of these voltammograms supported that the electropolymerization was more easy and clear in low dielectric constant for each monomer than that in high. This can be seen from the Figure 2b. After monomer oxidation at first cycle, polymer formation on electrode can be understood the increasing of peak current (P, in Figure 2b). The CVs of a conducting polymer coated electrode in monomer free solution by applied different scan rates

supply important and valuable information about whether the electropolymerization procedure is controlled diffusion or not. The procedure is controlled by diffusion if the peak current values of oxidation and reduction is linear with the square root of scan rate. For this reason, the related graphs of obtained electrode coated with Poly(Th) or Poly(3-dTh) were illustrated in Figure 3(a and b).

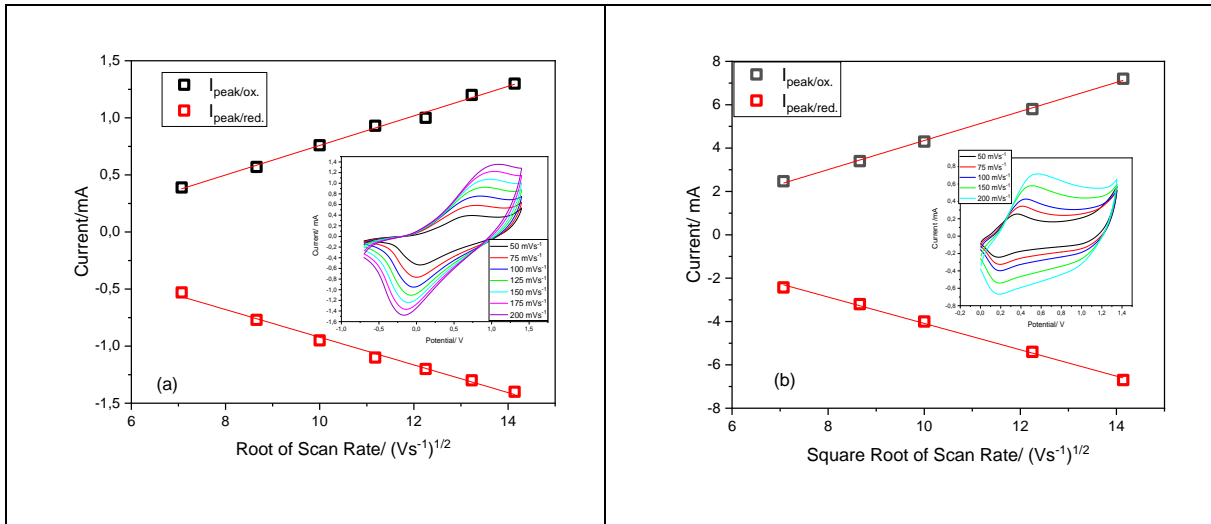


Figure 3. The graphs of the monomer free (inset) and the square root of scan rate vs. current of electrode coated with (a) Poly(Th) and (b) Poly(3-dTh) (solution ϵ : 4.33; solution volume: 5 mL; electrolyte: BF_3).

When investigated the figure, it was determined that the electropolymerization process of Th or 3-dTh were controlled diffusion in the solution dielectric constant of 4,33. The diffusion coefficient of Th ring having dodecyl group at 3 position was bigger than the that of Th, due to the magnitude of area between the oxidation and reduction peak current for each polymer. This magnitude was bigger for Poly(3-dTh) than the that of Poly(Th). The

electropolymerization procedure of Th bearing $-\text{R}$ group at 3-position was illustrated in Figure 4 as given in (Dolas et al., 2011). Th ring was affected by ions or molecules in solution. The ions or molecules during electropolymerization may be polarized or moved into polymer structure. These actions can be changed the electrical or capacitive feature of the electrode coated with the polymer. So this effect was shown in the figure.

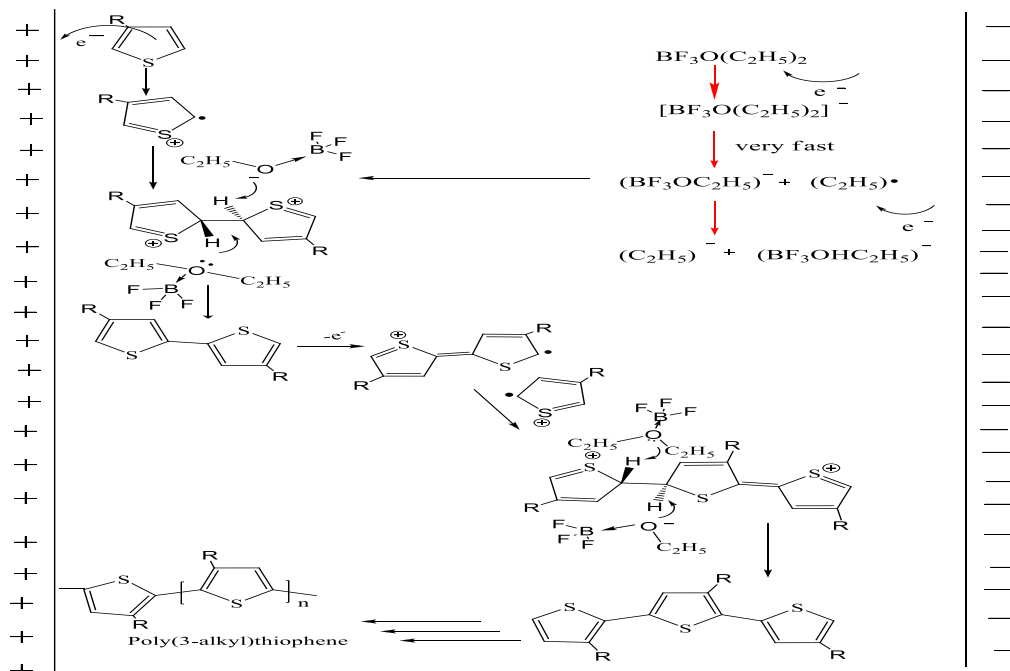


Figure 4. The electropolymerization process steps of Th bearing $-\text{R}$ group at 3-position in low dielectric constant condition. (Dolas, et. al. 2011)

To determine the above mentioned effect, the capacitive features of electrodes coated with Poly(Th), Poly(3-mTh), Poly(3-dTh), or Poly(3-imTh) in solutions having different dielectric constant were measured in monomer free medium. Obtained impedance data were transformed to

calculate the capacitive features such as double layer capacitance (C_{dl}), phase angle and specific capacitance (C_{sp}) according to (Dolas et al., 2011; Dolas et al., 2014; Dolas et al., 2016a; Dolas et al., 2016b). Calculated capacitive features were illustrated in Figure 5.

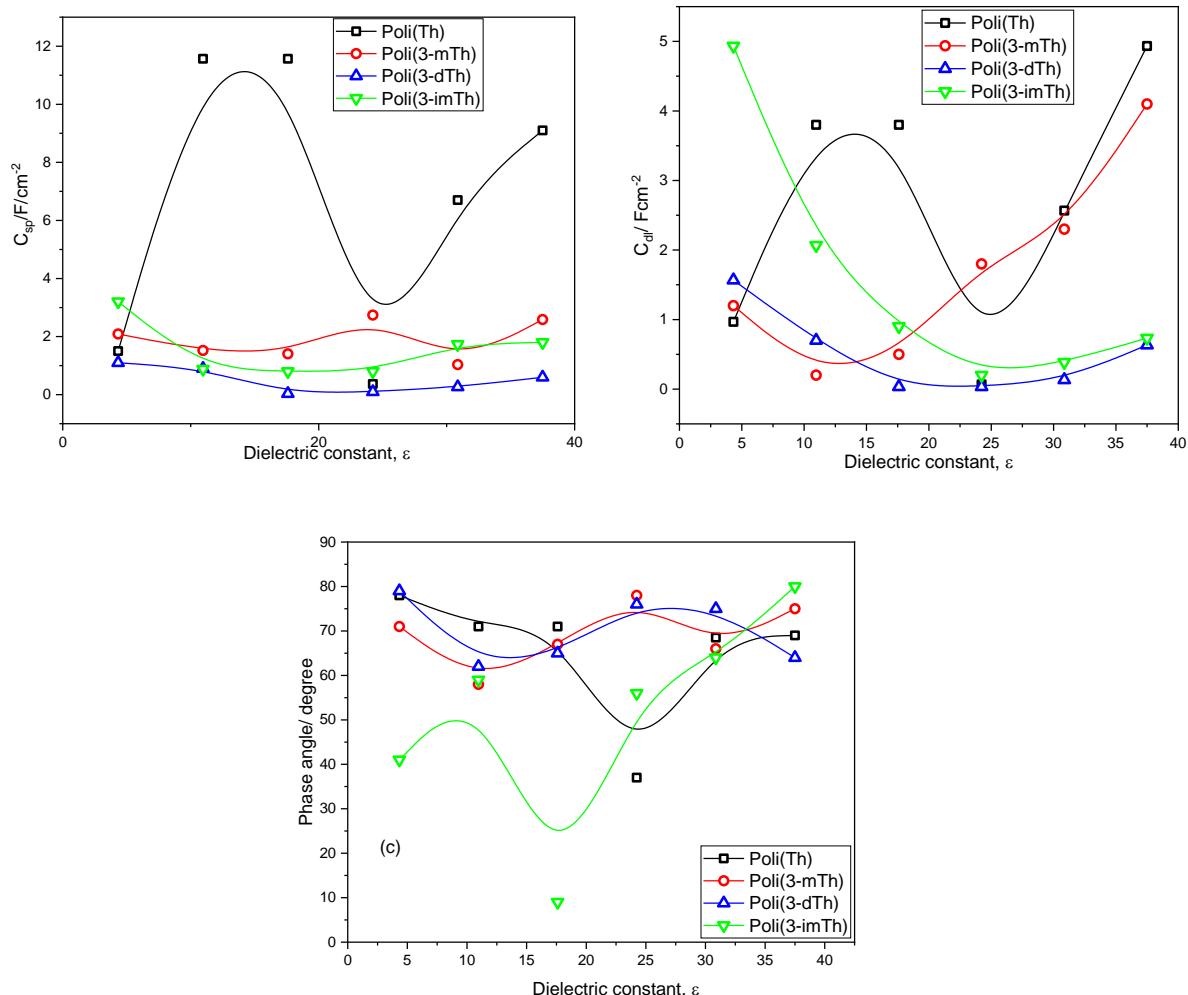


Figure 5. The capacitive feature plots a) C_{sp} graph, b) C_{dl} graph, and c) Phase angle graph

According to the Figure, the C_{dl} values of the electrodes coated with Poly (Th), Poly(3-mTh), Poy(3-dTh) and Poly(3-imTh) were in tendency of decreasing with dielectric constant. This result was expected because of organic medium. For all polymer examples C_{dl} value in the smallest dielectric constant medium was higher than that in the second smallest dielectric constant medium except for Poly(Th) (in Fig. 5b). But as generally, when C_{sp} and C_{dl} were decreased as $-R$ group length and ring structure in low

dielectric constant condition. Only the C_{dl} values of the electrodes coated with Poly(Th) or Poly(3-mTh) showed an increase. It was thought that was due to the fact that they were less conductive than other polymers. According to the data, the C_{dl} values changed to $0,200 Fcm^{-2}$ from $1,200 Fcm^{-2}$ for Poly(3-mTh); to $0,70 Fcm^{-2}$ from $1,566 Fcm^{-2}$ for Poly(3-dTh); to $2,066 Fcm^{-2}$ from $4,933 Fcm^{-2}$ for Poly(3-imTh) for the medium with to smallest dielectric constant. But this changing was

to $3,800 \text{ Fcm}^{-2}$ from $0,966 \text{ Fcm}^{-2}$ for Poly(Th). Also, C_{sp} values were in the same range for derivative polymer coatings, which the range of $0-3,333 \text{ Fcm}^{-2}$. This behavior can be attributed with $-R$ alkyl group bounded to Th ring due to that the $-R$ alkyl group was increasing the conductivity of the polymer, which decreasing capacitance. The phase angle curves of the electrodes were shown in Figure 5c. The values were in an increasing trend in the range of $60-85^\circ$. This increasing was more in Poly(3-imTh) than the other. It was about 65° . In previous studies, $R(C(R(QR)))$ and $R(C(R(QRW)))$ modellings were suggested as the electrical circuit modeling (ECM) for the electrodes coated with Poly(3-mTh) (Dolas, 2014) and Poly(3-dTh) (Dolas, 2011), respectively. According to the informations obtained

about impedance, it can be suggested that the general electrical circuit modeling (ECM) between the solution//coating//electrode system may be expressed with a simple circuit as $R(C(R(QR)))$. The components used in the circuit was explained as followed: the solution resistance was first component. The resistance expressed that the resistance of solution between the electrode coated with the polymer and electrolyte, R_s . Second component was double layer capacitance. It took place in the circuit as parallel combination of the pore resistance, R_p . charge transfer resistance and Constant phase element (CPE) were combined parallel to each other. This part formed third component was combined in series connection to R_p . This situation was illustrated in Figure 6.

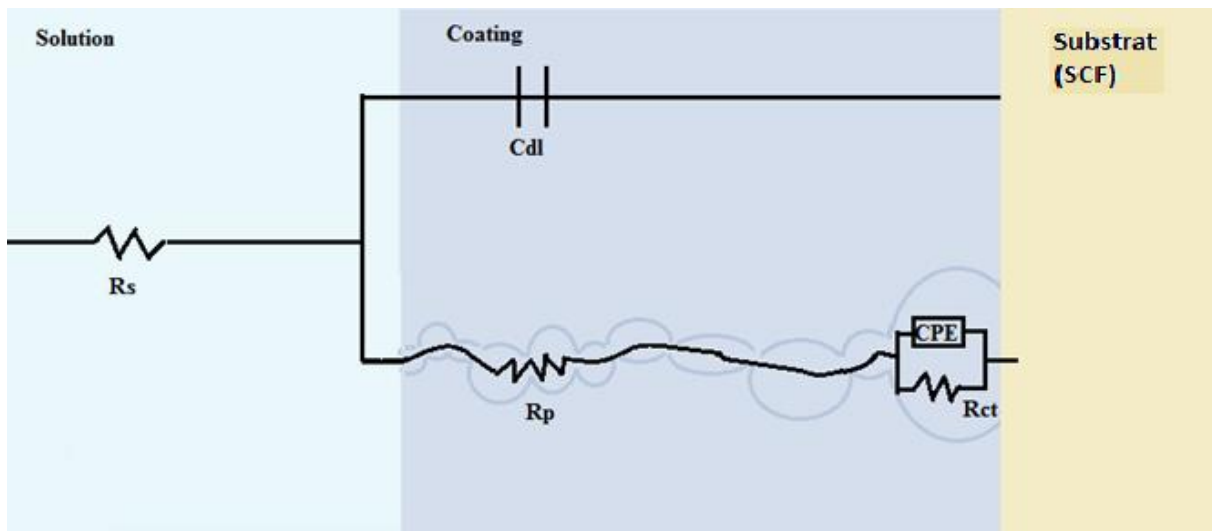
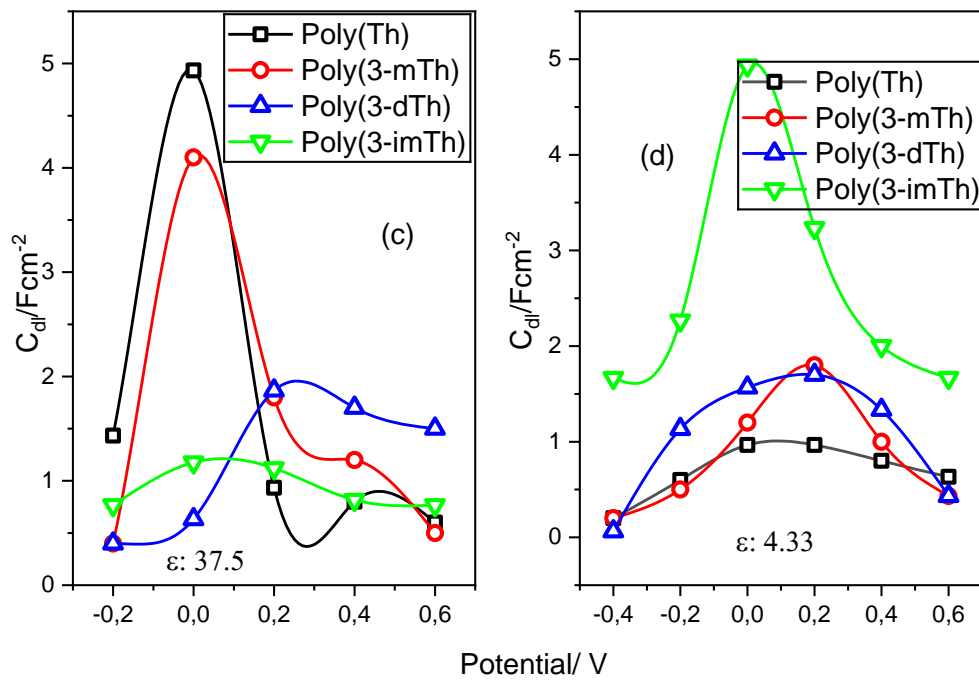
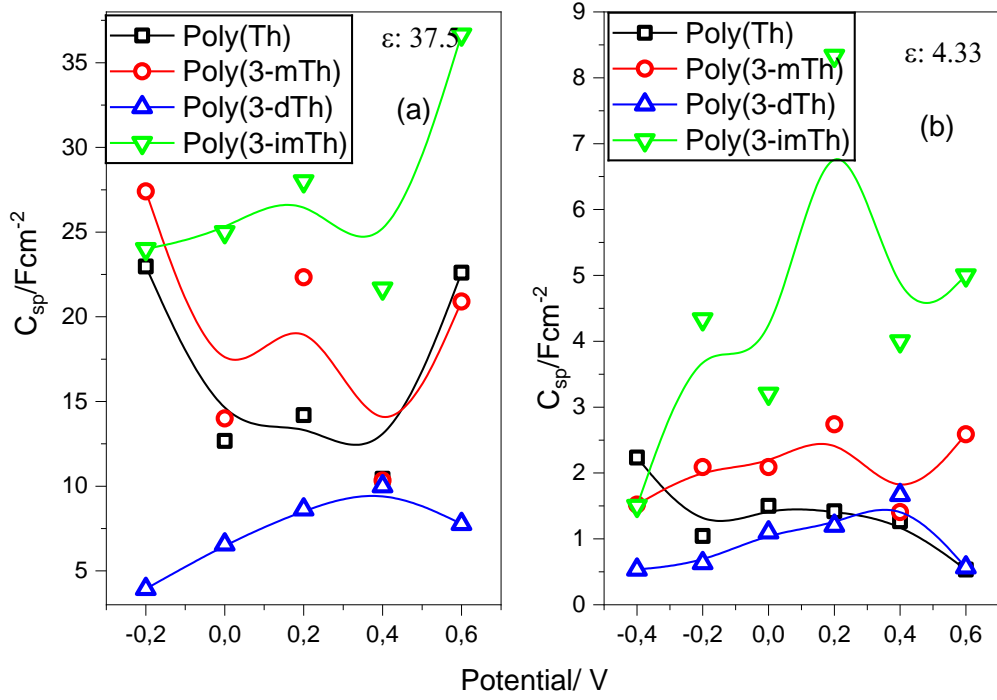


Figure 6. The estimation the electrical equivalent circuit of the electrode coated with conducting polymer during the impedance measurement in monomer free solution

To investigate the effect of applied voltage on capacitance values of the electrodes coated with Poly(Th) or derivatives, five different voltages were applied to the obtained electrodes by coating with the

Poly(Th) or its derivative polymers in solutions having two different dielectric constants during impedance measurements. The obtained data were shown in Figure 7, comparatively.



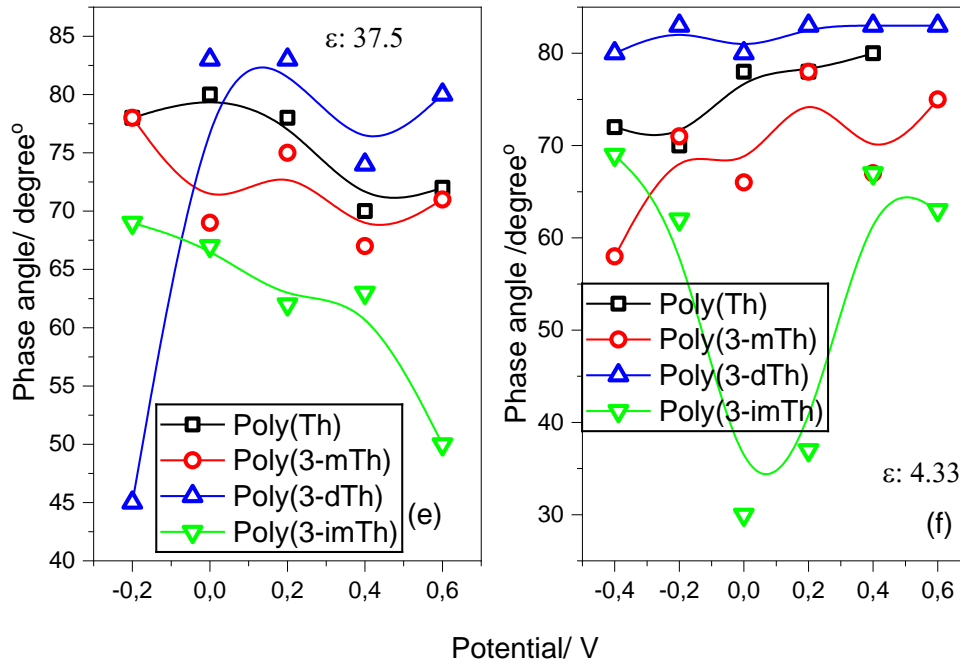


Figure 7. The comparison of the graphs of a) C_{sp} ; b) C_{dl} ; and c) phase angle for electrodes coated with Poly(Th), Poly(3-mTh), Poly(3-dTh) or Poly(3-imTh) in different solutions having dielectric constant by applied different voltages

As seen that the graphs, when a potential was applied the electrode coated with conducting polymer the capacitance property of the electrode was changed (fig 7 a-b). This behavior was expected because of the polarization of the electrode surface. The medium in the round of the electrode was affected the polarization due to the dielectric constant. Applied potential was shown the positive effect on ring group, while it was shown the negative effect on the heavy $-R$ group for C_{sp} and C_{dl} values. This effect was more in dielectric constant condition than the low one. So, in $\epsilon:37,5$ C_{sp} value was increased to $36,666 \text{ Fcm}^{-2}$ from $21,666 \text{ Fcm}^{-2}$ when potential was increased to $0,6 \text{ V}$ from $-0,2\text{V}$ for Poly(3-imTh). On the other hand, as the potential was applied, the C_{sp} values of coated electrode increased to $22,600 \text{ Fcm}^{-2}$ from $10,333 \text{ Fcm}^{-2}$ for Poly(Th), to $20,900 \text{ Fcm}^{-2}$ from $10,333 \text{ Fcm}^{-2}$ for Poly(3-mTh), but it decreased to $7,766 \text{ Fcm}^{-2}$ from $10,000 \text{ Fcm}^{-2}$ for Poly(3-dTh) in high dielectric constant condition. For the electrode coated with Poly(3-mTh)

and Poly(3-imTh), C_{sp} values were increased with applied potential. This increasing in solution having high dielectric constant were more than in low dielectric constant one. When the C_{dl} values were determined for each electrode, it can be seen that they were changed more stability according to C_{sp} values (Fig 7 c-d). Copolymer was shown more capacitance than the other in low dielectric constant condition. But for all electrodes were shown a peak value at 0V and decreased as applied high potential. This decreasing was more in high dielectric constant condition than in low one for all polymers except for copolymer. So, the decreasing was $4,333 \text{ Fcm}^{-2}$ for Poly(Th); $3,600 \text{ Fcm}^{-2}$ for Poly(3-mTh); $1,366 \text{ Fcm}^{-2}$ for Poly(3-dTh) and $0,400 \text{ Fcm}^{-2}$ for Poly(3-imTh) in high dielectric constant condition. To investigate the surface morphology of the electrode, the SEM and AFM images of the electrode surface coated with Poly(Th) in high dielectric constant solution or low dielectric constant solution were given in Figure 8.

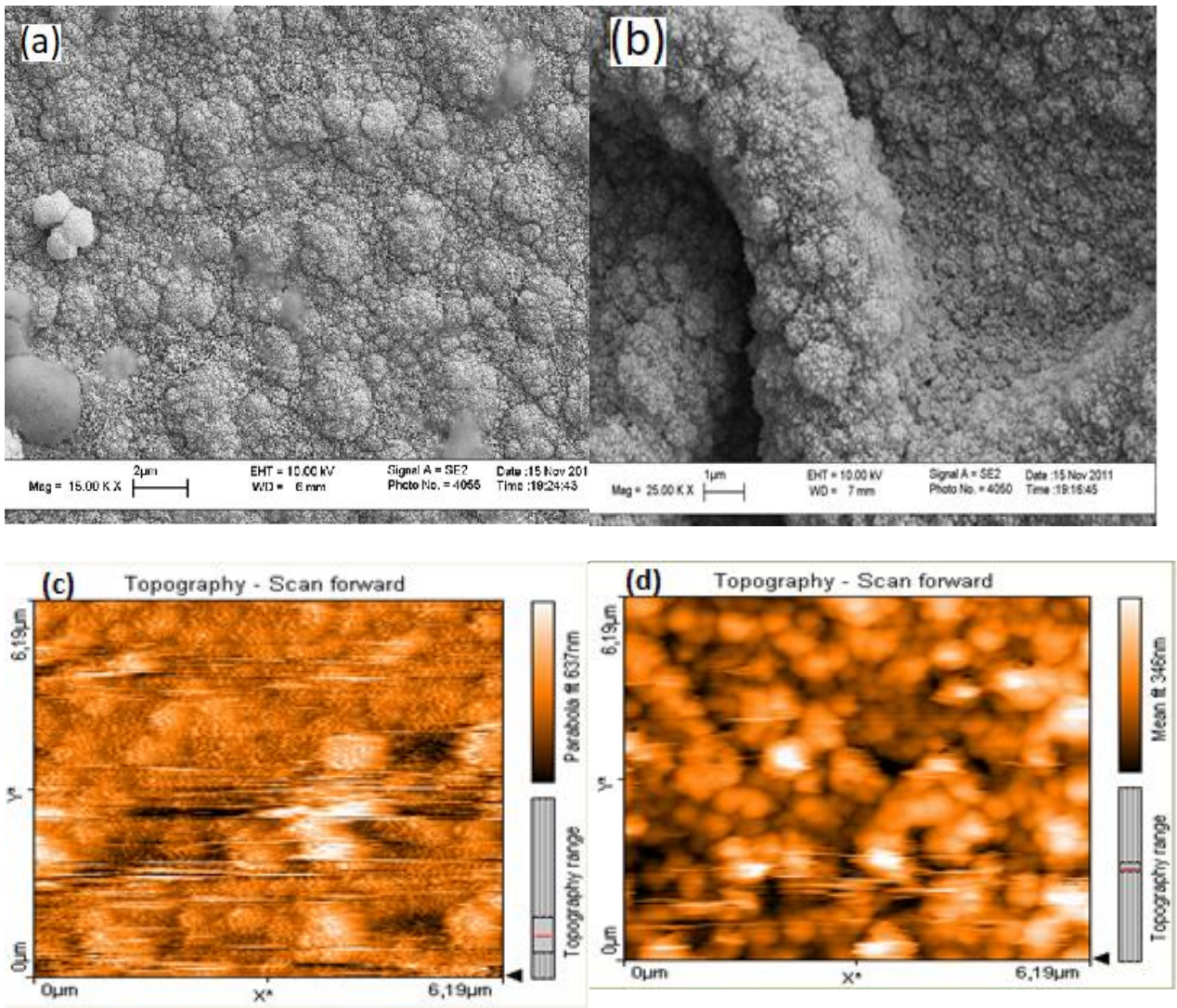


Figure 8. The SEM images of the electrode surface coated with Poly(Th) a) in $\epsilon:37,5$ b) $\epsilon:4,33$; and the AFM images of the electrode surface coated with Poly(Th) c) in $\epsilon:37,5$ d) $\epsilon:4,33$

As seen from the Figures, the ingredients of electropolymerization solution were affected the surface morphology of the electrode, because of dielectric constant. Figure 8a showed the surface image of electrode coated with the polymer in high dielectric constant solution. The typical brittle structure of conducting polymer can be easily see from the image. The morphology changed the more flexible form and cauliflower structure as the ingredients of the solution were changed, due to dielectric constant. Obtained from AFM images, the roughness value was 82.202 nm for $\epsilon:4,33$; when it was 234, 94 nm for $\epsilon: 37,5$.

CONCLUSION

In this study, the changing of the electrical and capacitive behavior of the electrodes coated with Poly(Th), Poly(3-mTh), Poly(3-dTh), or Poly(3-imTh) was investigated by using various effects. These effects were the $-R$ alkyl group bounded to Th ring at 3-position, the dielectric constant of electropolymerization solution and applied potential during impedance measurement. It was found that the P_{ox} and I_{max} values of Th, 3-mTh, 3-dTh and 3-imTh monomers and the C_{sp} and C_{dl} values of the electrode coated with its polymers were decreased by increasing the $-R$ group weight in high dielectric condition. According to the data, the C_{dl} values

changed to $0,200 \text{ Fcm}^{-2}$ from $1,200 \text{ Fcm}^{-2}$ for Poly(3-mTh); to $0,70 \text{ Fcm}^{-2}$ from $1,566 \text{ Fcm}^{-2}$ for Poly(3-dTh); to $2,066 \text{ Fcm}^{-2}$ from $4,933 \text{ Fcm}^{-2}$ for Poly(3-imTh) for the medium with to smallest dielectric constant. But this changing was to $3,800 \text{ Fcm}^{-2}$ from $0,966 \text{ Fcm}^{-2}$ for Poly(Th). Also, C_{sp} values were in the same range for derivative polymer coatings, which the range of $0-3,333 \text{ Fcm}^{-2}$. When the potential was applied to the electrode, C_{sp} value was decreased as -R weight was increased. In addition, it was seen that the value was increased for ring group in the same conditions. So, in $\epsilon:37,5$ C_{sp} value was increased to $36,666 \text{ Fcm}^{-2}$ from $21,666 \text{ Fcm}^{-2}$ when potential was increased to $0,6 \text{ V}$ from $-0,2\text{V}$ for Poly(3-imTh). It was determined that C_{sp} values for all the polymers coated electrodes were higher in low dielectric constant condition. Also, phase angle was increased by increasing the -R group weight in high dielectric constant condition, but decreased for ring structure. The phase angle was increased in low dielectric constant condition by increasing the applied potential.

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