



Conductive Polythiophene Nanoparticles Deposition on Transparent PET Substrates: Effect of Modification with Hybrid Organic-inorganic Coating

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ABSTRACT

In this work, polyethylene terephthalate (PET) substrate was treated using KOH solution and was modified using hybrid O-I coating containing polycaprolactone (PCL) as organic phase and tetraethoxysilane (TEOS) as inorganic phase. The coating was prepared through sol-gel process and applied on the surface by dip coater. Then, electrically conducting polythiophene (PTh) nanoparticles were deposited on the surfaces using chemical deposition method. A facile and rapid chemical oxidative deposition method using a binary organic solvent system during 12 min of reaction time was used and the effect of surface modification by hybrid coating on morphology of PTh nanoparticles were studied. The synthesized hybrid layer is transparent and its constituents are tunable, so, the hybrid layer is susceptible for wide range of application. The optical transparency of PTh nanoparticles coated on PET films characterized by UV-V is spectroscopy. Field-emission scanning electron microscopy (SEM) and laser particle-size analysis were used to study surface morphology, average size and size distribution of PTh nanoparticles. Electrical conductivity of PET substrate deposited by PTh nanoparticles was measured using four probe to be 1.7×10^{-5} S/cm.

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1. INTRODUCTION

Hybrid organic-inorganic materials are promising materials for various applications due to their excellent properties due to the combination of the organic and inorganic components [1-3]. Hybrid materials open a land of opportunities to discover new materials and new applications in numerous domains such as protective coatings [4], electronics and optics [5], organic light emitting diodes (OLEDs) [6], fuel cells [7], sensors [8], flame retarding [9], food packaging [10], energy, mechanics, automotive, housing, environment and medicine. Hybrids have recently attracted a strong interest in new fields of [11-13] material science since they combine flexibility of organics with chemical resistance and hardness of inorganics. They are transparent, due to the nanosized domains, and they can be applied as coatings on various substrates.

Hybrids commonly are obtained through sol-gel process. This process includes hydrolysis and the condensation through the formation of the inorganic network at temperatures below the softening or degradation of the organic part. Throughout the formation of the inorganic network, organic part is entrapped in the structure [14].

In recent years, electrically conducting polymers have attracted considerable attention in flexible electronic technology such as, solar cells, OLEDs, touch screens and flexible displays [15, 16]. Among these polymers, polythiophene (PTh) and its derivatives have a unique position because of privileged electrical, electrochemical, optical, and magnetic properties [17, 18]. PET substrate, because of its characteristics such as transparency, low cost, recyclability, thermal and chemical resistance is a popular polymer and has a wide range of application in electronic and optics. If PET is rendered conductive while keeping other properties unchanged, it will be one of the best candidates to be used in electronic and optical devices. Today's demand in many electronic devices such as solar cells and

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OLEDs, requires conductive films preferably flexible and/or transparent. In current light emitting diodes (LED) displays, a conductive transparent metal oxide such as indium thin oxide (ITO) being coated on glass substrates is used. These metal oxides have two drawbacks: they are fragile compounds and their implementation method is hard and expensive [19, 20]. Therefore, other conductive materials including conductive polymers have been considered as substituents for these conductive metal oxides because of low cost, versatility, ease of use and flexibility.

A possible way to improve electrical property of electrically insulating substrate is the implementation of a conductive layer onto the surface. Actually, coating on the PET substrate can be very rewarding since this flexible polymer is transparent and if conductive polymer were coated on its surface successfully, final product will be a conductive, transparent and flexible substrate which is a good candidate to be used in many areas of flexible electronic [21, 22]. In this regard, surface modification plays a leading role and it is the key factor that controls adhesion, morphology, quality and quantity of deposited coating.

There are various methods for PET surface treatment such as corona polling, Ion beam treatment [23], UV modification [24], various kinds of plasma treatment [25, 26] and treatment by chemical solvents which we chose as our treatment method because of low cost, versatility and ease of use. PET substrate was treated by 4 M aqueous KOH in order to improve the surface compatibility with final layer. This treatment increases wettability and surface hydrophilicity which was investigated by contact angle measurements. Aqueous KOH solution causes etching and creates porosities which makes the PET substrate suitable for the mechanical locking of the Hybrid material on the surface and this will not happen for untreated films. The second effect of aqueous KOH is chemical modification of the PET surface by creating polar groups which cause physical bonding and therefore, taking van der Waals forces into action for physical bonding and interactions. In recent years, application of hybrid materials in flexible electronics have attracted many researchers' attention [27, 28]. Sacconi and coworkers coated hybrid O-I layer on PET substrate and their final product could be used as capacitor [6]. If a conductive film can be coated on this hybrid layer, it can grant conductivity to the substrate [21, 29]. This leads to higher electrical strength will be ideal candidate for using in flexible electronics such as capacitors and resistors [6]. In this paper, PET substrate was treated by KOH solution. Hybrid coating were allowed to react in solution for a given time and then applied by deep coating method onto treated PET films. Finally, PTh nanoparticles were deposited on the surface, using chemical bath deposition method. The electrical conductivity and optical transparency of resulted film was measured using four

probe and UV-Vis spectroscopy. The morphology of obtained PTh nanoparticles were investigated by laser particle-size analyzer and FESEM techniques.

2. EXPERIMENTAL

2. 1. Materials and Equipments High purity tetraethoxysilane (TEOS), ϵ -caprolactone, ethylene Glycol, hydrochloric acid at 32% concentration (HCl), tetrahydrofuran (THF), ethanol (EtOH), potassium hydroxide (KOH), hydrogen peroxide 30% (H_2O_2), distilled Thiophene, anhydrous Iron (III) chloride ($FeCl_3$), dichloromethane (CH_2Cl_2) and acetonitrile (CH_3CN) were purchased from Merck and were used as received without further purification. 3-isocyanatopropyl- triethoxysilane, (ICPTES, Fluka), Tin (II) 2-ethylhexanoate, (Sigma), were used as received. General purpose transparent PET film with thickness of 100 μm was used as substrate. Contact angles were measured using sessile drop technique by goniometer equipped with a TZM-2 microscope attached to 3MP CMOS digital camera (BEL Company, Italy) under ambient laboratory conditions. A drop of distilled water (2 μL) was dropped on surface, and contact angle measurements were made within 30 s. The experiment were repeated for five points on three samples. Image J software was used to analysis optical and electronic microscopic images and contact angle measurements. The four-point microprobe method using a Keithley 224 high-current source was used to measuring electrical conductivity. The optical absorption was studied using a UV-vis spectrophotometer (Shimadzu spectrophotometer UV-1700) between 350 and 850 nm wavelengths. The morphology of the PTh nanoparticles on the modified films was observed using field emission scanning electron microscopy (FESEM, Hitachi, S-4160) on gold coated samples. Particle size distribution of PTh nanoparticles was determined by laser diffraction/ scattering particle size analyzer (Fritsch, Analysette 22 nanotech model).

2. 2. Film Treatment PET substrates were cut into 2 \times 2.5 cm² pieces, washed with deionized water and then ultrasonically cleaned in acetone before use. Cleaned PET films were immersed in 4 M aqueous solution of KOH for 2 h at 65°C. This solution treats PET surfaces and does not damage physical and chemical properties of substrate.

2. 3. Preparation of Hydroxy Terminated Poly (ϵ -Caprolactone) Hydroxy terminated poly (ϵ -caprolactone) (PCL-OH) were prepared by ring opening polymerization of CL in presence of EG as initiator and SnOct₂ as catalyst with the molar ratio of CL:EG:SnOct₂ equal to 20:1:0.02. The reaction was carried out for 24 hours at 120°C under magnetic

stirring and a clear yellowish solution of α, ω -hydroxy terminated PCL was obtained. [30]

2. 4. Preparation of Triethoxysilane Terminated Poly(ϵ -caprolactone)

ICPTES was added to the α, ω -hydroxy terminated PCL prepared at the previous section with molar ratio equal to 1:1 of OH:ICPTES. The reaction was carried out at 120°C in the same conditions reported above. After 1 hour, viscous solution of triethoxysilane terminated poly (ϵ -caprolactone) was obtained [31].

2.5. Preparation of PCL/ Silica Hybrid

Triethoxysilane functionalized polycaprolactone (PCL-Si/TEOS) were dissolved in 10 ml THF as a good solvent for PCL and was mixed until a homogeneous solution was obtained. Then, EtOH, water and HCl were added to the solution at the following molar ratios: EtO-/EtOH/H₂O/HCl: 1/1/1/0.1. EtOH makes system homogenous, water makes hydrolysis reaction possible and HCl is catalyst of reaction. Afterwards, the solution was stirred under 60°C for 1 h, to promote partial hydrolysis-condensation of the reactants. Over time, gradually viscosity increases and solution starts to gelation. Before gelation, the sol-gel solution was deposited onto treated PET substrates by dip coater. Samples were placed at room temperature for 30 min, and then they were heated at 70°C for 18 h, and finally postcured at 100°C for 2 h. Final coating was transparent coating with excellent adhesion to the PET substrate [31]

2.6. Polymerization and Deposition of Conducting Polythiophene

0.16 ml distilled thiophene monomer added to 10 ml dichloromethane. Another solution was prepared separately by adding 1.622 g anhydrous FeCl₃ as oxidant to 5 ml CH₃CN. Modified PET substrates were immersed in oxidant solution and then monomer solution was added dropwise to the solution under continues stirring at 0°C. The molar ratio of oxidant/monomer/surfactant in final solution was equal to 70:14:1. The polymerization was allowed to proceed for 12 min under stirring which leads to deposition of polythiophene on the surface. Coated substrates were sonicated in acetonitrile for removal of any residual oxidant and unreacted monomers and were stored in an airtight container after drying [21].

3. RESULTS AND DISCUSSION

3. 1. Surface Treatment

The effect of surface treatment on contact angle measurement of a drop of water is shown in Figure 1. The contact angle on the pristine PET is 71°, on PET surface treated by KOH

solution is 58° and for hybrid layer coated on substrate is 49° are shown in Figure 1-a to 1-c, respectively. Water contact angle decrease from 71° to 58° and to 49° meaning that PET substrates treatment and coating with hybrid material increases the surface hydrophilicity by introduction of polar groups on surface [21].

FESEM images of PET surface before and after KOH treatment are shown in Figure 2. As shown in Figures 2-a and 2-b, unmodified PET surface morphology is flat and smooth. On the other hand, surface roughening caused by KOH solution accompanying with the PET surface degradation can be seen in FESEM images of Figure 2-c and 2-d. Aqueous KOH solution causes etching and creates porosities that makes the substrate suitable for the mechanical locking of the PTh nanoparticles on PET. On the other hand, aqueous KOH cause chemical alternation of the PET surface by polar groups introducing. This groups takes van der Waals forces into action for physical bonding and interactions. The experimental data supporting this hypothesis is decreasing in water contact angle after surface treatment of the films which is due to hydrophilic groups attack on the surface [21]. Increase in total absorbance between 400 and 800 nm was calculated using Equation (1), in which W is the modified PET absorbance at a given wavelength, V is the the unmodified PET absorbance at the same wavelength and Y is the relative absorbance.



Figure 1. Decreasing in contact angle of a drop of water on PET surface, a) before and b) after treatment, c) after hybrid coating.

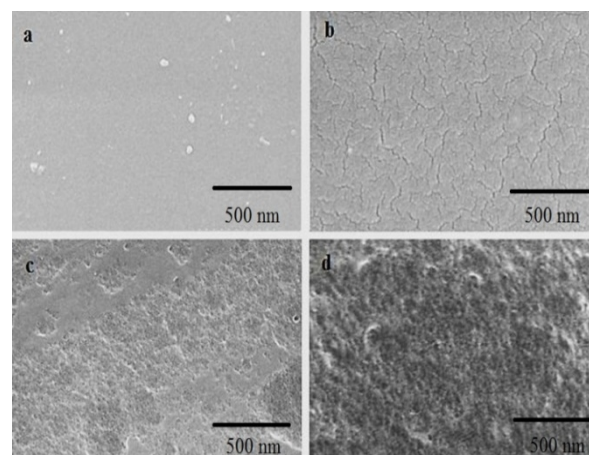


Figure 2. a and b) FESEM images of unmodified PET substrate, c and d) FESEM images of PET substrate modified with KOH solution.

Therefore, Y quantifies total absorbance in any given range and can be used for comparison of PET films before and after treatment. The results showed an increase in Y of about 27% after treatment with KOH solution and an increase of 14% in addition to that 27% after modification with hybrid layer.

$$Y = (W - V) / W * 100 \quad (1)$$

In order to evidence the observed decrease in transparency, FESEM images for PET before and after KOH treatment were studied. As can be seen in Figure 2, PET surface roughening and degradation caused by KOH solution and this can be an evidence for reduction in transparency.

3.2. Hybrid Coating and Polythiophene Deposition

The preparation of PCL-Si/TEOS hybrid material by sol-gel method reported in experimental section. Sol-gel reaction was progressed and resulted solution coated on treated PET substrate.

It is accepted that the presence of alkoxy silane groups on the polymeric chains increase the reactivity/miscibility with the metal alkoxide and promote a better interconnection between organic and inorganic phases. The α,ω -triethoxysilane terminated PCL was used to obtain shorter gelation time and better phase interconnection due to high reactivity of triethoxysilane end groups with hydroxyl groups.

PTh nanoparticles were deposited successfully on the PET substrate coated with hybrid material. Polymerization was carried out in solution in which FeCl_3 was used as the oxidant and dichloromethane/ acetonitrile as the binary solvent mixture. The solvent should be anhydrous organic with high dielectric constant and low nucleophilicity, like acetonitrile and dichloromethane. Water molecules in solution causes incorporation of carbonyl groups in PTh chains by nucleophilic attack of water molecules that breaks the conjugated structures and decrease the conductivity extremely. Final PET films had desired adhesion, flexibility and transparency. The layer coated on PET surface resists against forceps scraping. In addition, scotch tape test, which is a common qualitative adhesion test method, was performed on prepared samples and it was observed that the coating layer is not detectable on the quickly peeled off adhesive tape. Substrates conductivity measurement using four-probe method showed conductivity equal to 1.27×10^{-4} S/cm. This range of conductivity can be useful for flexible electronic applications. The hybrid coating composition is tunable. Thus, it can be used for controlling surface conductivity in electronic applications.

3.3. Surface Morphology and Size Distribution

Surface morphology and thickness of prepared films was studied by FESEM images. Figure 3 represents

FESEM cross section of PET substrate, coated with hybrid layer. As can be seen, hybrid layer has thickness of about $30 \mu\text{m}$ on the PET substrate. Morphology of prepared PTh nanoparticles in solution was investigated by FESEM method. PTh nanoparticles were synthesized in solution and the resulted powder was studied. As can be seen in Figure 4, the PTh particles have a spherical morphology with the homogenous size. Figure 5 represents FESEM images of PET substrate modified with hybrid O-I layer and deposited by PTh nanoparticles. As can be seen, nanoparticles are separated spheres with average size of about 50-200 nm. This morphology can explain the reason of low conductivity of substrate. For electrical conductivity, a uniform and integrated conductive layer is needed for charge carrier movement through the surface. As can be seen, spherical nanoparticles with diameters ranging from 20 to 100 nm can be seen in aggregated forms producing the globular morphology for PTh on PET.

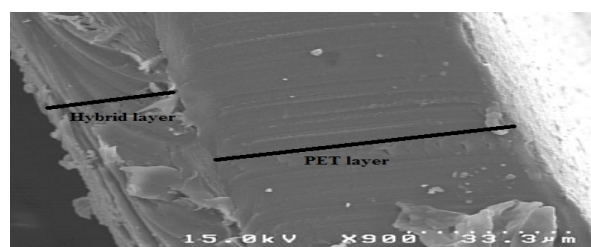


Figure 3. FESEM image showing cross-section view of hybrid O-I layer on PET substrate.

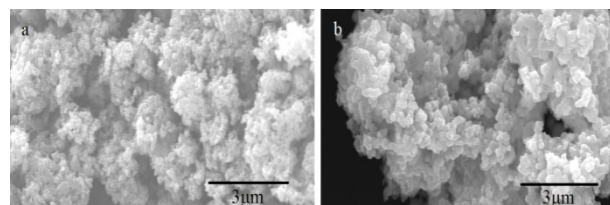


Figure 4. FESEM image of the PTh nanoparticles synthesized in the solution.

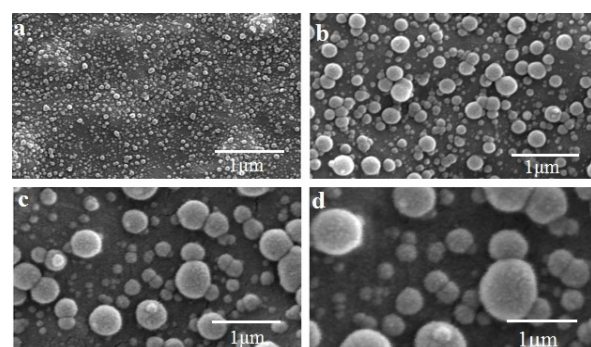


Figure 5. FESEM images of the morphology of PTh nanoparticles deposited on PET substrate modified with hybrid O-I layer at different magnification.

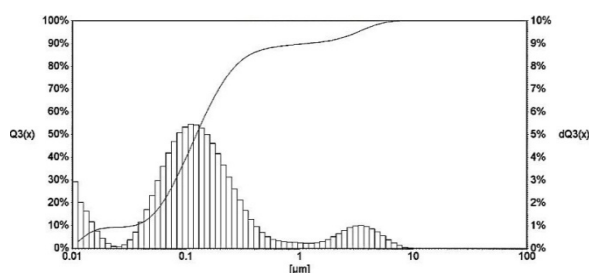


Figure 6. Particle size analysis of PTh nanoparticles dispersed in distilled water. Particles were synthesized in presence of different surfactants at 0°C, polymerization time of 12 min and surfactant/oxidant/monomer molar ratio of 1/70/14.

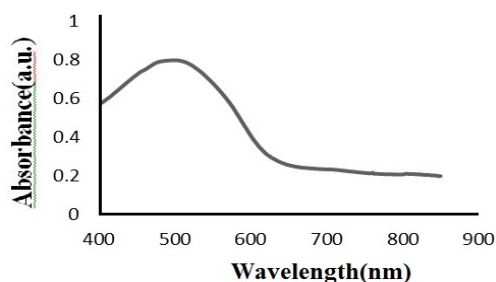


Figure 7. UV-Vis spectrum of the PET substrate, treated with KOH solution modified with hybrid O-I coating and coated with PTh nanoparticles.

Figure 6 represents particle size analysis of synthesized PTh nanoparticles. The particles were dispersed in the distilled water by sonication. As can be seen, size distribution of particles is narrow and similar and has an average diameter of about 100 nm.

Figure 7 represents the UV-Vis spectrum of PTh nanoparticles deposited on PET substrate modified with hybrid O-I coating. As can be seen the curve shows a pick between 400 and 600 nm. The absorption band between 420 and 600 nm is due to π - π transitions of the thiophene units which is an evidence for electrical conductivity [21].

6. CONCLUDING REMARKS

In this study, PET substrate was treated using KOH solution and was modified using hybrid O-I material containing PCL as organic phase and TEOS as inorganic phase. Then, PTh nanoparticles were deposited on these films and resulted product was studied using FE SEM and four probe methode. Final results showed separated spherical nanoparticles with the electrical conductivity of 1.7×10^{-5} S/cm and the average size of 50-200 nm. The film was transparent with excellent adhesion to the surface and excellent scratch resistance. The conductivity and flexibility of prepared hybrid coating can be controlled by tuning elements of the coated hybrid layer through sol-gel process.

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RESEARCH
NOTE

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در این مقاله بستر پلی اتیلن ترفتالات با استفاده از محلول پتاسیم هیدروکساید حاوی پلی کاپرولاکتان به عنوان فاز آلی و ترا اتوکسی سیلان به عنوان فاز معدنی اصلاح گردید. پوشش با فرایند سل-ژل تهیه گردید و با دیپ کوتر روی سطح اعمال گردید. سپس، نانوذرات پلی تیوفن رسانای الکتریسته با روش لایه نشانی شیمیایی روی سطح لایه نشانی شدند. یک روش لایه نشانی اکسیداسیونی سریع و راحت با استفاده از یک سیستم محلول دوتایی آلی در طی مدت ۱۲ دقیقه زمان واکنش استفاده شد و اثر اصلاح سطح با پوشش هیبریدی بر روی مورفولوژی نانوذرات پلی تیوفن بررسی و مطالعه گردید. پوشش هیبریدی تهیه شده شفاف است و قابلیت انعطاف پذیری بالا دارد. همچنین پوشش هیبریدی تهیه شده شفافیت نوری نانوذرات پلی تیوفن پوشش داده شده بر روی سطح با طیف نمایی ماوراء بنفش مطالعه شد. میکروسکوپ الکترونی روبش میدانی و آنالیز اندازه ذرات با لیزر برای مطالعه ی مورفولوژی سطح، متوسط اندازه ذرات و توزیع اندازه نانوذرات پلی تیوفن استفاده گردید. رسانایی الکتریکی نانوذرات پلی تیوفن لایه نشانی شده بر روی سطح با استفاده از فورپرپاب اندازه گیری شد و مقدار آن برابر $1.7 \times 10^{-6} \text{ S/cm}$ است.

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