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Influence of surface structure and morphology of PEDOT: PSS on its optical and electrophysical characteristics

This paper presents the results of a study of the effect of modification of the structure of the PEDOT: PSS polymer with hole conductivity on the optical and electrophysical properties of an organic solar cell. It was found that the modification of a polymer film with ethyl and isopropyl alcohols leads to a change in the morphology and roughness of the film surface. It has been determined that annealing of films in alcohol vapor promotes the formation of more uniform films. It is shown that upon modification of the PEDOT: PSS film in alcohol vapor the absorption spectrum shifts the absorption maximum of PEDOT to the short-wavelength region of the spectrum, the absorption of the aromatic PSS fragment decrease. X-ray phase analysis showed that after surface modification with alcohol vapor, the PEDOT and PSS chains change their structure. It is shown that the structural features of the surface morphology of PEDOT: PSS affect the electrophysical parameters of the films, such as the effective extraction rate and the effective time of flight of charge carriers. It was found that the modification of the surface of the PEDOT: PSS film leads to an improvement in the electrical transport properties of the films.

Keywords: PEDOT: PSS, Izopropanol, Ethanol, surface morphology, thermal annealing, aromatic fragment PSS, optical spectroscopy, impedance spectroscopy.

Introduction

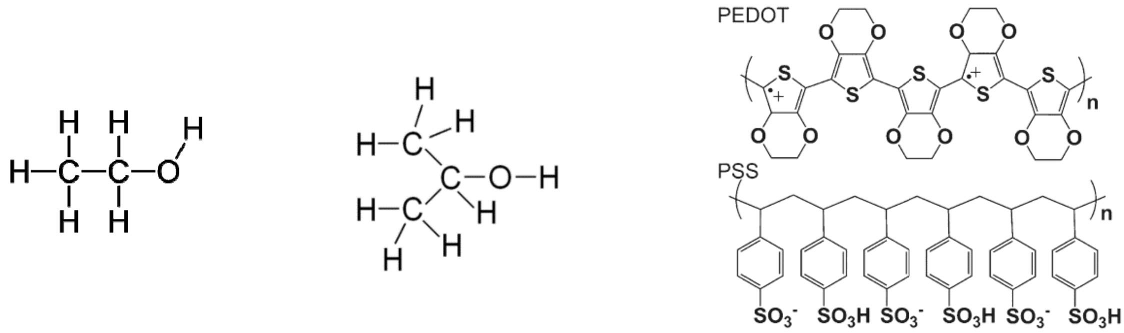
PEDOT is a relatively new member of the conductive polymer family. It has a fairly high electrochemical and thermal stability of electrical properties [1]. PEDOT is composed of ethylenedioxythiophene monomers (EDOT). However, EDOT is insoluble in many common solvents and is unstable in the neutral state as it rapidly oxidizes in the atmosphere. To improve its properties a polyelectrolyte solution (PSS) is added to EDOT, resulting in an aqueous suspension of PEDOT: PSS. Each phenyl ring of PSS monomer has one acidic SO_3H (sulfonate) group. Films with different properties can be obtained depending on the content of components, doping concentration, and particle size. The work function of the electron is approximately 5.2 eV. Due to the PSS content, the pH is between 1.5 and 2.5 at room temperature [2].

Morphology has a strong effect on the efficiency of generation and transport of charge carriers in PEDOT: PSS. The reason for this effect was the inhomogeneity of the films, which sharply reduces the efficiency of charge transport [3]. Thus, the main reason for the low indicators of transport characteristics is the tendency to aggregation and low solubility of active compounds, since crystallization of the components in PEDOT: PSS leads to the formation of polycrystalline domains, which are characterized by undesirable grain size and suboptimal crystalline ordering [4, 5]. However, the problems associated with the influence of the structural feature of PEDOT: PSS on fundamental parameters, such as the efficiency of electron transport of charge carriers, are still completely unsolved.

In this article, studies were carried out on the effect of structural changes in the PEDOT: PSS polymer on the morphological, optical, and electrophysical parameters of the films. To change the structure, the hole-conducting polymer PEDOT: PSS was annealed in vapors with a certain concentration of alcohols. The results were analyzed by comparing the surface morphology, optical and impedance spectra of PEDOT: PSS films.

Experimental

To change the surface morphology PEDOT: PSS was subjected to heat treatment at a certain ratio of ethyl and isopropyl alcohol vapors. The structural formulas of the compounds are shown in Figure 1. The preparation of the substrates was carried out according to the procedure [6, 7]. We used PEDOT: PSS (1 %, Ossila Al4083), Izopropanol, Ethanol (pure 99.9 % Sigma Aldrich). Before starting the experiments, the PEDOT: PSS solution was filtered through a 0.45 micro filter. Films PEDOT: PSS were obtained on a silica glass surface by centrifugation (on a SPIN150i centrifuge manufactured by Semiconductor Production System) at a rotation speed of 5000 rpm.



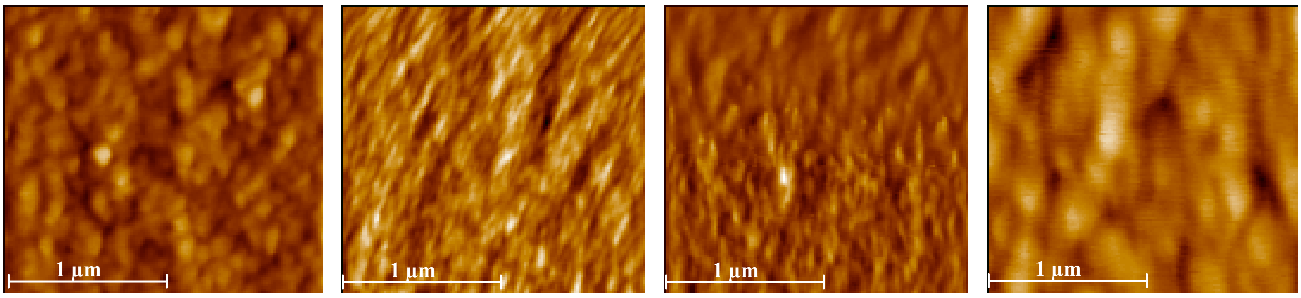
Ethanol Izopropanol PEDOT and PSS

Figure 1. Chemical structure of Ethanol, Izopropanol, PEDOT and PSS

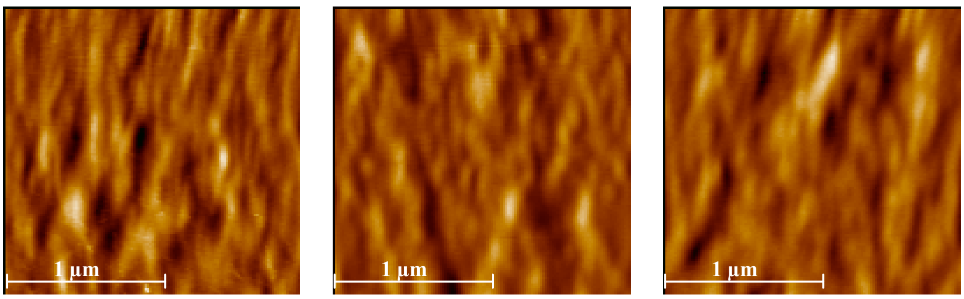
The topography of the film surface was studied using a JSPM-5400 atomic force microscope (AFM) (JEOL, Japan). A special modular program for analyzing scanning probe microscopy data (Win SPMII Data-Processing Software) was used to process the images obtained with AFM. Surface morphology and roughness of PEDOT: PSS thin films were analyzed from AFM images. The images of the surface of the PEDOT: PSS films were obtained in the semicontact scanning mode. The absorption spectra of the samples under study were recorded on an AvaSpec-ULS2048CL-EVO spectrometer (Avantes). The impedance spectra were measured using a P45X potentiostat-galvanostat in the impedance mode. The X-ray diffraction patterns of the films were obtained on a Rigaku SmartLab X-ray diffraction. To measure the impedance spectra on a CY-1700x-spc-2 sputtering apparatus (Zhengzhou CY Scientific Instruments Co., Ltd) an aluminum electrode 200 nm thick was applied to the surface of the films in a vacuum at a pressure of 10^5 Torr. Fitting of impedance spectra was carried out using the EIS-analyzer software package.

Results and Discussion

Images of the surface morphology of the PEDOT: PSS films are shown in Figure 2. Figure 2 shows that the PEDOT: PSS film has a granular structure. At the same time, large formations are observed against the background of a fine granular structure, the surface roughness is 0.63 nm.



PEDOT: PSS | 80 % PEDOT: PSS/ 20 % ethanol | 70 % PEDOT: PSS/ 30 % ethanol | 50 % PEDOT: PSS/ 50 % ethanol



80 % PEDOT: PSS/ 20 % isopropanol | 70 % PEDOT: PSS/ 30 % isopropanol | 50 % PEDOT: PSS/ 50 % isopropanol

Figure 2. Pictures of the surface morphology of films

After treatment with alcohol vapor for 10 minutes the proportion of large particles decreases significantly, the film surface becomes smooth, the surface roughness becomes 0.56 nm for ethyl alcohol, and 0.49 nm for isopropyl alcohol. A further increase in the concentration of alcohols in vapors leads to a smoothing of the film surface. Table 1 shows the roughness values of PEDOT: PSS films at different ratios of alcohols.

Table 1

Surface roughness of PEDOT: PSS films

Sample	R_a, nm
PEDOT: PSS	0,63
80 % PEDOT: PSS/20 % ethanol	0,56
70 % PEDOT: PSS/30 % ethanol	0,53
50 % PEDOT: PSS/50 % ethanol	0,48
80 % PEDOT: PSS/20 % isopropanol	0,49
70 % PEDOT: PSS/30 % isopropanol	0,47
50 % PEDOT: PSS/50 % isopropanol	0,45

X-ray diffraction patterns (XRD) of PEDOT: PSS thin films modified in ethyl and isopropyl alcohol vapors are shown in Figure 3. Two separate peaks observed at 2Θ at 3.5° and 25.6° in the original PEDOT: PSS film correspond to spatial lattices parameters d 25.2 Å and 3.5 Å, calculated according to Bragg's law $2d\sin\theta = \lambda$.

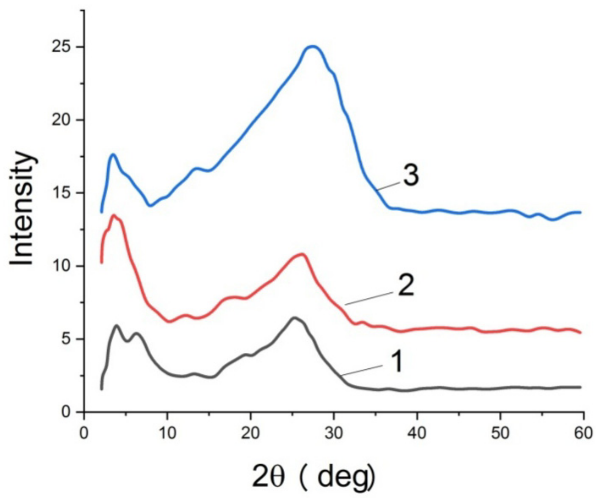
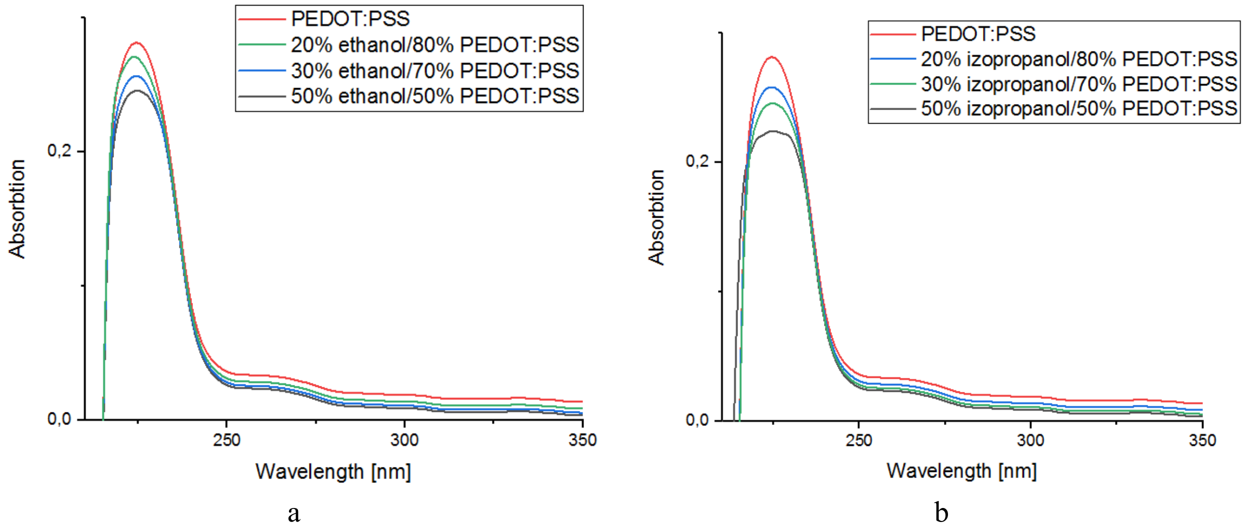


Figure 3. X-ray diffraction patterns (XRD) of PEDOT: PSS thin films:
1 — PEDOT: PSS, 2 — 50 % PEDOT: PSS/50 % ethanol, 3 — 50 % PEDOT: PSS/50 % isopropanol

The diffraction maximum observed at 3.5 °, with a spatial lattice of 25.2 Å, can be attributed to the distance between the lamellae (d 100 Å) of the PEDOT and PSS chains. After treatment in vapors of ethyl and isopropyl alcohols a slight change in the distance between the lamellae from 25.2 to 23.2 Å is observed (Fig. 3). A decrease in the distance between two points in the X-ray diffractogram indicates that the PEDOT and PSS chains pass from the benzoid structure to the quinoid structure; therefore, after modification in ethyl and isopropyl alcohol vapors, the structure becomes more planar.

Figure 4 shows the absorption spectra of the standart PEDOT: PSS films and films held for 10 minutes in alcohol vapor. The semiconducting polymer film PEDOT: PSS has a maximum at a wavelength of λ_1 = 224 nm with a spectral half-width of 28 nm (Fig. 4). In the absorption spectra of all PEDOT: PSS films, a shoulder with a maximum at 260 nm is observed, which is associated with the absorption of the aromatic fragment of PSS [8]. The position of the absorption maxima of the films obtained by holding PEDOT: PSS in alcohol vapor does not change, the half-width of the spectra increases. Table 2 shows the characteristics of the absorption spectra of the PEDOT: PSS films.



a) spectra of PEDOT: PSS/ethanol films; b) spectra of PEDOT: PSS/isopropanol films

Figure 4. Absorption spectra of PEDOT: PSS films

The absorbance value at the absorption maximum does not undergo significant changes. The treatment with alcohol vapor leads to a decrease in the values at the short-wave and long-wave maxima. It is known that, due to its hydrophobic properties, the PEDOT polymer does not dissolve in ethyl and isopropyl alcohols; in turn, PSS has a high degree of hydrophilic properties [9]. The treatment with alcohol vapor of the PEDOT: PSS film leads to a decrease in the absorption spectrum of the aromatic PSS fragment [10].

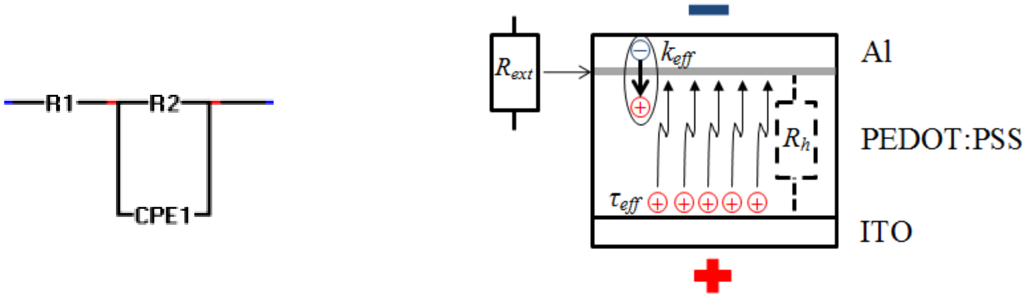
Table 2

Characteristics of the absorption spectra of PEDOT: PSS films upon annealing in the atmosphere

Sample	Adsorption peak		D_1	D_2	FWHM, пм
	λ_1, nm	λ_2, nm			
PEDOT: PSS	224	260	0,28	0,03	28
Ethanol					
80 % PEDOT: PSS/20 % ethanol	224	260	0,27	0,03	29
70 % PEDOT: PSS/30 % ethanol	224	260	0,26	0,03	29,4
50 % PEDOT: PSS/50 % ethanol	224	260	0,25	0,02	32
Isopropanol					
80 % PEDOT: PSS/20 % isopropanol	224	260	0,26	0,03	29
70 % PEDOT: PSS/30 % isopropanol	224	260	0,24	0,03	31
50 % PEDOT: PSS/50 % isopropanol	224	260	0,22	0,02	32

The observed changes in the absorption spectra of the films are associated with the structural features of PEDOT: PSS. Initially, without preliminary exposure to alcohol vapors, the film has a granular structure, with intense absorption of the aromatic PSS fragment in the spectrum. After keeping the film in alcohol vapor, the graininess of the film decreases, and the optical absorption density of PSS also decreases. Subsequent treatment with alcohol vapor leads to a further drop in the absorption of the aromatic fragment in the film. Thus, treatment with alcohol vapor leads to a change in the optical absorption spectra associated with the structural features of the film, due to a decrease in the amount of the aromatic PSS fragment.

The analysis of the impedance measurement results was carried out according to the diffusion-recombination model and for the fitting an equivalent circuit was used, shown in Figure 5 (a) [11]. Figure 5 (b) shows a diagram of the movement of charge carriers in a PEDOT: PSS film.



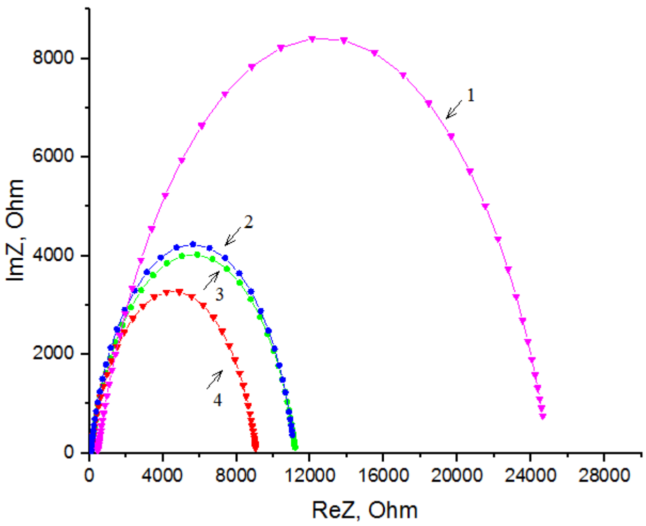
a b

a) equivalent electrical circuit; b) diagram of the movement of charge carriers in the film

Figure 5. Scheme of the transport of charge carriers in the film.

Figure 5 (a) shows the following parameters of the equivalent circuit for obtaining the hodograph of semiconductor films: R1 and R2 are the resistances corresponding to the resistances R_h and R_{ext}; CPE1, a constant phase element, is an equivalent circuit component that simulates the behavior of a double layer, but is an imperfect capacitor.

Figure 6 shows the impedance spectra of the standart PEDOT: PSS films and films treated in isopropyl alcohol vapor in a 50/50 ratio. Table 3 shows the values of the electrophysical parameters of the films, where k_{eff} is the effective rate of carrier extraction with PEDOT: PSS, τ_{eff} is the effective time of flight of charge carriers through the PEDOT: PSS, R_h is the resistance of the PEDOT: PSS film, R_{ext} is the resistance of charge carrier transfer to boundary PEDOT: PSS/electrode associated with the extraction of charge carriers with PEDOT: PSS. As can be seen from the data in Table 3, annealing in isopropyl alcohol vapor affects the resistance of the PEDOT: PSS film (R_h) and the resistance of charge carrier transfer at the PEDOT: PSS/electrode interface (R_{ext}). The resistance of the PEDOT: PSS film is rather high; after annealing at a temperature of 120 °C, a decrease in the resistance of the film by more than two times is observed. Aging in isopropyl alcohol vapor leads to a twofold decrease in the resistance of the PEDOT: PSS film without thermal annealing. Subsequent annealing slightly decreases the film resistance.



1 — PEDOT: PSS; 2 — PEDOT: PSS after thermal annealing; 3 — 50 % PEDOT: PSS/50 % isopropanol;
4 — 50 % PEDOT: PSS/50 % isopropanol after thermal annealing.

Figure 6. Light impedance spectra of PEDOT: PSS films at voltage parameters -500 mV and frequency from 100 kHz to 0.5 Hz

Changes are observed in the value of the resistance of charge carrier transfer at the interface PEDOT: PSS/electrode (R_{ext}). The R_{ext} resistance is most important for the unannealed PEDOT: PSS film. Upon thermal treatment of the film, R_{ext} is more than halved. At the same time, when the film is treated with alcohol vapors, R_h and R_{ext} are halved.

Table 3

The value of the electrophysical parameters of the films

Sample	R_h, Ohm	R_{ext}, Ohm	k_{eff}, s^{-1}	τ_{eff}, ms
Standart PEDOT: PSS	145,9	24532	44,3	22,57
Annealed film of standart PEDOT: PSS	65,5	11097	56,8	17,61
50 % PEDOT: PSS/50 % isopropanol	66,2	11161	92,5	10,81
Annealed film of 50 % PEDOT: PSS/50 % isopropanol	63,7	9010,6	93,5	10,70

It should be noted that the value of R_{ext} determines the efficiency of carrier injection from PEDOT: PSS films, and the smaller the value of R_{ext}, the greater the efficiency of accumulation of charge carriers in the cell. The quantities k_{eff} and τ_{eff} characterize the efficiency of carrier extraction from the PEDOT: PSS film and the effective time of flight of charge carriers along the PEDOT: PSS. Annealing the film insignificantly affects k_{eff} and τ_{eff}; at the same time, treatment with isopropyl alcohol vapor increases the efficiency of carrier extraction from PEDOT: PSS films by a factor of two and decreases the effective time of flight of charge carriers. The holes injected into the PEDOT: PSS diffuse to the electrode where they recombine with the electrons. The fast transport of the injected holes to the outer electrode is very important, since this reduces the likelihood of their reverse recombination. In our case, the fast transport of holes is ensured by changing the structure of PEDOT: PSS after treatment with isopropyl alcohol vapor; as a result, the efficiency of charge transport at the PEDOT: PSS/electrode interface increases.

Conclusions

An analysis of the experiments showed that a change in the structural features of PEDOT: PSS affects the generation and transport of charge carriers. It was found that thermal annealing of PEDOT: PSS films in isopropyl alcohol vapor leads to a change in the morphology and structure of the film, to an increase in the degree of domain homogeneity, and a decrease in roughness. X-ray phase analysis showed that after surface modification in ethyl and isopropyl alcohol vapors, the PEDOT and PSS chains pass from the benzoid structure to the quinoid structure. Upon modification of the PEDOT: PSS surface, a decrease in the absorption of the aromatic PSS fragment is observed in the absorption spectrum. It is shown that changes in the structure and morphology of the PEDOT: PSS surface affect the electrophysical parameters of the films. It was found that the modification of the surface of the PEDOT: PSS film leads to the optimization of the electrical transport characteristics of the film. Fast transport of holes is ensured by changing the structure of PEDOT: PSS after treatment with isopropyl alcohol vapor, as a result of which the efficiency of charge transport at the PEDOT: PSS/electrode interface increases.

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PEDOT: PSS құрылымы мен беттік морфологиясының оның оптикалық және электрофизикалық сипаттамаларына әсері

Мақалада кемтіктік өткізгіштігі бар PEDOT: PSS полимерінің құрылымын органикалық күн ұяшықтарының оптикалық және электрофизикалық қасиеттеріне модификациялаудың әсерін зерттеу нәтижелері келтірілген. Полимерлі қабықшаны этил және изопропил спирттерімен модификациялау қабықша бетінің морфологиясы мен кедір-бұдырлығының өзгеруіне әкелетіні анықталды. Спирттер буларындағы пленкаларды күйдіру біртекті пленкалардың түзілуіне ықпал ететіні дәлелденді. PEDOT: PSS қабықшасын спирттер буларында модификациялау кезінде, жұтылу спектрінде PEDOT максималды жұтылуы спектрдің қысқа толқындық аймағына ауысуы, сондай-ақ PSS хош иісті фрагментінің жұтылуының төмендеуі байқалады. Рентгендік фазалық талдау PEDOT және PSS тізбектерінің спирт буларының бетін өзгерткеннен кейін олардың құрылымын өзгертетінін көрсетті. PEDOT: PSS беттік морфологиясының құрылымдық ерекшеліктері, қабықшалардың тиімді шығару жылдамдығы және заряд тасымалдаушылардың тиімді ұшу уақыты сияқты электрофизикалық параметрлеріне әсер етеді. PEDOT: PSS қабықшасының бетін модификациялау қабықшалардың электрлік-транспорттық қасиеттерін жақсартуға әкелетіні анықталды.

Кілт сөздер: PEDOT: PSS, Izopropanol, Ethanol, беттік морфология, термиялық күйдіру, хош иісті PSS фрагменті, оптикалық спектроскопия, импеданс спектроскопиясы.

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Влияние структуры и морфологии поверхности PEDOT: PSS на его оптические электрофизические характеристики

Представлены результаты исследования влияния модификации структуры полимера PEDOT: PSS с дырочной проводимостью на оптические и электрофизические свойства органической солнечной ячейки. Установлено, что модификация полимерной пленки этиловым и изопропиловым спиртами приводит к изменению морфологии и шероховатости поверхности пленки. Доказано, что отжиг пленок в парах спиртов способствует формированию более однородных пленок. Показано, что при модификации пленки PEDOT: PSS в парах спиртов, в спектре поглощения наблюдается сдвиг максимума поглощения PEDOT в коротковолновую область спектра, а также уменьшение поглощения ароматического фрагмента PSS. Рентгенофазовый анализ показал, что после модификации поверхности парами спиртов цепи PEDOT и PSS меняют свою структуру. Показано, что структурные особенности морфологии поверхности PEDOT: PSS оказывают влияние на электрофизические параметры пленок, такие как эффективная скорость извлечения и эффективное время пролета носителей заряда. Установлено, что модификация поверхности пленки PEDOT: PSS приводит к улучшению электротранспортных свойств пленок.

Ключевые слова: PEDOT: PSS, Izopropanol, Ethanol, морфология поверхности, термический отжиг, ароматический фрагмент PSS, оптическая спектроскопия, импедансная спектроскопия.

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